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QUALITY ASSURANCE PROJECT PLAN

SITE WIDE GROUNDWATER (OPERABLE UNIT 03)

WEST LAKE LANDFILL SITE

June 5, 2019

Project #: 63N-001-001

SUBMITTED BY: Trihydro Corporation

1252 Commerce Drive, Laramie, WY 82070

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QUALITY ASSURANCE/QUALITY CONTROL PLAN
FOR SAMPLING ACTIVITIES
SITE WIDE GROUNDWATER (OPERABLE UNIT 03)
WEST LAKE LANDFILL SITE, BRIDGETON, MISSOURI
PREPARED BY: Trihydro Corporation
PREPARED FOR: OU-3 Respondents

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MICHAEL PHILLIPS, Ph.D. – Trihydro Corporation Quality Assurance Director	Date
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JACK HALL – MCL Quality Assurance Manager	Date
JOHN REYNOLDS – MCL Project Manager	Date

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List of Acronyms

ANSI	American National Standards Institute
ASTM	American Society of Testing and Materials
APM	Assistant Project Manager
CEC	Cation-Exchange-Capacity
CoC	Chain-of-Custody
CCV	Continuing Calibration Verification
COCs	Constituents of Concern
CLP	Contract Laboratory Program
DQO	Data Quality Objective
DOE	Department of Energy
FSP	Field Sampling Plan
FTL	Field Team Leader
GC/MS	Gas Chromatography/Mass Spectrometry
GPS	Global Positioning System
HASL	*EML Procedures Manual*, HASL-300, 28th Edition
HASP	Health and Safety Plan
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
MCL	Materials & Chemistry Laboratory, Inc.
MDL	Method Detection Limit

List of Acronyms (cont.)

µg/L	micrograms per Liter
mg/kg	milligrams per kilogram
mg/L	milligrams per Liter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NSL	Cotter Corporation
OU-3	Operable Unit 3
Pace	Pace Analytical Services, Inc.
Pace-I	Pace Analytical Services, Inc. in Indianapolis, Indiana
Pace-P	Pace Analytical Services, Inc. in Pittsburgh, Pennsylvania
Pace-E	Pace Analytical Services, Inc. Energy Services in Pittsburgh, Pennsylvania
pCi/g	average picocuries per gram
PM	Project Manager
QAD	Quality Assurance Director
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QAM	Quality Assurance Manual
SEM/EDS	Scanning Electron Microscope with Energy Dispersive x-ray Spectrometry
XRD	X-Ray Diffraction
RI/FS	Remedy Investigation/Feasibility Study
RL	Reporting Limit
RPD	Relative Percent Difference

List of Acronyms (cont.)

SOW	Scope of Work
SVOC	Semi-volatile Organic Compound
SM	Standard Method
SOP	Standard Operating Procedure
SW-846	Test Methods for Evaluating Solid Waste
Trihydro	Trihydro Corporation
EPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound
WP	Work Plan

1.0 PROJECT MANAGEMENT

Trihydro Corporation (Trihydro) prepared this quality assurance project plan (QAPP) on behalf of Bridgeton Landfill, LLC, Cotter Corporation (NSL), and the United States Department of Energy (DOE) (collectively OU-3 Respondents), for site-wide groundwater (Operable Unit 3 or OU-3), at the West Lake Landfill Site (Site) at 13570 St. Charles Rock Road in Bridgeton, Missouri. This QAPP contains the procedures that will be used to help ensure that data collected during OU-3 Remedial Investigation and Feasibility Study (RI/FS)-related sampling activities are complete, representative, comparable, accurate, and precise. The QAPP presents the management organization, project and quality assurance (QA) objectives, and QA/Quality Control (QA/QC) activities for the sampling program to complete assessment activities (as appropriate). It also describes the specific protocols that will be followed for sampling, sample handling and storage, chain-of-custody (CoC), field analyses, and laboratory analyses to promote QA/QC. The QA/QC procedures are structured in accordance with applicable technical standards, United States Environmental Protection Agency (EPA) requirements, regulations, guidance, and technical standards. It is intended to address the quality procedures associated with the sampling and analytical procedures outlined in the RI/FS Work Plan (WP) (Trihydro 2019a). The Field Sampling Plan (FSP) (Trihydro 2019b) describes the general approach and methods that will be used for collection of groundwater and other applicable samples (depending on what is needed and as outlined in the applicable work plan) from the Site.

This QAPP has been prepared in general accordance with EPA policy as presented in EPA Guidance for Quality Assurance Project Plans (QA/G-5) (EPA 2002), EPA Requirements for Quality Assurance Project Plans (QA/R-5; EPA 2001), Guidance for Data Usability in Risk Assessment," OSWER Directive No. 9285.7-09A (Apr. 1992), Establishment of Cleanup Levels for CERCLA Sites with Radioactive Contamination, Statement of Work (SOW) for the OU-3 RI/FS, Guidance for Data Quality Assessment: Practical Methods for Data Assessment QA/G-9 (EPA 2000), the Data Quality Assessment: A Reviewer's Guide QA/G-9R (EPA 2006a), Data Quality Assessment: Statistical Methods for Practitioners (EPA QA/G-9S) (EPA 2006b), EPA Guidance on Systematic Planning Using the Data Quality Objectives Process EPA QA/G-4 (EPA 2006c), and other relevant guidance documents applicable to the state and EPA region.

1.1 PROJECT/TASK ORGANIZATION

The OU-3 Respondents will direct this project. Trihydro will perform the field investigation, prepare reports, and perform any subsequent studies. Gary Risse, P.E., will serve as the Project Director for Trihydro. Trihydro will provide project management, with the following personnel supporting the project at this time: Stephen Lombardo, P.G. (PM or Project Manager), Michael Sweetenham, P.G. (APM or Assistant Project Manager), Charles VanHeuvelen P.G.

(FTL or Field Team Leader), and Michael Phillips, Ph.D. (QAD or Quality Assurance Director). The expanded project Organization Chart with project personnel is included as Figure 1-1.

The OU-3 Respondents and Trihydro PM will ensure that the sampling activities are conducted in accordance with the EPA Superfund Program criteria and the Missouri regulations and guidance documents. The OU-3 Respondents and Trihydro PM (or designee) will propose modifications to the WP (as needed) (Trihydro 2019a), make Site visits, and critically review the final reports to ensure that the QA objectives have been achieved. The EPA PM will provide written guidance to Trihydro to correct any deficiencies that may become evident. The EPA PM and the Trihydro PM (or qualified designee) may conduct audits of field and laboratory activities.

Pace Analytical Services, Inc. (Pace) in Indianapolis, Indiana (Pace-I) will analyze the groundwater (except volatile fatty acids) and soil samples (for Total Organic Carbon only). Pace in Pittsburgh, Pennsylvania (Pace-P) will provide radiochemistry analytical services. Pace Energy Services, LLC in Pittsburgh, Pennsylvania (Pace-E) will provide the volatile fatty acid analyses. Materials & Chemistry Laboratory, Inc. (MCL) in Oak Ridge, Tennessee will analyze most soil and extract samples, and perform specific extraction procedures. Each laboratory will prepare a report for the associated results. The data will be reviewed by the laboratories in accordance with the Pace-I, Pace-P, Pace-E, and MCL Quality Assurance Manuals (QAMs) (Attachments A-1, A-2, A-3, and A-4, respectively) with at least two levels of review prior to submittal to Trihydro and the OU-3 Respondents. The laboratory sample custodians (Pace and MCL sample custodians) will be responsible for managing the flow of samples when they arrive at the laboratory for analyses and will inform the Laboratory Project Managers.

MCL in Oak Ridge, Tennessee will analyze most soil (see Section 4.2.2) and extract samples (minus radiochemistry analyses and total organic carbon) and will perform specific extraction procedures. MCL analytical and quality assurance procedures and organizational information is included in Attachment A-4.

1.1.1 ROLES AND RESPONSIBILITIES

OU-3 Respondents

The OU-3 Respondents have the responsibility to review and approve reports and verify that they meet the requirements of the WP (Trihydro 2019a). Additionally, the OU-3 Respondents' responsibilities for the project may include:

- Review and approve reports (deliverables);
- Establish project policy and procedures to address the specific needs of the project as a whole; and
- Review and analyze overall task performance with respect to planned requirements and authorizations.

Trihydro Corporation Project Director

Trihydro will function as the primary contractor. They will be responsible for the proper implementation and management of work plans, sample collection, and preparation of reports. The Project Director maintains overall oversight and his responsibilities include the following:

- Review and approve reports (deliverables) before their submission to the OU-3 Respondents;
- Establish project policy and procedures to address the specific needs of the project as a whole; and
- Review and analyze overall task performance with respect to planned requirements and authorizations.

Trihydro Corporation Project Manager and Assistant Project Manager

The Trihydro PM and APM have the overall responsibility for phases of the investigation with oversight by the OU-3 Respondents. The Trihydro PM and APM are responsible for implementing the project and have the authority to commit the resources necessary to meet project objectives and requirements. The Trihydro PM and APM's primary function is to ensure that regulatory, technical, financial, and scheduling objectives are achieved successfully. The Trihydro PM and APM will:

- Select, coordinate, and schedule staff for the work assignments;
- Manage budgets and schedules;
- Prepare progress reports;
- Maintain and distribute the official approved QAPP;
- Monitor and direct subcontractors;
- Implement QA measures and any corrective action requirements;
- Attend review meetings;
- Interface with EPA;
- Perform final data assessment;
- Monitor and direct the field leaders;
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Prepare and assure quality of interim and final reports;
- Conduct initial Site safety training for project team personnel;

- Ensure Trihydro and subcontractor field team personnel have read and understand the Health and Safety Plan (HASP) (Trihydro 2019c);
- Ensure that work performed by Trihydro is conducted in accordance with safe practices outlined in this plan;
- Define project objectives and develop a detailed work plan schedule;
- Acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints;
- Orient field leaders and support staff concerning the project's special considerations;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Prepare monthly progress reports to the EPA;
- Interface and provide project status updates to the OU-3 Respondents; and
- Direct the organization of the data and final evidence file.

The Trihydro PM and APM have responsibility for ensuring that the project meets EPA Superfund objectives and Trihydro quality standards. Furthermore, the EPA Project Manager has the authority to inspect Trihydro's field methods; therefore, the Trihydro PM and APM will communicate the schedule of field events with the EPA PM. The Trihydro PM and APM will report directly to the EPA Project Manager and are responsible for technical QC and project oversight.

Trihydro Corporation Field Team Leader

The Trihydro FTL will conduct oversight of field activities. He will also be responsible for team supervision upon implementation of field activities, which will be in accordance with procedures in the associated FSP (Trihydro 2019b) and this QAPP. The Trihydro FTL has the overall responsibility for phases of the investigation in the field with oversight by the Trihydro PM and APM. The Trihydro FTL's primary function is to oversee the subsurface investigation and Site assessment activities. The Trihydro FTL will:

- Select, coordinate, and schedule staff for the work assignments;
- Plan and oversee field assessment activities;
- Manage all field subcontractors;
- Manage the field sample collection team;
- Evaluate shallow subsurface geology/hydrology and impacts;

- Prepare progress reports to the Trihydro PM; and
- Ensure that field staff conduct work in accordance with the Site HASP (Trihydro 2019c) and RSP.

The Trihydro FTL has the responsibility for ensuring that the field activities meet the guidelines identified in the FSP (Trihydro 2019b) and WP (Trihydro 2019a). The Trihydro FTL will report directly to the Trihydro PM.

Trihydro Quality Assurance Director

The Trihydro QAD will have direct access to contact the laboratories with QA/QC questions. He is responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations under EPA Superfund and Trihydro policies. The Trihydro QAD has the authority to stop work on the investigation as deemed necessary in the event of serious QA/QC issues. Specific functions and duties are:

- Audit field memoranda prepared by field personnel to ensure that the procedures for sample collection and sample custody are strictly adhered to;
- Review laboratory reports to ensure that adequate QA/QC procedures are imposed on the laboratory analytical results;
- Review and approve QA plans and procedures;
- Provide QA technical assistance to project staff;
- Report on the adequacy, status, and effectiveness of the QA program on a regular basis to the Trihydro Project Manager and Assistant Project Manager; and
- Distribute and re-distribute quality documents initially and upon revision.

The Trihydro QAD reports directly to the Trihydro PM and will be responsible for ensuring that procedures for this project are followed. In addition, the Trihydro QAD will be responsible for organizing technical staff to complete Tier I validation, and/or Tier II, Tier III, or Tier IV data validations of sample results from the analytical laboratory.

MCL data will be validated using Tier I data validation. The MCL data are specialty laboratory services for non-regulated results that will be used in transport and fate groundwater modeling. Data provided by Pace will be validated with at least a Tier II data validation, and the specific level of data validation will be determined by the final use of the data as discussed in Sections 6.1.2.2 and 6.1.2.3.

Laboratory Responsibilities

Pace-I and Pace-E (Volatile Fatty Acids only) will conduct the laboratory services during the groundwater and soil sampling activities. Pace-P will conduct the laboratory services for most radiochemistry sampling activities. Section 4.2.2 specifically describes where samples will be sent for analyses. The laboratory organization structures and internal responsibilities (for Pace-I, Pace-P, Pace-E, and MCL) are described in detail in the QAMs located in Attachments A-1, A-2, A-3, and A-4, respectively.

Pace and MCL Project Managers

The Pace and MCL PMs will report directly to the Trihydro PM/APM and will be responsible for the oversight of production and final review of the analytical reports and the case narratives to verify that any data quality issues are thoroughly explained and the requirements of this QAPP have been met. The PMs will serve as liaison between the laboratory and the Trihydro QAD. They will communicate any special project instructions that affect the way that analyses are to be performed, the data evaluated, sample turnaround time, or the results reported. The operations managers or designee will inform the Pace and MCL PMs of samples status and will:

- Coordinate laboratory analyses;
- Supervise in-house CoC;
- Schedule sample analyses;
- Oversee data review;
- Oversee preparation of analytical reports;
- Compare bottle orders against bottle sets for accuracy and to ensure proper chemical preservation of bottle sets before they are shipped to the Site;
- Approve final analytical reports prior to submission to Trihydro; and
- Sign the title page of the QAPP.

Pace Quality Assurance Officers

The Pace and MCL Quality Assurance Officers (QAOs) have the overall responsibility for data after it leaves the laboratory. In addition, the Pace and MCL QAOs (or designee) will:

- Oversee laboratory QA;
- Oversee QA/QC documentation;
- Conduct detailed data review per laboratory requirements;

- Determine whether to implement laboratory corrective actions, if required;
- Define appropriate laboratory QA procedures;
- Prepare and review laboratory standard operating procedures (SOPs); and
- Sign the title page of the QAPP.

Final responsibility for project quality rests with Trihydro's PM/APM. The Pace and MCL PMs, prior to release of data will provide independent QA review.

Pace and MCL Sample Custodians

The Pace and MCL Sample Custodians will report to the Laboratory PMs and be staffed by laboratory personnel. Responsibilities of the Pace and MCL Sample Custodians are:

- Receive and inspect the incoming sample containers;
- Record the condition of the incoming sample containers;
- Sign appropriate documents;
- Verify CoC documentation;
- Notify the laboratory manager and laboratory supervisors of sample receipt and inspection;
- Assign a unique identification number and customer number, and entering each into the sample receiving log;
- Initiate transfer of the samples to appropriate lab sections with the help of the laboratory manager
- Control and monitor access/storage of samples and extracts; and
- Provide when samples are received indicating the sample names, sample condition, and sample parameters to be analyzed.

The Pace and MCL technical staff will be responsible for sample analysis and identification of corrective actions. The staff will report directly to the operations managers or designee.

1.2 PROBLEM DEFINITION AND DESCRIPTION

Previous investigations for Operable Units 1 and 2 at the Site indicated that there have been releases of constituents of concern (COCs) from each of these units. Elevated concentrations of COCs including petroleum hydrocarbons, volatile organic compounds (VOCs), trace metals, trace anions, and various radionuclides have been found in groundwater, which will be sampled under this RI. Data gaps were identified in the Scope of Work that will be

addressed by the RI sample data in order to characterize the nature and extent of impacts in groundwater and evaluate sources of contamination.

The problem definition can be summarized as follows:

- Are chemical concentrations in groundwater at levels that exceed the EPA Maximum Contamination Levels, State-specific screening levels, GWPS, and/or naturally occurring background levels?
- Is the Site fully characterized in accordance with the work plan?
- Is there potential for workers, the public, or ecological receptors to be exposed to contaminants?
- Is there evidence that any potential COCs are migrating outside of the target area?
- Is there evidence that any potential COCs are being remediated?

2.0 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) are qualitative and quantitative statements used to clarify the study objectives, define the appropriate type of data to collect, determine the appropriate conditions from which to collect the data, determine the quality of the data used to support decisions at the Site, and specify tolerable limits on decision errors. Specific DQOs for this investigation are included in the WP (Trihydro 2019a) and are in accordance with EPA Guidance on Systematic Planning Using the Data Quality Objectives Process EPA QA/G-4 (EPA 2006c).

The process for developing the DQOs for the Site include the following seven steps:

1. State the Problem
2. Identify the Decision
3. Identify Inputs to the Decision
4. Define the Boundaries of the Investigation
5. Develop a Decision Rule
6. Specify Tolerable Limits on Decision Errors
7. Develop the Plan for Obtaining Data

2.1 CRITERIA FOR MEASUREMENT DATA

Six quantitative/qualitative measures of quality will be employed during Site activities:

- Precision
- Accuracy
- Completeness
- Representativeness
- Decision Rule
- Comparability
- Sensitivity

The QA objectives for these criteria and procedures to compare calculated values to the objectives are described below.

2.1.1 PRECISION

Precision is the degree of agreement between the numerical values of a set of replicate samples performed in an identical fashion. Field precision is assessed by the collection of blind duplicates at a rate of 1 duplicate for every 10 field samples. Field duplicate samples will be taken concurrently with the parent sample. For water field duplicates, the relative percent difference (RPD) for each constituent must be less than 30%. For soil field duplicates, the RPD for each constituent must be less than 50%.

Laboratory precision will be assessed through calculation of the RPDs for replicate analyses of samples including matrix spikes (MS), matrix spike duplicates (MSD), laboratory control samples (LCS), and laboratory control samples duplicates (LCSD). Precision will be based on the analytical data from the laboratory and field replicate analyses.

Precision may be reported as relative percent difference as expressed by the following formula:

$$RPD = \frac{(C_1 - C_2)}{\frac{(C_1 + C_2)}{2}} * 100\%$$

Where:

C1 and C2 are the concentrations of duplicate samples.

A summary of the laboratory control samples, including a description of use, frequency, and acceptance criteria is included in Table 2-1.

2.1.2 ACCURACY

Accuracy is the measure of agreement of a result to the accepted (or true) value. Errors may arise from personnel, instrumental, or method factors. Accuracy in the field is assessed through use of field, equipment, and trip blanks and adherence to sample handling procedures, preservation methods, and holding times (Table 2-2). At least one trip blank will be included in each cooler containing VOC samples. At least 1 equipment and field blank will be collected per 10 samples per sampling event. At least 1 matrix spike pair will be collected per 20 samples per sampling event. For soil samples the matrix spike pair will not provide a good accuracy measurement because of highly possible sample inhomogeneity. MCL will use duplicate LCSs to evaluate accuracy for each batch.

Field accuracy will be assessed based on the methods recommended by the EPA Contract Laboratory Program (CLP) for common laboratory contaminants. If a contaminant is detected in the equipment, field, or trip blanks, the reported value will be multiplied by 10. Then the associated environmental sample results will be compared to the blank

detection results. If the environmental sample results are found to be within 10 times the original blank detection, the data will be "JB" qualified and considered an estimated value due to possible cross-contamination. As noted in the Data Validation Variance Documentation (Attachment B), Trihydro uses a "10 times" rule for possible contaminants identified in the blank samples. However, if contaminants are detected in environmental samples at values below the original blank detection or the associated reporting limit (RL), the contaminants will be qualified with a "U" and considered non-detect at the RL.

Laboratory accuracy is assessed by evaluating LCS, LCSD, MS, MSD, and organic system monitoring compounds (surrogate) percent recoveries. Although LCSDs (or another form assessing laboratory precision) are not part of routine analyses for the laboratories, they will be analyzed when the MS is prepared from another client's sample or not prepared at all. Laboratory precision methods will be discussed with between Trihydro and the laboratories prior to the sampling event. Analytical accuracy is estimated from the recovery of spiked analytes from the matrix of interest. Laboratory performance in a clean matrix is estimated from the recovery of analytes in the LCS. The recovery of each spiked analyte in the MS, MSD (if performed), LCS, LCSD (if performed), and surrogate is completed using the following formula:

$$\text{Recovery} = \%R = \frac{(C_s - C_u)}{C_n} * 100\%$$

Where:

C_s = Measured concentration of the spiked sample aliquot

C_u = Measured concentration of the unspiked sample aliquot (use 0 for the LCS or surrogate)

C_n = Nominal (theoretical) concentration increase that results from spiking the sample, or the nominal concentration of the spiked aliquot (for LCS or surrogate)

2.1.3 COMPLETENESS

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Completeness is the ratio of the number of validated sample analyses to the total number of sample results required by the sampling program, calculated as follows:

$$\text{Completeness} = \frac{\text{Number of valid samples collected}}{\text{Number of samples planned or expected}} * 100\%$$

The field completeness objective for this project will be 95%. If necessary, the field crew may be required to return to the Site in order to meet completeness objectives. Trihydro will coordinate with EPA on these decisions.

Laboratory completeness is a measure of the amount of valid measurements obtained from the total number of laboratory measurements taken in this project. The laboratory completeness objective for this project, with respect to the critical parameters listed in the associated work plan is 95%. The ability to meet or exceed a completeness objective is dependent on the nature of samples submitted for analysis. If data cannot be reported without qualifications, project completeness goals may still be met if the qualified data (i.e., data of known quality even if not perfect) are suitable for specified project goals.

2.1.4 REPRESENTATIVENESS

Representativeness expresses the degree to which data accurately and precisely represent an environmental condition. Representativeness is a qualitative parameter, which is dependent upon the proper design of the sampling program and proper laboratory protocol. Adherence to methods specified in the work plan and careful calibration, maintenance, and monitoring of field instruments is necessary to ensure representativeness of field data. Field personnel will have previous data available at the time of sampling, will be able to qualitatively evaluate representativeness of field measurements in "real time," and can take corrective action if needed to ensure that field measurements are representative. Representativeness in the laboratory is ensured by using proper analytical procedures, meeting holding times, and analyzing and assessing field duplicate samples. In addition, overall data representativeness is a function of the design of the sampling program.

2.1.5 DECISION RULE

The decision rule defines the analytes of interest and associated action levels. Once the analytes and associated action levels are defined, a series of "if then" statements are established to define a path forward regarding the data. Decision rules are provided in the WP (Trihydro 2019a).

2.1.6 COMPARABILITY

Comparability expresses the confidence with which one data set can be compared with another. Comparability is dependent upon the proper design of a sampling program and will be satisfied by ensuring that the sampling plan is followed and that appropriate sampling protocols are used. Additionally, comparability is dependent upon the laboratory's ability to maintain required method certifications and adequately train personnel to analyze data in accordance with required analytical methods.

2.1.7 SENSITIVITY

Sensitivity is capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest. (EPA 2006a). Laboratory sensitivity can determine the minimum

concentration that can be measured by the method (method detection limit), by the instrument (instrument detection limit), or by a laboratory (quantitation limit). Methods of laboratory sensitivity are further discussed in the laboratories' QAMs (Attachments A-1, A-2, A-3, and A-4).

2.2 SPECIAL TRAINING AND CERTIFICATIONS

Field and Laboratory personnel will participate in necessary training and acquire certifications as determined by the Site. Trihydro Site personnel requirements for safety are described further in the Site-specific HASP (Trihydro 2019c). Laboratory personnel will conduct training in accordance with descriptions listed in their Quality Assurance Manuals (Attachments A-1, A-2, A-3, and A-4).

2.2.1 TRAINING

Field personnel are required to be familiar with the applicable company field procedures. The Trihydro PM will keep the training records for Trihydro field personnel. The training records for the laboratory personnel will be kept with the laboratory QA departments.

2.2.2 CERTIFICATION

Personnel involved in this project as PMs, Quality Officers, and the Trihydro FTL (and associated personnel) will be required to review this QAPP and sign the front cover (or equivalent) indicating that they are familiar with the QAPP. A record of the signature page(s) will be kept in the project file at Trihydro. The Kansas Department of Environment and Health primarily (Attachment A-5), certifies Pace-I to perform analyses. The Commonwealth of Pennsylvania Department of Environmental Protection (Attachment A-6) and American National Standards Institute (ANSI) National Accreditation Board (Attachment A-7), certifies Pace-P to perform analyses. The Pennsylvania Department of Environmental Protection, Bureau of Laboratories (Attachment A-8), certifies Pace-E to perform analyses. The Perry Johnson Laboratory Accreditation for ISO/IEC 17025 as part of the DOE DOECAP Program. (Attachment C-9) certifies MCL to perform analyses. A rigorous QA/QC program is maintained to provide clients with reliable data.

2.3 DOCUMENTATION AND RECORDS

Documentation and records will be maintained to help ensure field and laboratory observations and data are communicated appropriately and archived pursuant to the Site requirements. Detailed descriptions of these records are discussed below.

2.3.1 DOCUMENTATION

Field observations are critical to the verification and interpretation of the laboratory data. Field observations during sampling will be recorded on the field form and/or applicable electronic means (data-logger, global positioning system (GPS) unit, etc.). Field forms are presented in the FSP (Trihydro 2019b). In addition, the field activities will be documented in a bound field logbook with numbered pages. Entries in the logbook will be made with indelible ink. The information documented will include, at a minimum: field staff names that are involved in sample collection activities for the specific day; photos with descriptions and locations; sample collection times and container sizes; amounts and types of any measurements; weather conditions; and/or GPS coordinates collected at each sampling point. Field documentation procedures are outlined in the FSP (Trihydro 2019b).

2.3.2 RECORDS

Trihydro will be the custodian of records, and will maintain the contents of records for the Site activities, including relevant reports, logs, field notebooks, pictures, subcontractor reports and data reviews in a secured, limited access area and under custody of the Trihydro PM. Field data types may include field screening, water quality, fluid level, and location data. The final records may include:

- Field logbooks;
- Field data and data deliverables;
- Photographs;
- Drawings/Figures;
- Laboratory data deliverables;
- Data validation reports;
- Progress reports, quality assurance reports, interim project reports, etc.;
- Custody documentation;
- Groundwater sample collection logs with well screening parameters; and
- Soil sample collection logs.

Trihydro will maintain Site records at their Laramie, Wyoming, office for at least 10 years after the completion of Site activities, or as deemed necessary by OU-3 Respondents. Additionally, the laboratory will retain records for 10 years after analyses.

The laboratory records will be kept with the Pace and MCL Project Managers. Data package deliverables from the laboratory meeting the requirements of the EPA CLP-like specified data package deliverables, with modifications as required reflecting the use of EPA approved methods will be maintained by Trihydro and Pace. Sample custody and associated analyses will be completely documented. Data packages will contain information to completely document laboratory analysis procedures. MCL will provide data packages containing the necessary information to document the laboratory analysis procedure and process.

3.0 DATA QUALITY ASSESSMENT

Data quality assessments are the evaluation of data to determine if data obtained from environmental data operations are of the right type, quality, and quantity to support their intended use (EPA 2000). Data quality assessment completes the data life cycle by providing an assessment needed to determine if the planning objectives were achieved. Trihydro will use their data validation process and data validation reports to document the data quality assessment for the Site environmental data. Data quality assessment procedures will be performed in accordance with the Guidance for Data Quality Assessment: Practical Methods for Data Analysis QA/G-9 (EPA 2000) and the Data Quality Assessment: A Reviewer's Guide QA/G-9R (EPA 2006a).

Existing (or historic) and newly-collected data will be evaluated against a five-step statistical process described in detail in the Data Quality Assessment: Statistical Methods for Practitioners (EPA QA/G-9S) (EPA 2006b). Five linear-statistical steps will be employed during the evaluation of the data:

1. Review of the Site's objectives and sampling design: The goal of this activity is to develop quantitative statements of the reviewer's tolerance for uncertainty in the conclusions drawn from the data and in actions based on those conclusions.
2. Conduct a preliminary data review: The goal of this step is to review calculations of basic statistical methods and graphical representation data.
3. Select the statistical method: The goal of this step is to identify the appropriate statistical method that will be used to draw conclusions from the data.
4. Verify the assumptions of the statistical method: The goal of this step is to assess the validity of the statistical test chosen.
5. Draw conclusions from the data: The goal of this step is to use the chosen statistical test to draw conclusions to ensure that the data are adequate for the objectives described in Step 1.

4.0 DATA GENERATION AND ACQUISITION

The purpose of this section is to describe the data generation and acquisition that will be implemented by the project team. This appropriate tracking of data generation and acquisition will ensure that the data collected are of sufficient quality to meet overall project objectives for the RI. Generally, data are generated in the laboratories in accordance with the laboratories' QAMs (Attachments A-1, A-2, A-3, and A-4).

4.1 SAMPLE HANDLING AND CUSTODY

From sample collection through laboratory analysis to the final evidence files, the procedures for sample handling and custody of the samples are described below. A sample or evidence file is in one's custody if it is:

- In one's physical possession;
- In one's view, after being in one's possession;
- In one's physical possession and placed in a secured location; or
- In a secured area restricted to authorized personnel only.

As few people as practical should have custody of the samples to reduce the chance of mishandling.

4.1.1 FIELD CUSTODY PROCEDURES

The Trihydro FTL (or qualified designee) is generally responsible for successful implementation of field custody procedures. Specific field custody procedures are discussed, in detail, in the FSP (Trihydro 2019b).

4.1.2 LABORATORY CUSTODY PROCEDURES

The analytical laboratory assumes responsibility for the integrity and security of the samples after custody transfer is completed from the sampling team or the transportation service (if appropriate) to the laboratory. The laboratory custody procedures are described in the QAMs in Attachments A-1, A-2, A-3, and A-4. Sample receipt and disposal procedures are described in the SOPs in Attachment C. Analytical holding times and bottle requirements are included in Table 2-2.

4.2 ANALYTICAL METHODS

Both field and laboratory analytical procedures will be performed during this project. Field analytical procedures will be carried out primarily by the field team and will include groundwater multi-meter parameters. A summary of the field and laboratory analytical procedures are described below.

4.2.1 FIELD ANALYTICAL PROCEDURES

Groundwater samples will be analyzed using procedures discussed, in detail, in the FSP (Trihydro 2019b). Hand entry of field parameters will be subject to 100% QC checks. Data entered from dataloggers will be subject to spot checks (e.g., 10%) to confirm data were recorded and uploaded correctly. If problems are identified during spot checks, additional QC measures will be implemented.

The precision criteria for the multi-meter calibration readings will be ±10% from the indicated calibration standard. Calibration forms will be kept with the project field forms for each day of calibration.

4.2.2 LABORATORY ANALYTICAL PROCEDURES

The WP (Trihydro 2019a) includes tables and figures that delineate the sample locations. Groundwater and soil samples may be analyzed for the following:

- VOCs by Test Methods for Evaluating Solid Waste (SW-846) Method 8260C (performed by Pace-I)
- Semi-volatile organic compounds (SVOCs) by SW-846 Method 8270 (performed by Pace-I)
- Total and dissolved metals by SW-846 Methods 6010 and 6020 and mercury by SW-846 Method 7470A (performed by Pace-I)
- Chloride, fluoride, and sulfate by SW-846 Method 9056 (performed by Pace-I)
- Polychlorinated biphenyl-1221 (Aroclor-1221) by SW-846 Method 8082 (performed by Pace-I)
- Total Hardness by Standard Methods (SM) 2340B (performed by Pace-I)
- Total Dissolved Solids by SM 2540C (performed by Pace-I)
- Ferrous Iron by HACH Method 8146 (performed by Pace-I)
- Chemical Oxygen Demand by EPA Method 410.4 (performed by Pace-I)
- Sulfide by SM 4500-S2-D (performed by Pace-I)
- Nitrogen, Nitrate by EPA Method 353.2 (performed by Pace-I)

- Nitrogen, Nitrite by EPA Method 353.2 (performed by Pace-I)
- Nitrogen, Nitrite plus Nitrate by EPA Method 353.2 (performed by Pace-I)
- Phosphorus by EPA Method 365.1 (performed by Pace-I)
- Nitrogen, Ammonia by SM 4500-NH3 G (performed by Pace-I)
- Total Organic Carbon by SM 5310C (performed by Pace-I)
- Radium-226 by EPA Method 903.1 (performed by Pace-P)
- Radium-228 by EPA Method 904 (performed by Pace-P)
- Isotopic Uranium (U-234, U-235, U-238) by *EML Procedures Manual*, HASL-300, 28th Edition (HASL) 300 (performed by Pace-P)
- Isotopic Thorium (Th-228, Th-230, Th-232) by HASL 300 (performed by Pace-P)
- Volatile Fatty Acids by Method AM23G (performed by Pace-E)

Soil samples will be analyzed for the following:

- Total Organic Carbon by the Walkley-Black Procedure (performed by Pace-I)
- Radium-226 by EPA Method 903.1 (performed by Pace-P)
- Radium-228 by EPA Method 904 (performed by Pace-P)
- Isotopic Uranium (U-234, U-235, U-238) by HASL 300 (performed by Pace-P)
- Isotopic Thorium (Th-230, Th-232) by HASL 300 (performed by Pace-P)
- Total metals (Barium, Calcium, Iron, Manganese, Magnesium, Potassium, and Sodium) by SW-846 Methods 6010 (performed by MCL)
- Carbonate by SM 2320E (performed by MCL)
- Fluoride, Phosphate, and Sulfate by EPA Method 300.0 (performed by MCL)
- Ferric Iron calculated between 6010 Iron (performed by MCL)
- Ferrous Iron by SM 3500-Fe B (performed by MCL)
- Sulfide by Method EPA-OW-OST 376.3
- U(VI) by SOP MCL-7737 (performed by MCL)

- Cation Exchange Capacity (CEC) by EPA Method 9081 (performed by MCL)
- pH by EPA Method 9045D (performed by MCL)
- X-Ray Diffraction by SOP MCL-7712 (performed by MCL)
- Scanning Electron Microscope with Energy Dispersive x-ray Spectrometry (SEM/EDS)
- Percent Moisture by American Society of Testing and Materials (ASTM) D 2216-90 (performed by MCL)
- Sequential extraction analysis

Extract (following sequential extraction) samples will be analyzed for the following:

- Total Organic Carbon by SM 5310C (performed by Pace-I)
- Radium-226 by EPA Method 903.1 (performed by Pace-P)
- Radium-228 by EPA Method 904 calculated following Sequential Extraction by MCL (performed by Pace-P)
- Total Radium following Sequential Extraction by MCL (performed by Pace-P)
- Isotopic Thorium (Th-230, Th-232) by HASL 300 (performed by Pace-P)
- Total Uranium calculated following Sequential Extraction (performed by MCL)
- Total metals (Barium calculated following Sequential Extraction Step 1, Calcium calculated following Sequential Extraction Step 1, Iron calculated following Sequential Extraction Step 1, Manganese calculated following Sequential Extraction Step 1, Magnesium, Potassium, and Sodium) by SW-846 Methods 6010 (performed by MCL)
- Carbonate by SM 2320E (performed by MCL)
- Sulfate calculated based on Sequential Extraction Step 1 (performed by MCL)
- pH by EPA Method 9045D (performed by MCL)

A summary of the analytes and analytical methods are listed in Tables 2-3a, 2-3b, and 2-3c.

Outside of the analytical methods noted above and in Tables 2-3a, 2-3b, and 2-3c, samples to be tested for fate and transport related parameters may be subject to the following procedures that will be performed by MCL (summarized in more detail in Attachment C-35):

- X-Ray Diffraction (XRD)
- Further sequential extraction analysis

- SEM/EDS
- CEC

The laboratories will implement the project-required SOPs (Attachment C). The laboratory SOPs are based on the promulgated versions of analytical methods and other laboratory-developed procedures. These SOPs provide sufficient details to evaluate quality of the analytical methods and are applicable to the data goals and sample media of this investigation. The documentation of appropriate method validation for the project target compounds is included in Attachment C of this QAPP, and includes the criteria for acceptance, rejection, and qualification of data.

Additionally, the laboratories will be requested to send preliminary data for initial review within the standard turn-around-time for the analytical method. Non-conformances or re-analyses will be addressed by the Trihydro QAD with the lab as soon as possible to meet quality assurance and holding time requirements.

Tables 2-3a, 2-3b, and 2-3c include a constituent list and current laboratory determined RLs and method detection limits (MDLs) for each analyte in addition to the sampling methods for groundwater and soil. Laboratory MDLs have been determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" as noted in the laboratories' QAMs (Attachments A-1, A-2, A-3, and A-4). The laboratories will attempt through the standardized analytical methods to achieve these RLs or MDLs. However, RLs and MDLs are highly dependent on specific sample matrix effects, and some analyte MDLs may exceed applicable screening levels or standards. In order to achieve the most useable results, Trihydro will work with Pace and MCL to achieve the lowest possible RL or MDL within the appropriate levels of precision and accuracy (as feasible).

4.3 QUALITY CONTROL

The quality assurance objectives provide quantitative and qualitative measures of the ability to produce high quality results through a properly designed sampling and analysis program. The objectives of the overall QA/QC program are to:

- Document procedures, including changes from the work plan protocol;
- Conduct sampling and analytical procedures according to sound scientific principles;
- Monitor the performance of the field sampling team and laboratory with a systematic audit program and provide for corrective action necessary to assure quality;
- Evaluate the quality of the analytical data through a system of quantitative and qualitative criteria; and
- Record and archive data and observations properly.

4.3.1 FIELD QUALITY CONTROL CHECKS

The level of quality control effort will be consistent with that required under SW-846. The number/frequency for each quality assurance sample type is summarized below:

- Blind Duplicate Samples: 1 blind duplicate per 10 groundwater and soil samples will be collected for every analysis collected.
- Equipment Blanks: 1 equipment blank per 10 groundwater and soil samples will be collected for every analysis collected using non-disposable equipment.
- Field Blanks: 1 field blank per 10 groundwater and soil samples will be collected for every analysis collected.
- Trip Blanks: 1 trip blank within the shipping container containing samples for VOCs in groundwater samples.
- Matrix Spike: 1 for every 20 samples for soil (total organic carbon only) and groundwater samples.
- Matrix Spike Duplicate: 1 for every 20 samples for soil (total organic carbon only) and groundwater samples.

If a blind duplicate fails the acceptance criteria, the laboratory will be contacted to evaluate the possible cause of the error. If duplicate samples do not meet the acceptance criteria (30% for groundwater and 50% for soils), the parent and duplicate sample are qualified with "J" flags to indicate an estimated value. If the RPD is greater than or equal to 100%, associated samples will be qualified with "J" flags for detections of that constituent or "UJ" for non-detections. When corrective action is taken because of field quality control checks, the effectiveness of the corrective action will be measured based on the rate of reoccurrence of failure. In some cases, qualification of the data may be sufficient for evaluation of the data. In order to minimize the chance of cross-contamination, field and equipment blanks will be stored and shipped separately from source area samples, to the extent practicable. Additionally, in some cases, the field crew may be required to return to the Site in order to meet completeness objectives.

4.3.2 LABORATORY QUALITY CONTROL CHECKS

Pace has quality control programs in place to ensure the reliability and validity of the analyses performed at the laboratory. Analytical procedures are documented in writing as SOPs and each SOP includes a quality control section that addresses the minimum quality control requirements for the analytical procedure. The internal quality control checks differ slightly for each individual procedure, but, in general, the quality control requirements include the following items:

- Holding Times and Preservation
- Instrument Tunes for Gas Chromatography/Mass Spectrometry (GC/MS) Analyses

- Initial and Continuing Calibrations
- System Performance Checks
- Internal Standard Areas for GC/MS Analyses
- Laboratory Blanks
- System Monitoring Compounds (i.e. Surrogates)
- LCS/LCSD (LCSDs will only be performed if necessary, as discussed further in Section 2.1.2)
- MS/MSD
- Field Duplicates
- Laboratory Duplicates

Data obtained will be properly recorded. The data packages will be sufficient to perform Tier II, Tier III, or Tier IV data validations (as specified in the WP (Trihydro 2019a)). The laboratory will re-analyze samples analyzed in nonconformance with the quality control criteria if required by the respective SOPs (Attachment C) and determined necessary by the laboratory personnel, if sufficient sample volume/mass is available. It is expected that sufficient volumes/mass of samples will be collected to allow for reanalysis, when necessary. Preservation requirements, sample volumes, holding times, and sample containers are contained in Table 2-2. If the quality control fails and data are not usable, the laboratory will contact Trihydro. Trihydro and the OU-3 Respondents will determine the next steps on a case-by-case basis.

MCL will perform on the soil analyses the following QC samples:

- Field blanks and washes
- Field Duplicates
- Lab Duplicates
- Method Blanks
- LCS/LCSD
- Initial Calibration per SOP
- Instrument Calibration Verifications and Continuing Calibration Verifications-Second Source

MCL data and reports will be at least reviewed at two levels including a 100% QA staff review. The reports will include all the data present in the EPA Level 3 or 4 package (as requested in the WP (Trihydro 2019a)).

4.4 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

This section describes the procedures for maintaining the accuracy of instruments and measuring equipment which will be used for conducting field tests and laboratory analyses. Instruments and equipment will be maintained in order to promote the collection of precise and accurate data and to allow the project to proceed on schedule.

4.4.1 FIELD EQUIPMENT MAINTENANCE

The cornerstones of the field preventative maintenance program are the checking and calibration of field instruments before they are shipped or carried to the field, and the provision for backup instruments and equipment. Equipment used for sampling will be identified by the project field manager or field task manager prior to mobilization. Each instrument will be checked and certified by the shipper, rental company, or Trihydro FTL prior to each field event. Routine maintenance will be conducted in accordance with the FSP (Trihydro 2019b) and specific instrumentation manuals. Routine calibration will minimize the potential for inaccurate field measurements.

4.4.2 LABORATORY INSTRUMENTS MAINTENANCE

A routine preventative maintenance program is conducted by Pace and MCL to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees regularly perform routine scheduled maintenance and repair of, or coordinate with the vendor for the repair of, laboratory instruments. Performed maintenance is documented in the laboratory's operating record. Laboratory instruments are maintained in accordance with manufacturer's specifications. Attachments A-1, A-2 and A-3 provide the maintenance protocols used by the laboratory to ensure proper operation of laboratory equipment. MCL maintenance protocol are presented in Attachment A-4.

4.5 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Equipment will be calibrated in accordance with the specific SOPs or manufacturer guidelines. Instrument calibration will be checked anytime unexpected or unexplained readings are obtained and the instrument will be re-calibrated, if necessary.

4.5.1 FIELD INSTRUMENT CALIBRATION

Routine calibration will minimize the potential for inaccurate field measurements. Field instruments will be calibrated in accordance with procedures included in the FSP (Trihydro 2019b).

4.5.2 LABORATORY INSTRUMENT CALIBRATION

For a description of the calibration procedures for a specific laboratory instrument, refer to the applicable SOPs in Attachment C of this QAPP. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria, and the conditions that will require recalibration. The laboratory shall maintain the following information within their records: instrument identification, date of calibration, analyst, calibration solutions run, and the samples associated with these calibrations.

4.6 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Equipment and supplies will be inspected prior to use. Faulty or defective supplies will be replaced to protect the integrity of the samples. Trihydro's company quality program addresses the acceptance of supplies and consumables. Trihydro will track any non-conformance of supplies and consumables and note them in corresponding quality discussions in the RI report.

4.7 EVALUATION OF NON-MEASUREMENT SOURCES

Data acquired from non-measurement sources, such as computer databases, spreadsheets, programs, and literature files, will be presented with references and guidance on understanding the application of the non-direct sources. Historic data quality will be assessed using methods described in Section 3.0.

4.8 DATA MANAGEMENT

Both field and laboratory data shall be collected as part of this project. Overall project data quality will be managed through a system of review extending from field and laboratory through the data reduction and reporting process. The Trihydro QAD or delegate will review the data entered into the project database and check that no encoding errors were made during transfer from field or laboratory data sheets. Other data analysis elements include the evaluation of data through storage and retention of data. Electronic copies of relevant data will be retained by Trihydro Corporation through the duration of the project. Electronic copies (electronic scans of reports) of the data will also be retained by the Pace PMs. MCL does not produce Electronic Data Deliverables; however, completed laboratory report summaries will be retained by the PM.

Once sampling and laboratory analyses are completed, the Trihydro QAD (or designee) will complete an initial Tier I data evaluation/tracking form where general laboratory and field requirements are checked. The results of the form are stored in a Trihydro-managed database and a request is sent to appropriate personnel for completion of a Tier II, Tier III, or Tier IV data validation. As part of the Tier I process, the electronic data files (electronic data deliverables are not available for MCL data. Trihydro will produce these electronic files of the data for the database from electronic scans of the reports.) from the laboratory are uploaded into the Trihydro-managed database. Following completion of the data validation process, the final laboratory flags are submitted by a Trihydro data validation team member and entered into the Project Direct database by the Trihydro QAD (or designee). Data collected will be maintained on a secure electronic network at the Trihydro office in Laramie, Wyoming. The electronic network is backed up to a cloud database daily. Field and laboratory data management will be completed as described in the following sections.

4.8.1 FIELD DATA MANAGEMENT PROCEDURES

The field data will include field observations, field parameter measurements, and health and safety data. Data and observations will be recorded in the field logbook or field forms. These forms and field books will be scanned into electronic format and kept with the project files for reference during data evaluation. They will also be provided in the FSP (Trihydro 2019b).

4.8.2 LABORATORY DATA MANAGEMENT PROCEDURES

Laboratory data management procedures will be performed according to the following protocol. Raw analytical data will be recorded in numerically identified laboratory notebooks (referenced by the laboratory as logbooks, analytical prep sheets, or similar) or in the laboratory information management system (LIMS). Data will be recorded in this notebook, laboratory SOP, or LIMS along with other pertinent information, such as the sample identification number and the sample tag number. Other details, such as the analytical method used, name of analyst, date of analysis, sample matrix, reagent concentrations, instrument settings, and the raw data will also be recorded in the laboratory notebook, analytical prep sheets or LIMS. Each page of the notebook (if applicable) will be initialed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) (if applicable) will be maintained on file. Periodic review of these notebooks (if applicable) by the laboratories will take place prior to final data reporting. The Pace and MCL QAOs will maintain records of notebook (if applicable) entry inspections.

For this project, the equations that will be employed in reducing data are presented in the SOPs in Attachment C of this document. The formulae included in the SOPs make allowances for the effects of possible sample matrix interferences. Laboratories' QA Departments will perform two levels of review for each data set including an analyst and a second level reviewer trained to verify data. Unacceptable data shall be appropriately qualified in the project report. The QA

department will also review 10% of laboratory methods used on at least a quarterly basis, including a review of the raw data and data report for each reviewed method. Errors will be noted, and corrections made, but the original notations will be crossed out legibly, initialed and dated. Quality control data (e.g., laboratory duplicates, surrogates, MS/MSDs, LCS/LCSDs) will be compared to the historical limits unless a specific set of limits is set by the laboratory. Data considered acceptable will be entered into the LIMS and/or analytical reports (or similar). The data summary will be sent to the laboratories' QA Departments for review. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits, and any other anomalous conditions encountered during sample preparation and analysis. The Pace and MCL data package review departments are responsible for the review and assembly of each data package and they will ensure all sample and quality control data are included and accurate prior to issuance.

5.0 ASSESSMENT AND OVERSIGHT

The field and laboratory data collected during this investigation will be used to evaluate the extent of contamination. The QC results associated with each analytical parameter will be compared to the objectives presented in the SOPs included in Attachment C. Only data generated in association with QC results meeting these objectives will be considered reliable for decision-making purposes.

5.1 ASSESSMENTS AND RESPONSE ACTIONS

Performance and system audits will be completed to assess whether the appropriate personnel followed the QA and QC programs during field and laboratory activities. The Trihydro PM (or designee) will conduct internal field audits. The laboratories will conduct internal laboratory audits and the appropriate certification authorities may conduct external audits. Note that the members of the project team can stop work if an assessor in the field or laboratory observes that work is not in accordance with this QAPP, the FSP (Trihydro 2019b), the WP (Trihydro 2019a), or the Laboratory QAMs or SOPs. In this instance, the assessor will contact the project team promptly to communicate the issue and proposed corrective action.

5.1.1 FIELD AUDITS

The Trihydro PM may schedule audits of field activities. The evaluation is directed toward the extent to which the procedures in the work plan and this document are being followed. The Trihydro PM (or designee) will check to see that CoC procedures are being followed and that samples are being kept in custody at all times. Field documents pertaining to sample identification and control will be examined daily for completeness and accuracy by the Trihydro PM (or designee) to see that all entries are dated and signed, and the contents are legible, written in indelible material, and contain accurate and inclusive documentation of project activities. The Trihydro PM (or designee) will review field notebooks and field data forms. An example field-audit form is presented as Attachment D. If deficiencies are identified during the audit, the auditor will make a decision whether to repeat sample collection and analysis based on the extent of the deficiencies and their importance in the overall context of the project.

The external field audit is the responsibility of the EPA. External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the EPA. External field audits will be conducted according to the field activity information presented in this document. The external field audit process may include the assessment of (but not be limited to) the following:

- Sampling equipment decontamination procedures;
- Sample bottle preparation procedures;
- Sampling procedures;
- Examination of field sampling and safety plans;
- Sample vessel cleanliness and QA procedures;
- Procedures for verification of field duplicates;
- Procedures for the collection of filtered samples;
- Sample preservation and preparation for shipment; and
- Field screening practices.

5.1.2 LABORATORY AUDITS

The laboratories' QAOs will conduct the internal laboratory audits. The internal system audits will be done on at least an annual basis. The internal system audits will include an examination of laboratory documentation on sample receiving, sample login, sample storage, CoC procedures, sample preparation, sample analysis, instrument operating records, etc. The internal performance audits will be conducted as specified in the QAMs (Attachments A-1, A-2, A-3, and A-4). The performance audits may involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis. The Laboratory Quality Manager will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance. Laboratory audit procedures, criteria, and schedules are outlined in the QAMs located in Attachments A-1, A-2, A-3, and A-4.

An external audit may be conducted in association with certification of the laboratory. Failure of any or all audit procedures can lead to laboratory disqualification and the requirement that another suitable laboratory be chosen.

An external on-site review may consist of examination of the following items and procedures:

- Sample receipt procedures;
- Custody and sample security and login procedures;
- Sample tracking procedures;
- Instrument calibration records review;
- Instrument logs review;

- QA procedures review;
- Logbooks review;
- Sample preparation procedures;
- Sample storage procedures;
- Sample disposal procedures;
- Sample analytical SOP review;
- Field instrument review;
- Personnel interviews; and
- Glassware prep.

It is common practice when conducting an external laboratory audit to review one or more data packages from sample lots recently analyzed by the laboratory. This review would most likely include but not be limited to:

- Comparison of resulting data to the SOP or method, including coding for deviations;
- Verification of initial and continuing calibrations within control limits;
- Verification of surrogate recoveries and instrument timing results, where applicable;
- Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable;
- Review of recoveries from laboratory control sample analyses;
- Review of run logs with run times, ensuring proper order of analyses;
- Review of spike recoveries/QC sample data;
- Review of suspected manually integrated GC data and its cause (if applicable);
- Review of GC peak resolution for isolated compounds as compared to reference chromatograms (if applicable); and
- Assurance that samples were run within holding times.

Ideally, the data should be reviewed while on the premises, so that any data called into question can be discussed with the laboratory staff.

5.1.3 RESPONSE ACTIONS

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out of QC performance, which can affect data quality. Field team members may identify problems during sampling and laboratory analysts may identify problems during chemical analyses. Problems may be identified by the project managers and QAOs during the audit procedures. Corrective actions are described in the statements below.

Proposed and implemented laboratory corrective action will be documented in the regular QA reports to management. The Trihydro PM, or their designee, will only implement the proposed corrective action after approval from the OU-3 Respondents. If immediate corrective action is required, approvals secured by telephone from the Trihydro PM will be documented in an additional memorandum.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Trihydro PM, who in turn will notify the OU-3 Respondents. The OU-3 Respondents will be promptly notified from the time the problem was communicated to the Trihydro PM. If the problem is analytical in nature, information about the problem will be promptly communicated to the OU-3 Respondents. Implementation of corrective action will be confirmed in writing through the same channels. For problems that involve sampling that has not been done previously at a location, or for a new parameter, or for more conservative reporting limits, the corrective action will be determined based on the goals established in the specific work plan for that investigation. Note that the Trihydro PM has the ability to stop work due to a nonconformance issue.

Any nonconformance with the established QC procedures in this document will be identified and corrected in accordance with the QAPP. The Trihydro PM, or designee, will issue a nonconformance report for each nonconformance condition. The effectiveness of the applied corrective action will be measured based on internal audits and observations, which will be reported to the OU-3 Respondents.

5.1.3.1 FIELD CORRECTIVE ACTION

Corrective action in the field may be needed when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the QAPP, etc.), or if sampling procedures and/or field analytical procedures require modification, etc., due to unexpected conditions. It will be the responsibility of the Trihydro PM to ensure the corrective action has been implemented.

If the corrective action will supplement the existing sampling plan using existing and approved procedures in the QAPP, corrective action approved by the Trihydro PM will be documented. If corrective actions result in fewer samples (or analytical fractions), alternate locations, etc., which may cause project QA objectives not to be achieved, the OU-3 Respondents will be notified of the reason for the deviation.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The Trihydro PM (or designee) will identify deficiencies and recommend corrective action. The field team will implement the corrective actions. Corrective actions will be documented in the corresponding progress report.

Corrective actions will also be implemented and documented in the field records. Staff members will not initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, the Trihydro PM may stop work. If at any time a corrective action issue is identified which directly affects project objectives, the OU-3 Respondents will be notified immediately.

5.1.3.2 LABORATORY CORRECTIVE ACTION

In general, the inability to achieve the QA objectives discussed in this QAPP may result in laboratory corrective actions. A detailed description of laboratory responses to correct these deficiencies is presented in the laboratory SOPs. If the laboratory cannot correct the deficiencies, they will be handled in one of three ways:

- The laboratory will be asked to reanalyze the analyses in question, if sample holding times have not been exceeded. Otherwise, the laboratory may be asked to re-quantify relevant peaks in the chromatograms or reprocess other instrumental output, when applicable.
- Trihydro will demonstrate that the noncompliance does not compromise the successful achievement of the WP objectives (Trihydro 2019a).
- Additional samples will be collected and analyzed to eliminate the non-compliance.

The Trihydro QAD may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include re-sampling by the field team or re-injection/re-analysis of samples by the laboratory. These actions are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded, etc.). If the Trihydro QAD identifies a corrective action situation during data assessment, it is the Trihydro PM, OU-3 Respondents, and the EPA who will be responsible for approving the implementation of corrective action, including re-

sampling. The Trihydro QAD will document all corrective actions of this type. Laboratory noncompliance and corrective actions will be discussed in the subsequent progress reports.

5.2 REPORTS TO MANAGEMENT

Both field and laboratory data will be reported to Trihydro management? OU-3 respondents? as part of this project. Data reporting procedures shall be carried out for both field and laboratory operations, as described below.

5.2.1 FIELD DATA REPORTING

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities. Additionally, a separate QA section of the RI/FS report will be used to convey data usability, bias, results of the assessments, changes to the QAPP (if necessary), major personnel changes, corrective actions performed, and any other relevant QA information. Reports to management shall be completed by the Trihydro PM (or designee) and submitted to the OU-3 Respondents.

5.2.2 LABORATORY DATA REPORTING

The task of reporting laboratory data begins after the appropriate internal laboratory QA review has been concluded. The communication/notification, reporting requirements, and analyses requirements are described in greater detail in Trihydro's Tier I and Tier II Laboratory Performance Guidelines and Tier III and Tier IV Laboratory Performance Guidelines in Attachments E-1 and E-2, respectively. Standard turnaround times will be met by the laboratories unless otherwise requested. However, it should be noted that there may be a variation in the turnaround time for radiochemistry data and level IV data packages as they are more complex than the standard analytical suite and take longer to produce. Requirements may vary due to the analytical procedure requirements. These variations will be discussed with the Trihydro QAD prior to sample collection.

Any program of environmental measurement can produce outlier results that are outside the "expected" range of values. Outlier values may be the result of:

- A catastrophic unnatural (but real) occurrence, such as a spill;
- Inconsistent sampling or analytical chemistry methodology;
- Variation in field conditions (e.g., if construction work is being conducted near the Site);
- Errors in the transcription of data values or decimal points; or
- True but extreme variability in concentration measurements.

Documentation and validation of the cause of outliers will accompany any attempt to correct or delete data values, because valid but extreme values will not be altered. Outlier values will not be omitted from the raw data reported to the EPA and MDNR but will be identified as outliers within the data summary tables. Reasons for the outlying behavior will be provided in the data summary tables or in the Tier II, Tier III, or Tier IV data validation reports.

Data below detection limits will be expressed as determined by individual Method SOPs and each laboratories' QAMs. If possible (as determined by the laboratory SOP or QAM), the data will be flagged with a "J" when detected between the RL and MDL. Data above the detection limit will be expressed in units of micrograms per Liter (µg/L) or milligrams per Liter (mg/L) for groundwater or milligrams per kilogram (mg/kg) dry weight for soil. Solid radiochemistry results will be reported in units of average picocuries per gram (pCi/g). Water radiochemistry results will be reported in units of average picocuries per Liter (pCi/L).

The deliverables associated with the tasks identified in the WP will contain data quality information collected during the task. Those reports will be the responsibility of the respective laboratories' Project Manager or designee and will include the QC summary for the accuracy, precision, and completeness of the data, as well as the results of the performance and system audits, and any corrective action needed or taken during the project. The laboratory data are reported through the LIMS. A copy of the laboratory data report will be included in the reports to the OU-3 Respondents.

6.0 DATA VALIDATION AND USABILITY

Data generated through field activities or by the laboratory shall be reduced and validated prior to reporting. Data shall be disseminated by the laboratory and the Trihydro QAD after it has been subjected to data validation. This section covers procedures to compile, validate, and report the data collected during the groundwater and soil investigations.

6.1 DATA REVIEW, VERIFICATION, AND VALIDATION

The process of data validation is the examination of objective evidence that the requirements of the specified quality control acceptance criteria are met. Data validation procedures shall be performed for both field and laboratory operations, as described below. Data will be validated in accordance with the Trihydro data validation process.

6.1.1 FIELD DATA

The procedures to evaluate field data for this investigation include checking for transcription errors and review of field logbooks, on the part of the field team. The Trihydro FTL (or designee) will review the field notes after completion of sampling. The objectives of this review are to identify and correct errors in the field notes. The Trihydro QAD will review the field audit and field notes and determine whether the samples were collected and handled according to this QAPP.

6.1.2 LABORATORY DATA

Trihydro will perform data validation review on data received from the laboratory. The data validation will include Tier I, and Tier II, Tier III, or Tier IV data validation reviews as described in Sections 6.1.2.1, 6.1.2.2, and 6.1.2.3, respectively (MCL data will only require Tier I data validation). As described in Section 6.1.2.4, data qualifiers will be applied to the data based on the data validation review. These qualifiers will be maintained in the database with each data point. Organic data will be evaluated in accordance with the general validation criteria set forth in the EPA CLP National Functional Guidelines for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017 (EPA 2017a) with additional reference to EPA CLP National Functional Guidelines for Organic Data Review (EPA 1999), document number EPA 540/R-99/008 of October 1999 (EPA 1999). Data from inorganic analyses will be evaluated according to validation criteria set forth in the EPA CLP National Functional Guidelines for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017 (EPA 2017b), with additional reference to the EPA CLP National Functional Guidelines for Inorganic Data Review, document number EPA 540-R-04-004, October 2004 (EPA 2004). Review of duplicates will be conducted in accordance with EPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures (EPA 2013). MCL doesn't produce the EPA CLP forms, but

will submit the data and backup information developed in the analyses. Holding times shall be reviewed (in accordance with Table 2-2), and instrument performance check sample results shall be evaluated by trained reviewers independent of the laboratory. Data validation procedures will also adhere to the practices described in this QAPP. Each analytical data report will be validated by QAD (or qualified designee).

The data validator will also evaluate the overall completeness of the data package. Completeness checks will be administered on data to evaluate whether deliverables specified in the QAPP are present. The following sections describe data validation procedures in greater detail.

6.1.2.1 TIER I DATA VALIDATION

In addition to the field data validation procedures, the Tier I data validation is performed to verify and document that samples in the data set were analyzed according to the project requirements and that the laboratory analytical report is complete. An electronic Tier I validation checklist will be prepared in an electronic format for each laboratory analytical sample group. Tier I validations will be performed by a competent person with knowledge of the project requirements. The Tier I validation will include a review of the following elements:

- Review of the cover letter signed by the Laboratory PM or designee.
- Review of the case narrative discussing any technical problems or deviations from the analytical methods including if the laboratory received the samples in good condition. Samples are considered in good condition if the samples are at the proper temperature (4 degrees Centigrade [°C] ± 2°C) and sample receipt condition is acceptable (i.e., the bottles are not broken, and the cooler custody seals are intact).
- Review of date and time of receipt.
- Review of CoC forms to verify that samples were maintained under strict CoC with signatures from the field personnel and the lab personnel.
- Comparison of sampling dates to sample extraction dates and analysis dates to check that samples were extracted and/or analyzed within proper holding times.
- Review of target constituent list, analytical methods, and detection limits to verify conformance with the WP.
- Review of lab validation summary/chronicle describing client ID/analysis, laboratory identification number, prep number, collection date, extraction/prep date, analysis date, and analytical section manager sign off.
- Review of sample data report including the results listed in alphabetical order (or by analytical method) with sample preparation, extraction, cleanup, digestion, and analytical methods, analysis date, extraction date, analyst initials, and qualifiers included.

- Review of quality control summary report including date of analyses, parameters determined, system monitoring compound summary, method blank data, sample duplicates and control samples, surrogate spike recoveries, and MS and MSD results.
- Review of additional performance criteria specific to analytical methods.
- Evaluation of corrective actions that may have been necessary and possible data quality assessment items.

6.1.2.2 TIER II DATA VALIDATION

In addition to the Tier I validation requirements, the Tier II evaluation will include a review of the basic laboratory quality control data. A detailed data validation report, as shown in Attachment F-1, which provides sufficient detail to explain data qualifiers and data inadequacies, is produced by the reviewer. The Tier II data validation process provides sufficient detail for the data user to have an accurate idea of the data quality and reliability, and an understanding of how well the project objectives were met. The Tier II data validation is performed by a chemist or other trained scientist who is familiar with contract laboratory procedures. The Tier II data validation will include a review of Tier I elements as well as the following criteria:

- Review of field and laboratory blanks to evaluate possible contamination sources; consideration should be given to preparation techniques and frequencies, as well as the analytical results.
- Review of field duplicate data for evaluation of field and laboratory precision.
- Review of laboratory quality assurance data (MS/MSD recoveries and RPD calculations, surrogate spike recoveries, LCS/LCSD recoveries and RPD calculations) for compliance with method or project required acceptance criteria.
- Review of the analytical results to verify compliance with the specified project goals.
- Review of additional method specific performance criteria, as appropriate, if provided by the laboratory.

The following criteria will be evaluated during the Tier I and II data validation process:

- Chain-of-Custody: Is the COC complete and were the analytical method(s) specified?
- Sample Check in Conditions: Did the samples arrive at the correct temperature and with the correct container count? Were the sample labels complete and was integrity of the samples and the container maintained? Were the samples received properly preserved?
- Holding Times: Were the samples extracted/digested within the method specified holding times? Were the samples analyzed within the method specified holding time?

- Dilutions/Method Reporting Limits: Were any samples diluted to an extent that the resulting reporting limits were raised to a degree which would render the associated data points unsuitable for the projects data quality objectives? Were the dilutions necessary and unavoidable? Is re-analysis of the sample extract possible or feasible?
- Laboratory Control Samples/Laboratory Control Sample Duplicates (LCS/LCSD; Second Source Standards): Was the LCS/LCSD compound list complete and were required analytes contained in the spike solution? Was the LCS/LCSD performance within the method specified limits for each compound?
- Matrix Spike/Matrix Spike Duplicate Recovery: Was the specified sample from this project sample set used as the MS/MSD parent sample? Was the MS/MSD compound list complete and were required analytes contained in the spike solution? Were the MS/MSD recovery values within the method specified limits for each compound? The degree of matrix interferences in a sample can vary significantly, even within a sample set collected from the same site. Therefore, data qualifications will be assigned based on an evaluation of associated quality control data and the professional judgment of the reviewer.
- Duplicate Sample Repeatability (Field and Laboratory Duplicate Samples): Field duplicate RPD limits for groundwater are set at 0-30% and for soil are set at 0-50%, and laboratory RPD limits reference published or method specified limits. In cases where a compound is detected at concentrations less than five times the detection limit, the precision goals will not apply in accordance with EPA data validation guidelines. Repeatability (precision) failures will be "J" flagged. Duplicate samples and evaluation of field precision will be assessed on a case-by-case basis. The parent sample and duplicate sample may be flagged based on the results of the validation. Field duplicate samples will be evaluated in the overall quality of the associated data set.
- Surrogate Recoveries: Surrogate compound recoveries are expected to be within the method or laboratory specified acceptance limits.

6.1.2.3 TIER III AND TIER IV DATA VALIDATION

A detailed data validation report, as shown in Attachment F-2, which provides sufficient detail to explain data qualifiers and data inadequacies, is produced by the reviewer. A Tier IV data validation will include a review of the raw analytical data, which is examined in detail to check for correctness of concentration calculations, compound identification and anomalies in the data. A detailed data validation report, provides sufficient detail to explain data qualifiers and data inadequacies, is produced by the reviewer. The Tier III and IV data validation processes provide sufficient detail for the data user to have an accurate idea of the data quality and reliability, and an understanding of how well the project objectives were met. The Tier III and IV data validations that the data are adequately assessed, to allow their use in formal legal proceedings, risk assessments, and closures. Tier IV data validation (specifically) ensures that the data are adequately assessed, to allow its use in formal legal proceedings, risk assessments, and

closures. Tier III and IV data validations are performed by a chemist or other trained scientist who is familiar with contract laboratory procedures. The Tier III and IV data validation will include a review of Tier I elements as well as some or all of the following criteria:

- Review of field and laboratory blanks to evaluate possible contamination sources; consideration should be given to preparation techniques and frequencies, as well as the analytical results.
- Review of field duplicate data for evaluation of field and laboratory precision.
- Review of laboratory quality assurance data (MS/MSD recoveries and RPD calculations, surrogate spike recoveries, LCS/LCSD recoveries and RPD calculations) for compliance with method or project required acceptance criteria.
- Review of the analytical results to verify compliance with the specified project goals.
- Review of laboratory summary of tuning and calibration checks.
- Review of quality control packages and sample raw data and calculations (the raw data and calculations are reviewed specifically with Tier IV data validation).
- Review of serial dilutions (if applicable to the method requirements).
- Limited review of chromatograms.
- Review of initial and continuing calibration results (may have been conducted in a Tier II but required for a Tier III).
- Review of instrument performance results (if applicable to the method requirements).
- Review of internal standard results (if applicable to the method requirements).
- Review of ICP interference check sample results (if applicable to the method requirements).
- Review of method detection limit verifications.
- Review of instrument and calibrations performance summaries (if provided).
- Review of additional method specific performance criteria, as appropriate, if provided by the laboratory.

The following criteria will be evaluated during the Tier III/IV data validation process:

- Chain-of-Custody: Is the COC complete and were the analytical method(s) specified?
- Sample Check in Conditions: Did the samples arrive at the correct temperature and with the correct container count? Were the sample labels complete and was integrity of the samples and the container maintained?

- Holding Times: Were the samples extracted within the method specified holding times? Were the samples analyzed within the method specified holding time?
- Dilutions/Method Reporting Limits: Were any samples diluted to an extent that the resulting reporting limits were raised to a degree, which would render the associated data points unsuitable for the projects data quality objectives? Were the dilutions necessary and unavoidable? Is re-analysis of the sample extract possible or feasible? Were the same project quantitation limits used for each sampling event? If possible, from the laboratory, did the laboratory "J" flag detected results between the reporting limit and method detection limit?
- Laboratory Control Samples/Laboratory Control Sample Duplicates (LCS/LCSD; Second Source Standards): Was the LCS/LCSD compound list complete and were all required analytes contained in the spike solution? Was the LCS/LCSD performance within the method specified limits for each compound?
- Matrix Spike/Matrix Spike Duplicate Recovery: Was the specified sample from this project sample set used as the MS/MSD parent sample? Was the MS/MSD compound list complete and were all required chemicals contained in the spike solution? Were the MS/MSD recovery values within the method specified limits for each compound? The degree of matrix interferences in a sample can vary significantly, even within a sample set collected from the same site. Therefore, data qualifications will be assigned based on an evaluation of all associated quality control data and the professional judgment of the reviewer.
- Duplicate Sample Repeatability (Field and Laboratory Duplicate Samples): Field duplicate RPD limits for groundwater are set at 0-30% and for soil are set at 0-50%, and laboratory RPD limits reference published or method specified limits. In cases where an analyte is detected at concentrations less than five times the detection limit, the precision goals will not apply in accordance with EPA data validation guidelines. Repeatability (precision) failures will be "J" flagged. Duplicate samples and evaluation of field precision will be assessed on a case-by-case basis. The parent sample and duplicate sample may be flagged based on the results of the validation. Field duplicate samples will be evaluated in the overall quality of the associated data set.
- Surrogate Recoveries: Surrogate compound recoveries are expected to be within the method or laboratory specified acceptance limits.
- Internal Standards and Retention Time Windows (if available): The data sets will be required to fully meet the method specified requirements for these criteria.
- Initial and Continuing Calibration Verifications (ICV and CCV; if available): ICV and CCVs will be checked to confirm that they met the method specified limits for accuracy and periodicity. If an ICV and/or CCV failure is noted, the data validator will document that samples analyzed prior to the ICV and/or CCV failure were re-analyzed after the instrument was re-calibrated.

- Instrument Performance Checks (if available): The data validator will confirm that the method specified instrument performance checks were run and met the method requirements.

6.1.2.4 DATA VALIDATION QUALIFIERS

The data quality flags used to qualify analytical data will be similar to those outlined within the EPA Data Validation Functional Guidelines for Evaluating Environmental Analyses and Attachment B. The most commonly used data quality flags include:

- R Code: An "R" flag indicates data have not met the required analytical quality assurance requirements. These data are unusable even if field quality control requirements have been fulfilled. Based on the EPA CLP National Functional Guidelines, data may be rejected for several reasons which may include:
  - Analyses of samples outside of holding times. For samples analyzed outside of the holding times, the results may be accepted, qualified as estimated, or rejected based on professional judgement and the specific method(s) affected.
  - For samples received at a temperature above 6°C, the results may be accepted, qualified as estimated, or rejected based on professional judgement and the specific analyte(s) affected.
  - Samples that do not have proper preservation.
  - Gross evidence of cross contamination from method, field, equipment, or trip blank results.
  - Low, out of range surrogate recoveries for non-detect data or several out of range surrogate recoveries for both detects and non-detects.
  - Low LCS/LCSD recoveries for non-detect data.
  - Extremely low MS/MSD recoveries for non-detected data.
  - Other reasons for rejection may be found in the EPA CLP National Functional Guidelines.
- J Code: A "J" flag indicates that data have not met some of the analytical quality assurance requirements; however, the problem was not of sufficient magnitude to warrant classifying the data as unusable. Data in this category are qualitative (estimated) provided the field data meet all quality control requirements.
- J+ Code: A "J+" flag indicates that data have not met some of the analytical quality assurance requirements, with an indication of potential high bias based on a QC recovery above the upper control limit. The inaccuracy was not of sufficient magnitude to warrant classifying the data as unusable. Data in this category are estimated and may be biased high.

- J- Code: A "J-" flag indicates that data have not met some of the analytical quality assurance requirements, with evidence of potential low bias based on a QC recovery below the lower control limit. The inaccuracy was not of sufficient magnitude to warrant classifying the data as unusable. Data in this category are estimated and may be biased low.
- UJ Code: The material was analyzed for but was not detected. The sample detection limit is an estimated value.
- JB Code: A "JB" flag indicates that the result could be attributed to cross contamination. Specifically, this flag will be applied if the result for a field contaminant is within 10 times a field, equipment, trip, or method blank detected result.
- U Code: A "U" code indicates that the result was detected but, due to cross contamination, was determined to be undetected by the validator. The original values and the revised undetected result will be clearly noted on the report tables.

6.1.2.5 DATA DEFICIENCIES

The data set will be reviewed for conformance to the method-specified recovery or repeatability values for each individual constituent in each required quality control analysis. Data points that are associated with procedural or analytical irregularities will be evaluated according to the following protocol:

- Minor deficiencies: Deficiencies which are determined to have no significant effect on the accuracy of the data will be regarded as minor deficiencies. These occurrences will be noted and explained in the data validation report but will not affect the usability of the data points and the data will not be qualified.
- Significant deficiencies: Significant deficiencies are serious enough to call the veracity of a given data point(s) into question. In these cases, the deficiencies are judged to result in known or probable variation from the normal analytical method performance standards, with relation to the precision and/or accuracy of the data point. Subject data points will be qualified with the appropriate qualifiers per EPA data validation guidelines.
- Major deficiencies: Irregularities in the sample handling or analytical process which compromise the analytical result(s) to such an extent that the data are deemed unusable or unreliable. Such data points will typically be rejected, and the reason(s) will be explained in the data validation report on a sample-by-sample basis.

Quality control data will be discussed in detail in a quality assurance section of the RI report. Quality assurance information will be included in other chapters to the extent that it affects the interpretation of sample data.

6.2 RECONCILIATION WITH USER REQUIREMENTS

The data will be reconciled with this QAPP and the data quality objectives DQOs (described in the WP) to evaluate the data usability, including a comparison with the media-specific screening values, an evaluation of whether additional data gaps exist, and an assessment of the need for further remedial investigation or action.

Results of the data validation process and DQO assessment will be reported to the EPA with the RI/FS report. This process is documented in a Tier II, Tier III, or Tier IV Data Validation. Summary tables documenting analytical data will be denoted with any flags resulting from the Trihydro data validation process, in addition to the laboratory data qualifier flags. For example, samples that are rejected as part of the data validation process ("R" flag) would not meet the DQOs for the site.

As stated above, nonconformance with the quality assurance objectives will result in corrective action and will be reported to the OU-3 Respondents. The data review will include an evaluation of the precision, accuracy, representativeness, comparability, and completeness according to the limits specified with the laboratory reports.

7.0 REFERENCES

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TABLES

TABLE 2-1. LABORATORY CONTROL SAMPLES
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Lab	Analysis Group	Method Description	Method Code	Analyte Description	CAS Number	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %
Pace	Water	Metals	EPA 6020	Antimony	7440-36-0	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6020	Arsenic	7440-38-2	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Barium	7740-39-3	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6020	Beryllium	7440-41-7	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Boron	7440-42-8	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6020	Cadmium	7440-43-9	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Calcium	7440-70-2	80	120	NA	75	125	20
Pace	Water	Chemical Oxygen Demand	EPA 410.4	Chemical Oxygen Demand	NA	90	110	NA	90	110	20
Pace	Water	Chloride	EPA 9056	Chloride	16887-00-6	90	110	NA	80	120	15
Pace	Water	Ferrous Iron	SM 3500 Fe B	Ferrous Iron	15438-31-0	±15% of the true value	±15% of the true value	2000%	±20% of the true value	±20% of the true value	20
Pace	Water	Metals	EPA 6020	Chromium	7440-47-3	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Cobalt	7440-48-4	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6020	Copper	7440-50-8	80	120	NA	75	125	20
Pace	Water	Fluoride	EPA 9056	Fluoride	16984-48-8	90	110	NA	80	120	15
Pace	Water	Metals	EPA 6010	Iron	7439-89-6	80	120	NA	75	120	20
Pace	Water	RadChem	HASL 300	Isotopic Thorium	NA	75	125	NA	75	125	25
Pace	Water	RadChem	HASL 300	Isotopic Uranium	NA	75	125	NA	75	125	25
Pace	Water	Metals	EPA 6020	Lead	7439-92-1	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Magnesium	7439-95-4	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Manganese	7439-96-5	80	120	NA	75	125	20
Pace	Water	Metals	EPA 7470	Mercury	7439-97-6	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Nickel	7440-02-0	80	120	NA	75	125	20
Pace	Water	Nitrogen	EPA 353.2	Nitrogen	NA	90	110	NA	90	110	20
Pace	Water	Ammonia as Nitrogen	SM 4500-NH3 G	Ammonia as Nitrogen	7664-41-7	90	110	NA	90	110	20
Pace	Water	Nitrite as Nitrogen	EPA 353.2	Nitrite as Nitrogen	14797-65-0	90	110	NA	90	110	20
Pace	Water	Nitrate as Nitrogen	EPA 353.2	Nitrate as Nitrogen	14797-55-8	90	110	NA	90	110	20
Pace	Water	PCB	EPA 8082	PCB-1221(Aroclor 1221)	11104-28-2	NA	NA	NA	NA	NA	NA
Pace	Water	Phosphorus	EPA 365.1	Phosphorus	7723-14-0	90	110	NA	90	110	20
Pace	Water	RadChem	EPA 903.1	Radium-226	NA	73	135	NA	71	136	32
Pace	Water	RadChem	EPA 904.0	Radium-228	NA	60	135	NA	60	135	36
Pace	Water	RadChem	Calculated from Ra-226 and Ra-228	Total Radium	NA	NA	NA	NA	NA	NA	+/- 25%
Pace	Water	Metals	EPA 6020	Selenium	7782-49-2	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6020	Silver	7440-22-4	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Sodium	NA	80	120	NA	75	125	20
Pace	Water	Sulfate	EPA 9056	Sulfate	14808-79-8	90	110	NA	80	120	15
Pace	Water	Sulfide	SM 4500-S2-D	Sulfide	18496-25-8	90	110	NA	90	110	20
Pace	Water	Metals	EPA 6020	Thallium	7440-28-0	80	120	NA	75	125	20
Pace	Water	TDS	2540C	Total Dissolved Solids	NA	80	120	NA	N/A	N/A	10
Pace	Water	Total Hardness	EPA 2340B	Total Hardness	NA	NA	NA	NA	NA	NA	20
Pace	Water	TOC	SM 5310C	Total Organic Carbon	7440-44-0	90	110	NA	80	120	20
Pace	Water	Metals	EPA 6020	Vanadium	7440-62-2	80	120	NA	75	125	20
Pace	Water	Metals	EPA 6010	Zinc	7440-66-6	80	120	NA	75	125	20
Pace	Water	VOCs	8260C	Acetone	67-64-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Acrolein	107-02-8	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Acrylonitrile	107-13-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Benzene	71-43-2	78	117	NA	50	135	20
Pace	Water	VOCs	8260C	Bromobenzene	108-86-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Bromodichloromethane	75-27-4	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Bromoform	75-25-2	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Bromomethane (Methyl Bromide)	74-83-9	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Bromochloromethane	74-97-5	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	2-Butanone (MEK)	78-93-3	NA	NA	NA	NA	NA	NA

TABLE 2-1. LABORATORY CONTROL SAMPLES
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Lab	Analysis Group	Method Description	Method Code	Analyte Description	CAS Number	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %
Pace	Water	VOCs	8260C	*n*-Butylbenzene	104-51-8	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	*sec*-Butylbenzene	135-98-8	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	*tert*-Butylbenzene	98-06-6	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Carbon disulfide	75-15-0	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Carbon tetrachloride	56-23-5	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Chlorobenzene	108-90-7	79	112	NA	39	135	20
Pace	Water	VOCs	8260C	Chloroethane (Ethyl Chloride)	75-00-3	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Chloroform	67-66-3	76	118	NA	52	134	20
Pace	Water	VOCs	8260C	Chloromethane (Methyl Chloride)	74-87-3	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	2-Chlorotoluene	95-49-8	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	4-Chlorotoluene	106-43-4	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Cyclohexane	110-82-7	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Dibromochloromethane	124-48-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,2-Dibromoethane (EDB)	106-93-4	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Dibromomethane (Methylene Bromide)	74-95-3	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	*trans*-1,4-Dichloro-2-butene	110-57-6	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,2-Dichlorobenzene	95-50-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,3-Dichlorobenzene	541-73-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,4-Dichlorobenzene	106-46-7	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Dichlorodifluoromethane	75-71-8	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,1-Dichloroethane (DCA)	75-34-3	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,2-Dichloroethane (EDC)	107-06-2	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,1-Dichloroethene	75-35-4	72	123	NA	51	138	20
Pace	Water	VOCs	8260C	*cis*-1,2-Dichloroethene	156-59-2	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	*trans*-1,2-Dichloroethene	156-60-5	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,2-Dichloropropane	78-87-5	75	124	NA	54	135	20
Pace	Water	VOCs	8260C	1,3-Dichloropropane	142-28-9	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	2,2-Dichloropropane	594-20-7	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,1-Dichloropropene	563-58-6	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	*cis*-1,3-Dichloropropene	10061-01-5	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	*trans*-1,3-Dichloropropene	10061-02-6	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,4-Dioxane (p-Dioxane)	123-91-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Ethylbenzene	100-41-4	80	118	NA	31	147	20
Pace	Water	VOCs	8260C	Ethyl methacrylate	97-63-2	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Hexachloro-1,3-butadiene	87-68-3	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	n-Hexane	110-54-3	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	2-Hexanone	591-78-6	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Iodomethane	74-88-4	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Isopropylbenzene (Cumene)	98-82-8	81	117	NA	25	151	20
Pace	Water	VOCs	8260C	p-Isopropyltoluene	99-87-6	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Methyl Acetate	79-20-9	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Methylcyclohexane	108-87-2	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Methylene Chloride (Dichloromethane)	75-09-2	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1-Methylnaphthalene	90-12-0	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	2-Methylnaphthalene	91-57-6	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	4-Methyl-2-pentanone (MIBK)	108-10-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Methyl-tert-butyl-Ether (MTBE)	1634-04-4	71	124	NA	51	142	20
Pace	Water	VOCs	8260C	Naphthalene	91-20-3	67	126	NA	40	135	20
Pace	Water	VOCs	8260C	*n*-Propylbenzene	103-65-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Styrene	100-42-5	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,1,1,2-Tetrachloroethane	630-20-6	NA	NA	NA	NA	NA	NA

TABLE 2-1. LABORATORY CONTROL SAMPLES
WEST LAKE LANDFILL OU-3
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Lab	Analysis Group	Method Description	Method Code	Analyte Description	CAS Number	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %
Pace	Water	VOCs	8260C	1,1,2,2-Tetrachloroethane	79-34-5	73	117	NA	52	131	20
Pace	Water	VOCs	8260C	Tetrachloroethene (PCE)	127-18-4	76	116	NA	34	140	20
Pace	Water	VOCs	8260C	Toluene	108-88-3	77	115	NA	43	134	20
Pace	Water	VOCs	8260C	1,2,3-Trichlorobenzene	87-61-6	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,2,4-Trichlorobenzene	120-82-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,1,1-Trichloroethane (TCA)	71-55-6	74	126	NA	50	141	20
Pace	Water	VOCs	8260C	1,1,2-Trichloroethane	79-00-5	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,1,2-Trichlorotrifluoroethane*	76-13-1	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Trichloroethene (TCE)	79-01-6	76	120	NA	40	141	20
Pace	Water	VOCs	8260C	Trichlorofluoromethane	75-69-4	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,2,3-Trichloropropane	96-18-4	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	1,2,4-Trimethylbenzene	95-63-6	76	118	NA	19	148	20
Pace	Water	VOCs	8260C	1,3,5-Trimethylbenzene	108-67-8	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Vinyl Acetate	108-05-4	NA	NA	NA	NA	NA	NA
Pace	Water	VOCs	8260C	Vinyl Chloride (Chloroethene)	75-01-4	64	155	NA	46	164	20
Pace	Water	VOCs	8260C	Xylenes, Total	1330-20-7	78	118	NA	29	145	20
Pace	Water	VOCs	8260C	4-Bromofluorobenzene (surr)	460-00-4	85	111	NA	85	111	NA
Pace	Water	VOCs	8260C	Dibromofluoromethane (surr)	1868-53-7	89	116	NA	89	116	NA
Pace	Water	VOCs	8260C	Toluene-d8 (surr)	2037-26-5	87	110	NA	87	110	NA
Pace	Water	SVOCs	8270C	Acenaphthene	83-32-9	42	124	NA	38	124	20
Pace	Water	SVOCs	8270C	Acenaphthylene	208-96-8	41	128	NA	37	128	20
Pace	Water	SVOCs	8270C	Acetophenone	98-86-2	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Anthracene	120-12-7	66	131	NA	71	122	20
Pace	Water	SVOCs	8270C	Atrazine	1912-24-9	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Benzaldehyde	100-52-7	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Benz[a]anthracene	56-55-3	67	136	NA	75	123	20
Pace	Water	SVOCs	8270C	Benzo[a]pyrene	50-32-8	47	143	NA	56	120	20
Pace	Water	SVOCs	8270C	Benzo[b]fluoranthene	205-99-2	51	139	NA	56	123	20
Pace	Water	SVOCs	8270C	Benzo[g,h,i]perylene	191-24-2	47	143	NA	60	119	20
Pace	Water	SVOCs	8270C	Benzo[k]fluoranthene	207-08-9	49	142	NA	56	121	20
Pace	Water	SVOCs	8270C	Benzyl alcohol	100-51-6	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Biphenyl (1,1 - biphenyl or Diphenyl)	92-52-4	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	bis(2-chloroethoxy) methane	111-91-1	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	bis(2-chloroethyl) ether	111-44-4	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	bis(2-chloro-1-methylethyl) ether*	108-60-1	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	bis(2-ethylhexyl) phthalate	117-81-7	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	4-Bromophenyl phenyl ether	101-55-03	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Butyl benzyl phthalate	85-68-7	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	4-Chloroaniline	106-47-8	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	4-Chloro-3-methylphenol (p-chloro-m-Cresol)	59-50-7	27	134	NA	29	118	20
Pace	Water	SVOCs	8270C	2-Chloronaphthalene	91-58-7	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2-Chlorophenol	95-57-8	21	109	NA	22	95	20
Pace	Water	SVOCs	8270C	4-Chlorophenyl phenyl ether	7005-72-3	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Caprolactam	105-60-2	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Carbazole	86-74-8	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Chrysene	218-01-9	67	134	NA	74	120	20
Pace	Water	SVOCs	8270C	Dibenz[a,h]anthracene	53-70-3	48	145	NA	57	125	20
Pace	Water	SVOCs	8270C	Dibenzofuran	132-64-9	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Di-*n*-butyl phthalate	84-74-2	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	3,3'-Dichlorobenzidine	91-94-1	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2,4-Dichlorophenol	120-83-2	NA	NA	NA	NA	NA	NA

TABLE 2-1. LABORATORY CONTROL SAMPLES
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Lab	Analysis Group	Method Description	Method Code	Analyte Description	CAS Number	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %
Pace	Water	SVOCs	8270C	Diethyl phthalate	84-66-2	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2,4-Dimethylphenol	105-67-9	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Dimethylphthalate	131-11-3	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	4,6-Dinitro-2-methylphenol**	534-52-1	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2,4-Dinitrophenol	51-28-5	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2,4-Dinitrotoluene	121-14-2	35	144	NA	36	130	20
Pace	Water	SVOCs	8270C	2,6-Dinitrotoluene	606-20-2	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Di-*n*-octyl phthalate	117-84-0	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Fluoranthene	206-44-0	66	141	NA	73	131	20
Pace	Water	SVOCs	8270C	Fluorene	86-73-7	57	127	NA	45	133	20
Pace	Water	SVOCs	8270C	Hexachlorobenzene	118-74-1	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Hexachloro-1,3-butadiene	87-68-3	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Hexachlorocyclopentadiene	77-47-4	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Hexachloroethane	67-72-1	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Indeno[1,2,3-cd]pyrene	193-39-5	49	141	NA	58	119	20
Pace	Water	SVOCs	8270C	Isophorone	78-59-1	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2-Methylphenol (o-Cresol)	95-48-7	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	3 & 4-Methylphenol (m & p Cresols)	108-39-4, 106-44-5	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	1-Methylnaphthalene	90-12-0	12	118	NA	36	121	20
Pace	Water	SVOCs	8270C	2-Methylnaphthalene	91-57-6	10	123	NA	30	107	20
Pace	Water	SVOCs	8270C	*N*-Nitroso-di-*n*-propylamine	621-64-7	31	121	NA	32	110	20
Pace	Water	SVOCs	8270C	*N*-Nitrosodiphenylamine	86-30-6	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Naphthalene	91-20-3	10	116	NA	25	90	20
Pace	Water	SVOCs	8270C	2-Nitroaniline	88-74-4	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	3-Nitroaniline	99-09-2	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	4-Nitroaniline	100-01-6	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	Nitrobenzene	98-95-3	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2-Nitrophenol	88-75-5	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	4-Nitrophenol	100-02-7	10	92	NA	10	80	20
Pace	Water	SVOCs	8270C	Pentachlorophenol	87-86-5	24	144	NA	23	137	20
Pace	Water	SVOCs	8270C	Phenanthrene	85-01-8	66	128	NA	73	118	20
Pace	Water	SVOCs	8270C	Phenol	108-95-2	10	68	NA	10	63	20
Pace	Water	SVOCs	8270C	Pyrene	129-00-0	71	132	NA	78	117	20
Pace	Water	SVOCs	8270C	1,2,4,5-tetrachlorobenzene	95-94-3	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2,3,4,6-tetrachlorophenol	58-90-2	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2,4,5-Trichlorophenol	95-95-4	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2,4,6-Trichlorophenol	88-06-2	NA	NA	NA	NA	NA	NA
Pace	Water	SVOCs	8270C	2,4,6-Tribromophenol (surr)	118-79-6	23	126	NA	23	126	NA
Pace	Water	SVOCs	8270C	2-Fluorobiphenyl (surr)	321-60-8	25	110	NA	25	110	NA
Pace	Water	SVOCs	8270C	2-Fluorophenol (surr)	367-12-4	10	78	NA	10	78	NA
Pace	Water	SVOCs	8270C	Nitrobenzne-d5 (surr)	4165-60-0	22	108	NA	22	108	NA
Pace	Water	SVOCs	8270C	Phenol-d5 (surr)	4165-62-2	10	61	NA	10	61	NA
Pace	Water	SVOCs	8270C	Terphenyl-d14 (surr)	1718-51-0	12	150	NA	12	150	NA
Pace	Water	Volatile Fatty Acids	AM23G	Acetic Acid	64-19-7	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Water	Volatile Fatty Acids	AM23G	Butyric Acid	107-92-6	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Water	Volatile Fatty Acids	AM23G	Formic Acid	64-18-6	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Water	Volatile Fatty Acids	AM23G	Hexanoic Acid	142-62-1	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Water	Volatile Fatty Acids	AM23G	Lactic Acid	50-21-5	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Water	Volatile Fatty Acids	AM23G	Pentanoic Acid	109-52-4	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Water	Volatile Fatty Acids	AM23G	Propionic Acid	79-09-4	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Water	Volatile Fatty Acids	AM23G	Pyruvic Acid	127-17-3	± 30% of its true value	± 30% of its true value	NA	70	130	NA

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Pace	Water	Volatile Fatty Acids	AM23G	i-Hexanoic Acid	646-07-1	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Water	Volatile Fatty Acids	AM23G	i-Pentanoic Acid	503-74-2	± 30% of its true value	± 30% of its true value	NA	70	130	NA
Pace	Soil	Total Organic Carbon	9060 Walkley Black Procedure	Total Organic Carbon	503-74-2	80	120	20	80	120	20.00
Pace	Soil	Percent Moisture	ASTM D 2974-87	Percent Moisture	NA	NA	NA	10	NA	NA	NA
Pace	Soil	RadChem	HASL 300	Isotopic Thorium	NA	75	125	NA	75	125	25
Pace	Soil	RadChem	HASL 300	Isotopic Uranium	NA	75	125	NA	75	125	25
Pace	Soil	RadChem	EPA 903.1	Radium-226	NA	73	135	NA	71	136	32
Pace	Soil	RadChem	EPA 904.0	Radium-228	NA	60	135	NA	60	135	36
Pace	Soil	RadChem	Calculated from Ra-226 and Ra-228	Total Radium	NA	NA	NA	NA	NA	NA	+/- 25%
MCL	Soil	QC methods are performed for all analyses unless otherwise specified. Reference the method- specific SOP for further information.			NA	NA	NA	± 25%[a]	NA	NA	± 40%[b]

Notes:

a: Generally for all analyses unless otherwise noted.

b: For laboratory duplicates only

Notes:

CAS-Chemical Abstracts Service
EPA-United States Environmental Protection Agency
LCS-Laboratory Control Sample
MCL-Materials and Chemistry Laboratory, Inc.
MS-Matrix Spike
NA-Not Available
Pace-Pace Analytical Services, Inc.
PCB-Polychlorinated Biphenyl
RPD-Relative Percent Difference
SVOC-Semi-Volatile Organic Compound
TDS-Total Dissolved Solids
TOC-Total Organic Carbon
VOC-Volatile Organic Compound

TABLE 2-2. HOLDING TIME AND PRESERVATION REQUIREMENTS
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Matrix	Analytical Group	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Water	Polychlorinated Biphenyls-1221 (EPA 8082)	One 100mL widemouth amber glass bottle	Cool to ≤6°C	Extract within 6 months of collection and analyze within 40 days of extraction
Water	Chloride Fluoride Sulfate (EPA 9056)	One 125mL plastic or glass bottle	Cool to ≤6°C	Nitrate or Nitrite: Analysis must be completed within 48 hours of collection date/time. Other Anions: Analysis must be completed within 28 days of collection date.
Water	Ferrous Iron (HACH Method 8146)	One 250mL amber glass bottle	Cool to ≤6°C 2 mL of Hydrochloric Acid per 100 mL of sample	Ferrous Iron must be completed within 24 hours of collection date/time. No headspace should be present in the sample bottle.
Water	Barium, Boron, Calcium, Cobalt, Iron, Magnesium, Manganese, Nickel, Sodium, Zinc (EPA 6010)	250mL in plastic container	Nitric Acid to pH <2* Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Water	Antimony, Arsenic, Beryllium, Cadmium, Chromium, Copper, Lead, Selenium, Silver, Thallium, Vanadium (EPA 6020)	250mL in plastic container	Nitric Acid to pH <2* Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Water	Mercury (EPA 7470)	500mL in plastic container	Nitric Acid to pH <2* Ambient	Analysis must be completed within 28 days of collection date.
Water	Total Hardness (EPA 2340B)	250mL in plastic container	Nitric Acid to pH <2* Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Water	Semivolatile Organic Compounds (EPA 8270)	1 x1L or 100mL amber glass container with Teflon-lined lid, preferably widemouth	Cool to ≤6°C	Sample must be extracted within 7 days of collection date and extract must be analyzed within 40 days of extraction date.
Water	Volatile Organic Compounds (EPA 8260)	Minimum 3 VOA vials. Additional sample is required if MS/MSD is required.	Acidified w/ 1:1 Hydrochloric Acid to pH<2, no headspace Cool to ≤6°C	pH>2: Analysis must be completed within 7 days of collection date. pH <2: Analysis must be completed within 14 days of collection date. (pH determined post analysis)
Water	Total Dissolved Solids (SM 2540C)	250mL minimum in plastic container	Cool to ≤6°C	Sample must be analyzed within 7 days of collection date.
Water	Chemical Oxygen Demand (EPA 410.4)	One 250mL plastic or glass container	Sulfuric Acid to pH <2 Cool to ≤6°C	Sample must be analyzed within 28 days of collection date.

TABLE 2-2. HOLDING TIME AND PRESERVATION REQUIREMENTS
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Matrix	Analytical Group	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Water	Sulfide (SM 4500-S2-D)	250mL in plastic container. Fill container completely without overflowing.	pH>9 with 1mL of 1:1 Sodium Hydroxide plus 0.5mL of 1N Zinc Acetate per 250mL sample. Cool to ≤6°C	Preserved: Analysis must be completed within 7 days of collection. Unpreserved: Analysis must be completed within 24 hours of collection.
Water	Nitrogen, Nitrate + Nitrite (EPA 353.2) Nitrogen, Nitrite (EPA 353.2) Nitrogen, Nitrate (EPA 353.2)	250mL in plastic container.	For combined nitrate/nitrite analysis Sulfuric Acid to pH <2 For nitrate or nitrite individually, unpreserved. Cool to ≤6°C	For preserved samples, analysis must be completed within 28 days of collection date. For unpreserved samples, analysis must be completed within 48 hours of collection.
Water	Phosphorus (EPA 365.1)	125mL in plastic or glass container.	Sulfuric Acid to pH <2 Cool to ≤6°C	Sample must be analyzed within 28 days of collection date.
Water	Ammonia as Nitrogen (SM 4500-NH3 G)	250mL in plastic or glass container.	Sulfuric Acid to pH <2 Cool to ≤6°C	Sample must be analyzed within 28 days of collection date.
Water	Total Organic Carbon (SM 5310C)	250mL amber glass bottle	Sulfuric Acid or Phosphoric Acid to pH <2 Cool to ≤6°C	Sample must be analyzed within 28 days of collection date.
Water	Radium-226 (EPA Methods 903.1) Radium-228 (EPA Methods 904.0) Isotopic Uranium (U-234, U-235, U-238) and Isotopic Thorium (Th-228, Th-230, Th-232) (HASL 300)	1L plastic or glass container	Nitric acid pH<2	Sample must be analyzed within 180 days
Water	Volatile Fatty Acids	2-40mL amber glass VOA vials	benzalkonium chloride (BAK) per sample	Store just above freezing but below 6 degrees C. 14 day hold time
Soil	Total Organic Carbon (Walkley-Black Procedure)	4 oz glass container	Thermal to ≤6°C-Preservation On ice ≤6° Celsius-Shipment	Sample must be analyzed within 28 days of collection date.
Soil	Radium-226 (EPA Methods 903.1) Radium-228 (EPA Methods 904.0) Isotopic Uranium (U-234, U-235, U-238) and Isotopic Thorium (Th-230, Th-232) (HASL 300)	1L plastic or glass container	Nitric acid pH<2	Sample must be analyzed within 180 days

TABLE 2-2. HOLDING TIME AND PRESERVATION REQUIREMENTS
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Matrix	Analytical Group	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Soil	Barium, Calcium, Iron, Magnesium, Manganese, Potassium, Sodium(EPA 6010)	1L vacuum-sealed bag (only 1L of sample needed for all soil analysis)	Frozen on dry ice	Analyze with 6 months
Soil	Carbonate (SM 2320E)			Analyzed within 24 hours after extraction
Soil	Fluoride, Phosphate, and Sulfate by EPA Method 300.0			Analyze within 28 days
Soil	Ferrous Iron and Ferric Iron by SM 3500-Fe B			Analyze immediately after extraction
Soil	Sulfide by Method EPA-OW-OST 376.3			Analyze within 180 days
Soil	U(VI) by SOP MCL-7737			Analyze within 7 days after extraction
Soil	Cation Exchange Capacity by EPA Method 9081			Analyze within 6 months
Soil	pH by EPA Method 9045D			Analyze within 28 days after extraction
Soil	X-Ray Diffraction by SOP MCL-7708			Analyzed within 24 hours after extraction
Soil	Percent Moisture by American Society of Testing and Materials (ASTM) D 2974-87			Analyze within 6 months
Solid Sample	Radium-226 (EPA Methods 903.1) Total Radium Total Thorium Total Uranium	1L plastic or glass container	Nitric acid pH<2	Analyze within 28 days
Aqueous Extracts	Total metals (Barium calculated based on Sequential Extraction Step 1, Calcium calculated based on Sequential Extraction Step 1, Iron calculated based on Sequential Extraction Step 1, Manganese calculated based on Sequential Extraction Step 1, Magnesium, Potassium, and Sodium) by SW-846 Methods 6010	Lab container	Cool to ≤6°C	Analyze within 6 months

TABLE 2-2. HOLDING TIME AND PRESERVATION REQUIREMENTS
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Matrix	Analytical Group	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Aqueous Extracts	Carbonate by SM 2320E	Lab container	Cool to ≤6°C	Analyze within 24 hours after extraction
Aqueous Extracts	Sulfate calculated based on Sequential Extraction Step 1	Lab container	Cool to ≤6°C	Analyze within 28 days
Aqueous Extracts	pH by EPA Method 9045D	Lab container	Cool to ≤6°C	Analyze within 24 hours after extraction

Notes:
L: Liter
mL: milliliter
oz: Ounce
SM: Standard Methods for the Examination of Water and Wastewater
SOP: Standard Operating Procedure
EPA: United States Environmental Protection Agency
VOA: Volatile Organic Analysis

* Samples received at pH >2 must be preserved to pH <2 with HNO_3 and be allowed to equilibrate for 24 hours before being prepared for analysis. Acidification date and time are recorded in the Sample Preservation Logbook.

TABLE 2-3a. LIST OF CONSTITUENTS AND DESIRED REPORTING LIMITS FOR GROUNDWATER SAMPLING
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Target Analytes	CAS Number	Method	EPA Maximum Contamination Level (µg/L)[2]	MDNR Groundwater Protection Standards (µg/L)[3,4]	Lowest Screening Level EPA MCL/MDNR GWPS (µg/L)	Pace Analytical Services, LLC		Comparison of Laboratory Limits to Water Cleanup Levels
						MRL (µg/L)	MDL (µg/L)	
Metals[5]								
Antimony	7440-36-0	EPA 6020	6.0	6	6	1	0.146	MRL below Screening Level
Arsenic	7440-38-2	EPA 6020	10.0	10	10	1	0.116	MRL below Screening Level
Barium	7740-39-3	EPA 6010	2000.0	10	10	10	0.83	GWPS equals MRL
Beryllium	7440-41-7	EPA 6020	4.0	4	4	0.2	0.039	MRL below Screening Level
Boron	7440-42-8	EPA 6010	NL	NL	Not Applicable	100	2.77	Not Applicable
Cadmium	7440-43-9	EPA 6020	5.0	5	5	0.2	0.33	MRL below Screening Level
Calcium	7440-70-2	EPA 6010	NL	NL	Not Applicable	1000	250	Not Applicable
Chromium	7440-47-3	EPA 6020	100.0	2	2	2	0.305	GWPS equals MRL
Cobalt	7440-48-4	EPA 6010	NL	10	Not Applicable	10	0.68	GWPS equals MRL
Copper	7440-50-8	EPA 6020	1300.0	1300	1300	1	4.43	MRL below Screening Level
Iron	7439-89-6	EPA 6010	NL	NL	Not Applicable	100	22.36	Not Applicable
Lead	7439-92-1	EPA 6020	15.0	1	1	1	0.119	GWPS equals MRL
Magnesium	7439-95-4	EPA 6010	NL	NL	Not Applicable	1000	500	Not Applicable
Manganese	7439-96-5	EPA 6010	NL	NL	Not Applicable	10	0.6	Not Applicable
Mercury	7439-97-6	EPA 7470	2.0	2	2	2	0.1	MRL below Screening Level
Nickel	7440-02-0	EPA 6010	NL	10	10	10	1.77	GWPS equals MRL
Potassium	7440-09-7	EPA 6010	NL	NL	Not Applicable	1000	91.92	Not Applicable
Selenium	7782-49-2	EPA 6020	50.0	50	50	1	0.275	MRL below Screening Level
Silver	7440-22-4	EPA 6020	NL	0.5	0.50	0.5	0.066	GWPS equals MRL
Thallium	7440-28-0	EPA 6020	2.0	2	2	1	0.132	MRL below Screening Level
Vanadium	7440-62-2	EPA 6020	NL	1	1	1	0.215	GWPS equals MRL
Zinc	7440-66-6	EPA 6010	NL	20	20	20	1.46	GWPS equals MRL
Sodium	7440-23-5	EPA 6010	NL	NL	Not Applicable	1000	250	Not Applicable
Semi-Volatile Organic Compounds								
Acenaphthene	83-32-9	8270C	NL	10	10	10	5	GWPS equals MRL
Acenaphthylene	208-96-8	8270C	NL	10	10	10	5	GWPS equals MRL
Acetophenone	98-86-2	8270C	NL	10	10	10	5	GWPS equals MRL
Anthracene	120-12-7	8270C	NL	10	10	10	5	GWPS equals MRL
Atrazine	1912-24-9	8270C	3.0	3	3	10	5	**Screening Level below MDL**
Benzaldehyde	100-52-7	8270C	NL	NL	Not Applicable	50	25	Not Applicable
Benz[a]anthracene	56-55-3	8270C	NL	10	10	10	5	GWPS equals MRL
Benzo[a]pyrene	50-32-8	8270C SIM	0.2	0.2	0.20	0.1	0.013	MRL below Screening Level
Benzo[b]fluoranthene	205-99-2	8270C	NL	10	10	10	5	GWPS equals MRL
Benzo[g,h,i]perylene	191-24-2	8270C	NL	10	10	10	5	GWPS equals MRL
Benzo[k]fluoranthene	207-08-9	8270C	NL	10	10	10	5	GWPS equals MRL
Benzyl alcohol	100-51-6	8270C	NL	10	10	10	10	GWPS equals MRL
Biphenyl (1,1 - biphenyl or Diphenyl)	92-52-4	8270C	NL	NL	Not Applicable	10	5	Not Applicable
bis(2-chloroethoxy) methane	111-91-1	8270C	NL	10	10	10	5	GWPS equals MRL

TABLE 2-3a. LIST OF CONSTITUENTS AND DESIRED REPORTING LIMITS FOR GROUNDWATER SAMPLING
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Target Analytes	CAS Number	Method	EPA Maximum Contamination Level (µg/L)[2]	MDNR Groundwater Protection Standards (µg/L)[3,4]	Lowest Screening Level EPA MCL/MDNR GWPS (µg/L)	Pace Analytical Services, LLC		Comparison of Laboratory Limits to Water Cleanup Levels
						MRL (µg/L)	MDL (µg/L)	
bis(2-chloroethyl) ether	111-44-4	8270C	NL	10	10	10	5	GWPS equals MRL
bis(2-chloro-1-methylethyl) ether*	108-60-1	8270C	NL	10	10	10	5	GWPS equals MRL
bis(2-ethylhexyl) phthalate	117-81-7	8270C	NL	6	6	10	5	MDL below Screening Level
4-Bromophenyl phenyl ether	101-55-03	8270C	NL	10	10	10	5	GWPS equals MRL
Butyl benzyl phthalate	85-68-7	8270C	NL	10	10	10	5	GWPS equals MRL
4-Chloroaniline	106-47-8	8270C	NL	NL	Not Applicable	10	10	Not Applicable
4-Chloro-3-methylphenol (p-chloro-m-Cresol)	59-50-7	8270C	NL	10	10	10	7	GWPS equals MRL
2-Chloronaphthalene	91-58-7	8270C	NL	10	10	10	5	GWPS equals MRL
2-Chlorophenol	95-57-8	8270C	NL	10	10	10	5	GWPS equals MRL
4-Chlorophenyl phenyl ether	7005-72-3	8270C	NL	10	10	10	5	GWPS equals MRL
Caprolactam	105-60-2	8270C	NL	NL	Not Applicable	10	5	Not Applicable
Carbazole	86-74-8	8270C	NL	NL	Not Applicable	10	5	Not Applicable
Chrysene	218-01-9	8270C	NL	10	10	10	5	GWPS equals MRL
Dibenz[a,h]anthracene	53-70-3	8270C	NL	10	10	10	5	GWPS equals MRL
Dibenzofuran	132-64-9	8270C	NL	10	10	10	5	GWPS equals MRL
Di-*n*-butyl phthalate	84-74-2	8270C	NL	10	10	10	5	GWPS equals MRL
3,3'-Dichlorobenzidine	91-94-1	8270C	NL	NL	Not Applicable		10	Not Applicable
2,4-Dichlorophenol	120-83-2	8270C	NL	10	10	10	5	GWPS equals MRL
Diethyl phthalate	84-66-2	8270C	NL	10	10	10	5	GWPS equals MRL
2,4-Dimethylphenol	105-67-9	8270C	NL	10	10	10	5	GWPS equals MRL
Dimethylphthalate	131-11-3	8270C	NL	10	10	10	5	GWPS equals MRL
4,6-Dinitro-2-methylphenol	534-52-1	8270C	NL	20	20	20	10	GWPS equals MRL
2,4-Dinitrophenol	51-28-5	8270C	NL	50	50	50	25	GWPS equals MRL
2,4-Dinitrotoluene	121-14-2	8270C	NL	10	10	10	5	GWPS equals MRL
2,6-Dinitrotoluene	606-20-2	8270C	NL	10	10	10	5	GWPS equals MRL
Di-*n*-octyl phthalate	117-84-0	8270C	NL	10	10	10	5	GWPS equals MRL
Fluoranthene	206-44-0	8270C	NL	10	10	10	5	GWPS equals MRL
Fluorene	86-73-7	8270C	NL	10	10	10	5	GWPS equals MRL
Hexachlorobenzene	118-74-1	8270C	1.0	1	1	10	5	**Screening Level below MDL**
Hexachloro-1,3-butadiene	87-68-3	8270C	NL	10	10	10	5	GWPS equals MRL
Hexachlorocyclopentadiene	77-47-4	8270C	50.0	50	50	10	5	MRL below Screening Level
Hexachloroethane	67-72-1	8270C	NL	10	10	10	5	GWPS equals MRL
Indeno[1,2,3-cd]pyrene	193-39-5	8270C	NL	10	10	10	5	GWPS equals MRL
Isophorone	78-59-1	8270C	NL	10	10	10	5	GWPS equals MRL
2-Methylphenol (o-Cresol)	95-48-7	8270C	NL	NL	Not Applicable	10	5	Not Applicable

TABLE 2-3a. LIST OF CONSTITUENTS AND DESIRED REPORTING LIMITS FOR GROUNDWATER SAMPLING
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Target Analytes	CAS Number	Method	EPA Maximum Contamination Level (µg/L)[2]	MDNR Groundwater Protection Standards (µg/L)[3,4]	Lowest Screening Level EPA MCL/MDNR GWPS (µg/L)	Pace Analytical Services, LLC		Comparison of Laboratory Limits to Water Cleanup Levels
						MRL (µg/L)	MDL (µg/L)	
3 & 4-Methylphenol (m & p Cresols)[1]	108-39-4, 106-44-5	8270C	NL	NL	Not Applicable	10	10	Not Applicable
1-Methylnaphthalene	90-12-0	8270C	NL	NL	Not Applicable	10	5	Not Applicable
2-Methylnaphthalene	91-57-6	8270C	NL	10	10	10	5	GWPS equals MRL
N-Nitroso-di-*n*-propylamine	621-64-7	8270C	NL	10	10	10	5	GWPS equals MRL
N-Nitrosodiphenylamine	86-30-6	8270C	NL	10	10	10	5	GWPS equals MRL
Naphthalene	91-20-3	8270C	NL	10	10	10	5	GWPS equals MRL
2-Nitroaniline	88-74-4	8270C	NL	10	10	10	5	GWPS equals MRL
3-Nitroaniline	99-09-2	8270C	NL	10	10	10	5	GWPS equals MRL
4-Nitroaniline	100-01-6	8270C	NL	10	10	10	5	GWPS equals MRL
Nitrobenzene	98-95-3	8270C	NL	10	10	10	5	GWPS equals MRL
2-Nitrophenol	88-75-5	8270C	NL	10	10	10	5	GWPS equals MRL
4-Nitrophenol	100-02-7	8270C	NL	50	50	50	50	GWPS equals MRL
Pentachlorophenol	87-86-5	8270C	1.0	50	1	50	25	GWPS equals MRL
Phenanthrene	85-01-8	8270C	NL	10	10	10	5	GWPS equals MRL
Phenol	108-95-2	8270C	NL	10	10	10	5	GWPS equals MRL
Pyrene	129-00-0	8270C	NL	10	10	10	5	GWPS equals MRL
1,2,4,5-tetrachlorobenzene	95-94-3	8270C	NL	10	10	10	5	GWPS equals MRL
2,3,4,6-tetrachlorophenol	58-90-2	8270C	NL	10	Not Applicable	10	5	GWPS equals MRL
2,4,5-Trichlorophenol	95-95-4	8270C	NL	10	10	10	5	GWPS equals MRL
2,4,6-Trichlorophenol	88-06-2	8270C	NL	10	10	10	5	GWPS equals MRL
Volatile Organic Compounds								
Acetone	67-64-1	8260C	NL	100	100	100	50	GWPS equals MRL
Acrolein	107-02-8	8260C	NL	NL	Not Applicable	50	25	Not Applicable
Acrylonitrile	107-13-1	8260C	NL	NL	Not Applicable	100	50	Not Applicable
Benzene	71-43-2	8260C	5	5	5	5.0	1.0	MRL below Screening Level
Bromobenzene	108-86-1	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
Bromodichloromethane	75-27-4	8260C	80	100	80	5.0	2.5	MRL below Screening Level
Bromoform	75-25-2	8260C	80	100	80	5.0	2.5	MRL below Screening Level
Bromomethane (Methyl Bromide)	74-83-9	8260C	NL	5	5	5.0	3.9	GWPS equals MRL
Bromochloromethane	74-97-5	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
2-Butanone (MEK)	78-93-3	8260C	NL	25	25	25	12	GWPS equals MRL
n-Butylbenzene	104-51-8	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
sec-Butylbenzene	135-98-8	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
tert-Butylbenzene	98-06-6	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
Carbon disulfide	75-15-0	8260C	NL	10	10	10	5.0	GWPS equals MRL
Carbon tetrachloride	56-23-5	8260C	5	5	5	5.0	2.5	MRL below Screening Level
Chlorobenzene	108-90-7	8260C	100	100	100	5.0	2.5	MRL below Screening Level
Chloroethane (Ethyl Chloride)	75-00-3	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
Chloroform	67-66-3	8260C	80.0	100	80	5.0	2.5	MRL below Screening Level

TABLE 2-3a. LIST OF CONSTITUENTS AND DESIRED REPORTING LIMITS FOR GROUNDWATER SAMPLING
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Target Analytes	CAS Number	Method	EPA Maximum Contamination Level (µg/L)[2]	MDNR Groundwater Protection Standards (µg/L)[3,4]	Lowest Screening Level EPA MCL/MDNR GWPS (µg/L)	Pace Analytical Services, LLC		Comparison of Laboratory Limits to Water Cleanup Levels
						MRL (µg/L)	MDL (µg/L)	
Chloromethane (Methyl Chloride)	74-87-3	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
2-Chlorotoluene	95-49-8	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
4-Chlorotoluene	106-43-4	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
Cyclohexane	110-82-7	8260C	NL	NL	Not Applicable	100	50	Not Applicable
Dibromochloromethane	124-48-1	8260C	80	100.0	80.00	5.0	2.5	MRL below Screening Level
1,2-Dibromoethane (EDB)	106-93-4	8260C SIM	0.1	5	0	5.0	2.5	**Screening Level below MDL**
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	8260C SIM	0.2	10	0	10	5.0	**Screening Level below MDL**
Dibromomethane (Methylene Bromide)	74-95-3	8260C	NL	5	5	5.0	2.7	GWPS equals MRL
trans-1,4-Dichloro-2-butene	110-57-6	8260C	NL	100	100	100	50	GWPS equals MRL
1,2-Dichlorobenzene	95-50-1	8260C	600	600	600	5.0	2.5	MRL below Screening Level
1,3-Dichlorobenzene	541-73-1	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
1,4-Dichlorobenzene	106-46-7	8260C	75	75	75	5.0	2.5	MRL below Screening Level
Dichlorodifluoromethane	75-71-8	8260C	NL	5	5	5.0	5.0	GWPS equals MRL
1,1-Dichloroethane (DCA)	75-34-3	8260C	NL	5	5	5.0	0.6	GWPS equals MRL
1,2-Dichloroethane (EDC)	107-06-2	8260C	5	5	5	5.0	0.6	MRL below Screening Level
1,1-Dichloroethene	75-35-4	8260C	7	7	7	5.0	2.5	MRL below Screening Level
cis-1,2-Dichloroethene	156-59-2	8260C	70	70	70	5.0	0.65	MRL below Screening Level
trans-1,2-Dichloroethene	156-60-5	8260C	100	100	100	5.0	0.86	MRL below Screening Level
1,2-Dichloropropane	78-87-5	8260C	5	5	5	5.0	2.5	GWPS equals MRL
1,3-Dichloropropane	142-28-9	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
2,2-Dichloropropane	594-20-7	8260C	NL	5	5	5.0	4.2	GWPS equals MRL
1,1-Dichloropropene	563-58-6	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
cis-1,3-Dichloropropene	10061-01-5	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
trans-1,3-Dichloropropene	10061-02-6	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
1,4-Dioxane (p-Dioxane)	123-91-1	8260C	NL	NL	Not Applicable	100	50	Not Applicable
Ethylbenzene	100-41-4	8260C	700	700	700	5.0	2.5	MRL below Screening Level
Ethyl methacrylate	97-63-2	8260C	NL	100	Not Applicable	100	50	GWPS equals MRL
Hexachloro-1,3-butadiene	87-68-3	8260C	NL	5	Not Applicable	5.0	2.5	GWPS equals MRL
n-Hexane	110-54-3	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
2-Hexanone	591-78-6	8260C	NL	25	25	25	12	GWPS equals MRL
Iodomethane	74-88-4	8260C	NL	10	10	10	6.8	GWPS equals MRL
Isopropylbenzene (Cumene)	98-82-8	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
p-Isopropyltoluene	99-87-6	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
Methyl Acetate	79-20-9	8260C	NL	NL	Not Applicable	50	25	Not Applicable
Methylcyclohexane	108-87-2	8260C	NL	NL	Not Applicable	50	25	Not Applicable
Methylene Chloride (Dichloromethane)	75-09-2	8260C	5	5	5	5.0	5.0	GWPS equals MRL
1-Methylnaphthalene	90-12-0	8260C	NL	NL	Not Applicable	10	10	Not Applicable

TABLE 2-3a. LIST OF CONSTITUENTS AND DESIRED REPORTING LIMITS FOR GROUNDWATER SAMPLING
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Target Analytes	CAS Number	Method	EPA Maximum Contamination Level (µg/L)[2]	MDNR Groundwater Protection Standards (µg/L)[3,4]	Lowest Screening Level EPA MCL/MDNR GWPS (µg/L)	Pace Analytical Services, LLC		Comparison of Laboratory Limits to Water Cleanup Levels
						MRL (µg/L)	MDL (µg/L)	
2-Methylnaphthalene	91-57-6	8260C	NL	10	10	10	10	GWPS equals MRL
4-Methyl-2-pentanone (MIBK)	108-10-1	8260C	NL	25	25	25	12	GWPS equals MRL
Methyl-tert-butyl-Ether (MTBE)	1634-04-4	8260C	NL	4	4	4	2.1	GWPS equals MRL
Naphthalene	91-20-3	8260C	NL	1.4	1.4	1.4	1.4	GWPS equals MRL
n-Propylbenzene	103-65-1	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
Styrene	100-42-5	8260C	100	100	100	5.0	2.5	MRL below Screening Level
1,1,1,2-Tetrachloroethane	630-20-6	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
1,1,2,2-Tetrachloroethane	79-34-5	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
Tetrachloroethene (PCE)	127-18-4	8260C	5	5	5	5.0	0.93	MRL below Screening Level
Toluene	108-88-3	8260C	1000	1000	1000	5.0	2.5	MRL below Screening Level
1,2,3-Trichlorobenzene	87-61-6	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
1,2,4-Trichlorobenzene	120-82-1	8260C	70	70	70	5.0	2.5	MRL below Screening Level
1,1,1-Trichloroethane (TCA)	71-55-6	8260C	200	200	200	5.0	0.89	MRL below Screening Level
1,1,2-Trichloroethane	79-00-5	8260C	5	5	5	5.0	2.5	MRL below Screening Level
1,1,2-Trichlorotrifluoroethane	76-13-1	8260C	NL	NL	Not Applicable	5.0	2.5	Not Applicable
Trichloroethene (TCE)	79-01-6	8260C	5	5	5	5.0	0.8	MRL below Screening Level
Trichlorofluoromethane	75-69-4	8260C	NL	100	100	5.0	2.5	MRL below Screening Level
1,2,3-Trichloropropane	96-18-4	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
1,2,4-Trimethylbenzene	95-63-6	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
1,3,5-Trimethylbenzene	108-67-8	8260C	NL	5	5	5.0	2.5	GWPS equals MRL
Vinyl Acetate	108-05-4	8260C	NL	50	50	50	25	GWPS equals MRL
Vinyl Chloride (Chloroethene)	75-01-4	8260C	2	2	2	2.0	0.97	MRL below Screening Level
Xylenes, Total	1330-20-7	8260C	10000	10000	10000	10	5.0	MRL below Screening Level
PCBs								
PCB-1221 (Aroclor 1221)	11104-28-2	EPA 8082	0.5	0.5	0.50	0.2	0.1	MRL below Screening Level
General Chemistry								
Chemical Oxygen Demand	Not Applicable	EPA 410.4	NL	NL	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Ferrous Iron	15438-31-0	SM-3500-Fe-B	NL	NL	Not Applicable	0.05	0.015	Not Applicable
Chloride	16887-00-6	EPA 9056	NL	NL	Not Applicable	250	130	Not Applicable
Fluoride	16984-48-8	EPA 9056	4000	4000	4000	100	31	MRL below Screening Level
Isotopic Thorium	Not Applicable	HASL 300	NL	NL	Not Applicable	Not Applicable	1pCi/L	Not Applicable
Isotopic Uranium	Not Applicable	HASL 300	NL	NL	Not Applicable	Not Applicable	1pCi/L	Not Applicable
Nitrite + Nitrate as Nitrogen	Not Applicable	EPA 353.2	10000	10000	10000	Not Applicable	24	MRL below Screening Level
Nitrogen as Ammonia	Not Applicable	SM 4500-NH3 G	NL	NL	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Nitrite as Nitrogen	Not Applicable	EPA 353.2	1000	1000	1000	Not Applicable	6.7	MRL below Screening Level
Nitrate as Nitrogen	Not Applicable	EPA 353.2	10000	10000	10000	Not Applicable	20	MRL below Screening Level
Phosphorus	7723-14-0	EPA 365.1	NL	NL	Not Applicable	50	14	Not Applicable

TABLE 2-3a. LIST OF CONSTITUENTS AND DESIRED REPORTING LIMITS FOR GROUNDWATER SAMPLING
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Target Analytes	CAS Number	Method	EPA Maximum Contamination Level (µg/L)[2]	MDNR Groundwater Protection Standards (µg/L)[3,4]	Lowest Screening Level EPA MCL/MDNR GWPS (µg/L)	Pace Analytical Services, LLC		Comparison of Laboratory Limits to Water Cleanup Levels
						MRL (µg/L)	MDL (µg/L)	
Radium- 226	Not Applicable	EPA 903.1	5 pCi/L	5 pCi/L	5 pCi/L	Not Applicable	1pCi/L	MDL below Screening Level
Radium- 228	Not Applicable	EPA 904.0	5 pCi/L	5 pCi/L	5 pCi/L	Not Applicable	1pCi/L	MDL below Screening Level
Sulfate	14808-79-8	EPA 9056	NL	NL	Not Applicable	250	130	Not Applicable
Sulfide	18496-25-8	SM 4500-S2-D	NL	NL	Not Applicable	100	50	Not Applicable
Total Dissolved Solids	Not Applicable	SM 2540C	NL	NL	Not Applicable	10000	5515	Not Applicable
Total Hardness	Not Applicable	EPA 2340B	NL	NL	Not Applicable	Not Applicable	Not Applicable	Not Applicable
pH	Not Applicable	9045D	NL	NL	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Total Organic Carbon	7440-44-0	SM 5310C	NL	NL	Not Applicable	1000	166	Not Applicable
Lactic Acid	50-21-5	AM23G Scrubbed	NL	Not Applicable	Not Applicable	200	48	Not Applicable
Acetic Acid	64-19-7	AM23G Scrubbed	NL	Not Applicable	Not Applicable	100	28	Not Applicable
Proprionic Acid	79-09-4	AM23G Scrubbed	NL	Not Applicable	Not Applicable	100	4	Not Applicable
Formic Acid	64-18-6	AM23G Scrubbed	NL	Not Applicable	Not Applicable	500	98	Not Applicable
Butyric Acid	107-92-6	AM23G Scrubbed	NL	Not Applicable	Not Applicable	100	11	Not Applicable
Pyruvic Acid	127-17-3	AM23G Scrubbed	NL	Not Applicable	Not Applicable	100	5	Not Applicable
i-Pentanoic	503-74-2	AM23G Scrubbed	NL	Not Applicable	Not Applicable	100	6	Not Applicable
Pentanoic Acid	109-52-4	AM23G Scrubbed	NL	Not Applicable	Not Applicable	100	11	Not Applicable
i-Hexanoic	646-07-1	AM23G Scrubbed	NL	Not Applicable	Not Applicable	200	29	Not Applicable
Hexanoic Acid	142-62-1	AM23G Scrubbed	NL	Not Applicable	Not Applicable	200	32	Not Applicable

Notes:

MRL-Method Reporting Limit
MDL-Method Detection Limit
NL-Not listed in referenced document
RL-Reporting Limit
RSL-Regional Screening Level
µg/L-micrograms per Liter
MDNR-Missouri Department of Natural Resources
EPA-United States Environmental Protection Agency
GWPS - Groundwater Protection Standards
SIM -Selective Ion Monitoring

1-Screening levels for 4-methylphenol were used because it was more conservative than those of 3-methylphenol.
2-EPA. Regional Screening Levels (RSLs) - Generic Tables ((TR=1E-06THQ=0.1)). November 2019
3-MDNR Groundwater Protection Standards (GWPS)-Screening levels were generated using guidelines from the Bridgeton Landfill Revised Groundwater Monitoring Plan and Assessment Plan; Appendix A. Feezor Engineering. March 2019.
4-The GWPSs and background levels from the groundwater monitoring report were used to populate the GWPS column for comparison to the MRL and MDLs. Updated GWPSs and statistically-determined background levels will be developed as part of the sampling and analyses for operable unit 3.
5-Metals analyzed as both total and dissolved.
Items bolded to indicate that reporting limits do not meet screening levels.

TABLE 2-3b. LIST OF PACE CONSTITUENTS AND DESIRED REPORTING LIMITS FOR SOIL SAMPLING
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Target Analytes	CAS Number	Method	EPA Maximum Contamination Level (mg/kg)[1]	Missouri Soil Screening Levels (mg/kg)[2]	Lowest Screening Level EPA MCL/Missouri Soil Screening Levels (mg/kg)	Pace Analytical Services, LLC		Comparison of Laboratory Limits to Water Cleanup Levels
						MRL	MDL	
Total Organic Carbon	7440-44-0	Walkley-Black Procedure	NL	NL	NL	644.2 mg/kg (LOQ)	193.27 mg/kg (LOD)	NA
Radium- 226	Not Applicable	EPA 903.1	5 pCi/L	NL	5 pCi/L	Not Applicable	1pCi/L	MDL is Below the Screening Level
Radium- 228	Not Applicable	EPA 904.0	5 pCi/L	NL	5 pCi/L	Not Applicable	1pCi/L	MDL is Below the Screening Level
Isotopic Thorium	Not Applicable	HASL 300	NL	NL	NL	1pCi/L	1pCi/L	NA
Percent Moisture	NA	ASTM D 2974-87	NL	NL	NL	0.1 %w/w	0.1 %w/w	NA

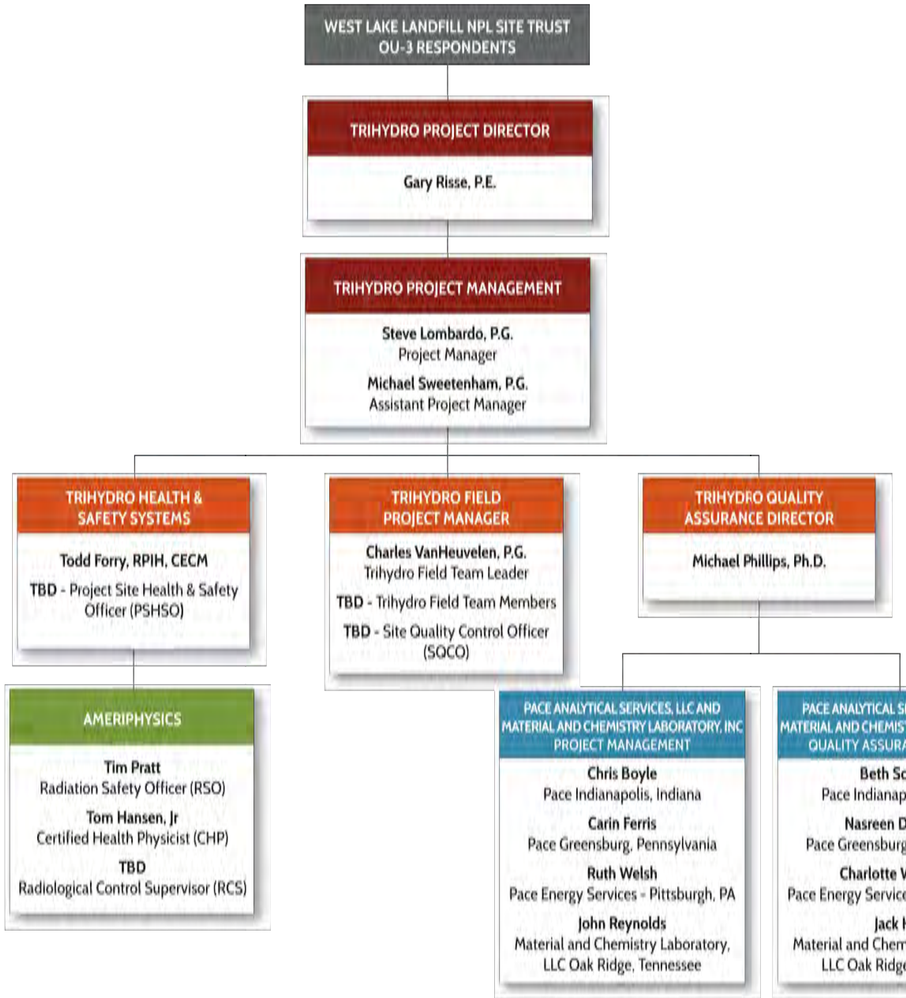
Notes:
Items bolded to indicate that reporting limits do not meet screening levels.
1-EPA. 2018. Regional Screening Levels (RSLs) - Generic Tables ((TR=1E-06THQ=0.1)). November.
2-MDNR. 2006. Table B-1. MRBCA Technical Guidance. April.
Abbreviations:
ASTM-American Society of Testing and Material
LOD-Limit of Detection
LOQ-Limit of Quantitation
MCL-Maximum Contamination Level
MRL-Method Reporting Limit
MDL-Method Detection Limit
MDNR: Missouri Department of Natural Resources
mg/kg-milligrams per kilogram
MRBCA-Missouri Risk-Based Corrective Action
NA-Not Applicable
NL-Not listed in referenced document
RSL-Regional Screening Level
mg/kg-milligrams per kilogram
EPA-United States Environmental Protection Agency

TABLE 2-3c. LIST OF CONSTITUENTS AND DESIRED REPORTING LIMITS FOR MCLI SAMPLE ANALYSES
WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION/FEASIBILITY STUDY QUALITY ASSURANCE PROJECT PLAN

Fate and Transport Model Input Parameter	Description	Method Reference	Concentration Units	Reporting Limit	MDL's
Radionuclides	U-234	EPA 6020	mg/kg	0.005	0.002
	U-235	EPA 6020	mg/kg	0.05	0.01
	U-238	EPA 6020	mg/kg	1	0.3
	Total U	EPA 6020	mg/kg	1	0.3
Major Cations ans Anions	Barium	EPA 3050, EPA 6010	mg/kg	2	0.1
	Calcium	EPA 3050, EPA 6010	mg/kg	20	2.7
	Carbonate	Water Leach, SM 2320E	mg/kg	per method	NA
	Fluoride	Water Leach, EPA 300	mg/kg	2	0.2
	Iron	EPA 3050, EPA 6010	mg/kg	20	5.1
	Magnesium	EPA 3050, EPA 6010	mg/kg	20	8
	Manganese	EPA 3050, EPA 6010	mg/kg	20	0.1
	Phosphate	Water Leach, EPA 300	mg/kg	12	0.4
	Potassium	EPA 3050, EPA 6010	mg/kg	20	8
	Sodium	EPA 3050, EPA 6010	mg/kg	20	8
	Sulfate	Water Leach, EPA 300	mg/kg	12	0.3
Attenuation Capacity	Cation Exchange Capacity	EPA 9081	meq/100g	per method	NA
pH & Redox Indicators	pH	EPA 9045D	std	0.05	NA
	Uranium (VI)	MCL-7737	mg/kg	per method	NA
	Sulfide	Method EPA-OW-OST 376.3	mg/kg	per method	NA
	Ferric Iron	EPA 6010 by difference with Ferrous	mg/kg	per method	NA
	Ferrous Iron	SM 3500-Fe B	mg/kg	per method	NA
Major Minerals	X-Ray Diffraction	MCL-7712	Wt %	3-5	NA
Radionuclide Speciation	Sequential Extraction Analysis	MCL-7775 Appendix 37	NA	NA	NA
Mineral Reactivity	Scanning Electron Microscope with Energy Dispersive X-Ray Spectrometry	MCL-7708	Identification	NA	NA

Abbreviations:
MCL-Materials and Chemistry Laboratory, Inc.
MDL-Method Detection Limit
mg/kg-milligrams per kilogram
NA-Not Applicable
meq/100g-measured in milliequivalents per 100 grams
mg/kg-milligrams per kilogram
SM-Standard Methods
EPA-United States Environmental Protection Agency
Wt%-Percent Weight

FIGURE



TBD - To be Determined

Trihydro CORPORATION
1252 Commerce Drive
Laramie, Wyoming 82070
www.trihydro.com
(P) 307/745.7474 (F) 307/745.7729

FIGURE 1-1

PROJECT ORGANIZATIONAL CHART

WEST LAKE LANDFILL OU-3
REMEDIAL INVESTIGATION / FEASIBILITY STUDY
QUALITY ASSURANCE PROJECT PLAN

Drawn By: REP	Checked By: MH	Scale: NONE	Date: 5/16/19	File: 63N-FSP_ORGCHART_20190509

ATTACHMENT A

QUALITY ASSURANCE MANUALS AND LABORATORY CERTIFICATIONS

A-1. QUALITY ASSURANCE MANUAL, QUALITY ASSURANCE/QUALITY CONTROL POLICIES AND PROCEDURES, PACE ANALYTICAL SERVICES, LLC-INDIANAPOLIS

A-2. QUALITY ASSURANCE MANUAL, QUALITY ASSURANCE/QUALITY CONTROL POLICIES AND PROCEDURES, PACE ANALYTICAL SERVICES, LLC-PITTSBURGH

A-3. QUALITY ASSURANCE MANUAL, QUALITY ASSURANCE/QUALITY CONTROL POLICIES AND PROCEDURES, PACE ANALYTICAL ENERGY SERVICES, LLC-PITTSBURG

A-4. QUALITY ASSURANCE PLAN MCL-7701, MATERIAL AND CHEMISTRY LABORATORY, LLC, OAK RIDGE, TENNESSEE

A-5. KANSAS DEPARTMENT OF HEALTH & ENVIRONMENT ACCREDITATION, PACE ANALYTICAL SERVICES, LLC-INDIANAPOLIS

A-6. COMMONWEALTH OF PENNSYLVANIA DEPARTMENT OF ENVIRONMENTAL PROTECTION CERTIFICATION PROGRAM, PACE ANALYTICAL SERVICES, LLC-PITTSBURGH

A-7. ANAB CERTIFICATE OF ACCREDITATION, U.S. DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V5.1.1), PACE ANALYTICAL SERVICES, LLC-PITTSBURGH

A-8. LABORATORY ACCREDITATION & CERTIFICATION, PACE ANALYTICAL ENERGY SERVICES, LLC, PITTSBURGH

A-9. PERRY JOHNSON LABORATORY ACCREDITATION, INC., CERTIFICATE OF ACCREDITATION, MATERIAL AND CHEMISTRY LABORATORY, INC., OAK RIDGE, TENNESSEE

ATTACHMENT A-1

QUALITY ASSURANCE MANUAL, QUALITY ASSURANCE/QUALITY CONTROL POLICIES AND PROCEDURES, PACE ANALYTICAL SERVICES, LLC-INDIANAPOLIS

QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures

Pace Analytical Services, LLC – Indianapolis
7726 Moller Road
Indianapolis, IN 46268
(317)228-3100

APPROVAL

Steve Sayer
General Manager
(317)228-3100

June 20, 2018
Date

Beth Schrage
Quality Manager
(317)228-3100

June 19, 2018
Date

Anne Troyer
Technical Director
(317)228-3100

June 19, 2018
Date

Table of Contents

1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"
Pace Analytical Services LLC - Mission Statement

1.1. Introduction to Pace

1.1.1. Pace Analytical Services, LLC (Pace) is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. Pace offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. This document defines the Quality System and Quality Assurance (QA)/Quality Control (QC) protocols.

1.1.2. Pace laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Methods are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 of this document is a representative listing of general analytical protocol references.

1.2. Statement of Purpose

1.2.1. To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. Pace management is committed to maintaining the highest possible standard of service and quality for our customers by following a documented quality system that is compliant with all current applicable state, federal, and industry standards, such as the NELAC Standard, the TNI Standard, and ISO standards and is in accordance with the stated methods and customer requirements. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the Pace network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work.

1.4. Core Values

1.4.1. The following are the Pace Core Values:

- **Integrity**
- **Value Employees**
- **Know Our Customers**
- **Honor Commitments**

- **Flexible Response To Demand**
- **Pursue Opportunities**
- **Continuously Improve**

1.5. Code of Ethics and Standards of Conduct

1.5.1. **Code of Ethics**:

1.5.1.1. Each Pace employee is responsible for the propriety and consequences of his or her actions;

1.5.1.2. Each Pace employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where Pace does business or seeks to do business;

1.5.1.3. Each Pace employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

1.5.1.4. Each Pace employee must recognize and understand that our daily activities in environmental laboratories affect public health as well as the environment and that environmental laboratory analysts are a critical part of the system society depends upon to improve and guard our natural resources:

1.5.2. **Standards of Conduct:**

1.5.2.1. **Data Integrity**

1.5.2.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at Pace are the cornerstones of the company. Employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

1.5.2.1.2. Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work including commercial, financial, over-scheduling, and working condition pressures.

1.5.2.1.3. The data integrity system includes in-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.5.2.1.4. Any documentation related to data integrity issues, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.5.2.2. **Confidentiality**

1.5.2.2.1. Pace employees must not use or disclose confidential or proprietary information except when in connection with their duties at Pace. This is effective over the course of employment and for an additional period of two years thereafter.

1.5.2.2.2. Confidential or proprietary information, belonging to either Pace and/or its customers, includes but is not limited to test results, trade secrets, research and development

matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and customer information, inventions, materials composition, etc.

1.5.2.3. **Conflict of Interest**

1.5.2.3.1. Pace employees must avoid situations that might involve a conflict of interest or could appear questionable to others. This includes participation in activities that conflict or appear to conflict with the employees' Pace responsibilities. This would also include offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally (such as bribes, gifts, kickbacks, or illegal payments).

1.5.2.3.2. Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other problematic activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company, or participation in any outside business during the employee's work hours.

1.5.3. Strict adherence by each Pace employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of Pace and to continue the pursuit of our common mission to protect our environment and improve our health.

1.5.4. Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

1.5.5. Compliance: all employees undergo annual Data Integrity/Ethics training which includes the concepts listed above. All employees also sign an annual Ethic Policy statement.

1.6. Anonymous Compliance Alertline

1.6.1. An ethical and safe workplace is important to the long-term success of Pace and the well-being of its employees. Pace has a responsibility to provide a work environment where employees feel safe and can report unethical or improper behavior in complete confidence. With this in mind, Pace has engaged Lighthouse Services, Inc. to provide all employees with access to an anonymous ethics and compliance alertline for reporting possible ethics and compliance violations. The purpose of this service is to ensure that any employee can report anonymously and without fear of retaliation.

1.6.2. Lighthouse Services provides a toll-free number along with several other reporting methods, all of which are available 24 hours a day, seven days a week for use by employees and staff.

1.6.3. Telephone: English speaking USA and Canada: (844)-970-0003.

1.6.4. Telephone: Spanish speaking North America: (800)-216-1288.

1.6.5. Website: www.lighthouse-services.com/pacelabs.

1.6.6. Email: reports@lighthouse-services.com (must include company name with report).

1.7. Laboratory Organization

1.7.1. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office. See Attachment III for the Corporate Organizational structure.

1.7.2. A Senior General Manager (SGM) oversees all laboratories and service centers in their assigned region. Each laboratory or facility in the company is then directly managed by an SGM, a General Manager (GM), an Assistant General Manager (AGM), or an Operations Manager (OM). Quality Managers (QM) or Senior Quality Managers (SQM) at each laboratory report directly to the highest level of local laboratory management, however named, that routinely makes day-to-day decisions regarding that facility's operations. The QMs and SQMs will also receive guidance and direction from the corporate Director of Environmental Quality.

1.7.3. The SGM, GM, AGM or OM, or equivalent functionality in each facility, bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of these managers, the SQM/QM serves as the next in command, unless the manager in charge has assigned another designee. He or she assumes the responsibilities of the manager, however named, until the manager is available to resume the duties of their position. In the absence of both the manager and the SQM/QM, management responsibility of the laboratory is passed to the Technical Director, provided such a position is identified, and then to the most senior department manager until the return of the lab manager or SQM/QM. The most senior department manager in charge may include the Client Services Manager (CSM) or the Administrative Business Manager (ABM) at the discretion of the SGM/GM/AGM/OM.

1.7.4. A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The laboratory SGM/GM/AGM/OM or SQM/QM has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 35 consecutive calendar days, the primary accrediting authority shall be notified in writing.

1.7.5. The SQM/QM has the responsibility and authority to ensure the Quality System is implemented and followed at all times. In circumstances where a laboratory is not meeting the established level of quality or following the policies set forth in this Quality Assurance Manual, the SQM/QM has the authority to halt laboratory operations should he or she deem such an action necessary. The SQM/QM will immediately communicate the halting of operations to the SGM/GM/AGM/OM and keep them posted on the progress of corrective actions. In the event the SGM/GM/AGM/OM and the SQM/QM are not in agreement as to the need for the suspension, the Chief Operating Officer (COO) and Director of Environmental Quality will be called in to mediate the situation.

1.7.6. The technical staff of the laboratory is generally organized into the following functional groups:

- Organic Extractions
- Wet Chemistry Analysis
- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis

1.7.7. The organizational structure for Pace – Indianapolis is listed in Attachment II. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its

accrediting authorities per their individual required timeframes, not to exceed 30 days. The QAM will remain in effect until the next scheduled revision.

1.8. Laboratory Job Descriptions

1.8.1. Senior General Manager

- Oversees all functions of all the operations within their designated region;
- Oversees the development of local GMs/AGMs/OMs within their designated region;
- Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Oversees the preparation of budgets and staffing plans for all operations within their designated region;
- Ensures compliance with all applicable state, federal and industry standards;
- Works closely with Regional Sales Management.

1.8.2. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Presents the Ethics/Data Integrity training annually to all employees in their facilities as an instructor-led training.
- Ensures compliance with all applicable state, federal and industry standards.

1.8.4. Quality Manager

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Environmental Quality;
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors QA/QC activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Environmental Quality office). The QM is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Environmental Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews select laboratory data and final reports;
- Reviews tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Reviews and maintains records of proficiency testing results;

- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors corrective and preventive actions;
- Maintains calibration of support equipment such as balances and thermometers;
- Maintains the currency of the Quality Manual.

1.8.5. **Technical Director**

- Monitors the standards of performance in quality assurance and quality control data;
- Monitors the validity of analyses performed and data generated;
- May review tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Serves as the manager of the laboratory in the absence of the SGM/GM/AGM/OM and SQM/QM;
- Provides technical guidance in the review, development, and validation of new methodologies.

1.8.6. **Administrative Business Manager**

- Responsible for financial and administrative management for the entire facility;
- Provides input relative to tactical and strategic planning activities;
- Organizes financial information so that the facility is run as a fiscally responsible business;
- Works with staff to confirm that appropriate processes are put in place to track revenues and expenses;
- Provide ongoing financial information to the SGM/GM/AGM/OM and the management team so they can better manage their business;
- Utilizes historical information and trends to accurately forecast future financial positions;
- Works with management to ensure that key measurements are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios;
- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.7. **Client Services Manager**

- Oversees all the day to day activities of the Client Services Department which includes Project Management and, possibly, Sample Control;
- Responsible for staffing and all personnel management related issues for Client Services;

- Serves as the primary senior consultant to customers on all project related issues such as set up, initiation, execution and closure;
- Performs or is capable of performing all duties listed for that of Project Manager.

1.8.8. **Project Manager**

- Coordinates daily activities including taking orders, reporting data and analytical results;
- Serves as the primary technical and administrative liaison between customers and Pace;
- Communicates with operations staff to update and set project priorities;
- Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.);
- Works with customers, laboratory staff, and other appropriate Pace staff to develop project statements of work or resolve problems of data quality;
- Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers and maintain customer records;
- Mediation of project schedules and scope of work through communication with internal resources and management;
- Responsible for preparing routine and non-routine quotations, reports and technical papers;
- Interfaces between customers and management personnel to achieve customer satisfaction;
- Manages large-scale complex projects;
- Supervises less experienced project managers and provide guidance on management of complex projects;
- Arranges bottle orders and shipment of sample kits to customers;
- Verifies login information relative to project requirements and field sample Chains-of-Custody;
- Enters project and sample information in the Laboratory Information Management System (LIMS) for scheduling, tracking and reporting purposes.

1.8.9. **Project Coordinator**

- Enters project and sample information in the Laboratory Information Management System (LIMS) for scheduling, tracking and reporting purposes.

1.8.10. **Department Manager/Supervisor**

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Trains analysts or oversees training of analysts in laboratory operations and analytical procedures;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.11. **Quality Assurance Analyst**

- Assists the SQM/QM in the performance of quality department responsibilities as delegated by the SQM/QM;

- Reviews select laboratory data and final reports;
- Generates and reviews QC data validation packages;
- Assists in monitoring QA/QC data;
- Assists in internal audits;
- Assists in maintaining training records;
- Assists in maintaining the document control system.

1.8.12. Group Supervisor/Leader

- Trains analysts in laboratory operations and analytical procedures;
- Organizes and schedules analyses with consideration for sample holding times;
- Implements data verification procedures by assigning data verification duties to appropriate personnel;
- Evaluates instrument performance and supervises instrument calibration and preventive maintenance programs;
- Reports non-compliance situations to laboratory management including the SQM/QM.

1.8.13. Laboratory Analyst

- Performs detailed preparation and analysis of samples according to published methods and laboratory procedures;
- Processes and evaluates raw data obtained from preparation and analysis steps;
- Generates final results from raw data, performing primary review against method criteria;
- Monitors quality control data associated with analysis and preparation. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks;
- Reports data in LIMS, authorizing for release pending secondary approval;
- Conducts routine and non-routine maintenance of equipment as required;
- Performs or is capable of performing all duties associated with that of Laboratory Technician.

1.8.14. Laboratory Technician

- Prepares standards and reagents according to published methods or in house procedures;
- Performs preparation and analytical steps for basic laboratory methods;
- Works under the direction of a Laboratory Analyst on complex methodologies;
- Assists Laboratory Analysts on preparation, analytical or data reduction steps for complex methodologies;
- Monitors quality control data as required or directed. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

1.8.15. Field Technician

- Prepares and samples according to published methods, PACE Quality Assurance Manual and/or customer directed sampling objectives;
- Capable of the collection of representative environmental or process samples;
- Reviews project documentation for completeness, method compliance and contract fulfillment;

- Train less experienced environmental technicians and provide guidance on sampling and analysis;
- Responsible for project initiation and contact follow-up;
- Develop sampling plans and prepare test plan documents.

1.8.16. **Sample Receiving Personnel**

- Signs for incoming samples and verifies the data entered on the Chain of custody forms;
- Stages samples according to EPA requirements;
- Assists Project Managers and Coordinators in filling bottle orders and sample shipments;
- May enter project and sample information in the Laboratory Information Management System (LIMS) for scheduling, tracking and reporting purposes;
- Manages sample storage areas and sample disposal procedures.

1.8.17. **Systems Administrator or Systems Manager**

- Assists with the creation and maintenance of electronic data deliverables (EDDs);
- Coordinates the installation and use of all hardware, software and operating systems;
- Performs troubleshooting on all aforementioned systems;
- Trains new and existing users on systems and system upgrades;
- Maintains all system security passwords;
- Maintains the electronic backups of all computer systems.

1.8.18. **Safety/Chemical Hygiene Officer**

- Maintains the laboratory Chemical Hygiene Plan;
- Plans and implements safety policies and procedures;
- Maintains safety records;
- Organizes and/or performs safety training;
- Performs safety inspections and provides corrective/preventative actions;
- Assists personnel with safety issues.

1.8.19. **Hazardous Waste Coordinator**

- Evaluates waste streams and helps to select appropriate waste transportation and disposal companies;
- Maintains complete records of waste disposal including waste manifests and state reports;
- Assists in training personnel on waste-related issues such as waste handling and storage, waste container labeling, proper satellite accumulation, secondary containment, etc.;
- Conducts a weekly inspection of the waste storage areas of the laboratory.

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through web-based training systems. Employees are provided with several training activities for their particular job description and scope of duties. These training activities may include:

- Hands-on training led by supervisors;
- Job-specific training checklists and worksheets;
- Lectures and instructor-led training sessions;
- Method-specific training;
- External conferences and seminars;
- Reading Standard Operating Procedures (SOPs);
- Reading the Quality Assurance Manual and Safety Manual/Chemical Hygiene Plan;
- Core training modules (basic lab skills, etc.);
- Quality system training modules (support equipment use, corrective actions/root causes, etc.);
- Data Integrity/Ethics training;
- Specialized training by instrument manufacturers;
- On-line courses.

1.9.2. All procedures and training records are maintained and available for review during laboratory audits. Additional information can be found in the *Training Procedures* SOP or its equivalent replacement.

1.10. Laboratory Safety and Waste

1.10.1. It is the policy of Pace to make safety and waste compliance an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety as well as those working in the immediate area by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the local Safety Manual/Chemical Hygiene Plan.

1.11. Security and Confidentiality

1.11.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by Pace staff. Keyless door locks are accessible only to authorized personnel through the use of assigned key fobs. All visitors, including PACE staff from other facilities, must sign the Visitor's Logbook maintained by the receptionist. A staff member will accompany them during the duration of their stay on the premises unless the SGM/GM/AGM/OM, SQM/QM, or Technical Director specify otherwise. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out.

1.11.2. Additional security is provided where necessary, (e.g., specific secure areas for sample, data, and customer report storage), as requested by customers, or cases where national security is of concern. These areas are lockable within the facilities, or are securely offsite. Access is limited to specific individuals or their designees.

1.11.3. All information pertaining to a particular customer, including national security concerns will remain confidential. Data will be released to outside agencies only with written authorization from the customer or where federal or state law requires the company to do so.

1.12. Communications

1.12.1. Management within each lab bears the responsibility of ensuring that appropriate communication processes are established and that communication takes place regarding the effectiveness of the management/quality system. These communication processes may include email, regular staff meetings, senior management meetings, etc.

1.12.2. Corporate management bears the responsibility of ensuring that appropriate communication processes are established within the network of facilities and that communication takes place at a company-wide level regarding the effectiveness of the management/quality systems of all Pace facilities. These communication processes may include email, quarterly continuous improvement conference calls for all lab departments, and annual continuous improvement meetings for all department supervisors, quality managers, client services managers, and other support positions.

2.0. SAMPLE CUSTODY

2.1. Project Initiation

2.1.1. Prior to accepting new work, the laboratory reviews its performance capability. The laboratory confirms that sufficient personnel, equipment capacity, analytical method capability, etc., are available to complete the required work. Customer needs, certification requirements, and data quality objectives are defined and the appropriate sampling and analysis plan is developed to meet the project requirements by project managers or sales representatives. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability, and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or daily operations meetings.

2.1.2. Additional information regarding specific procedures for reviewing new work requests can be found in the *Review of Analytical Requests* SOP or its equivalent replacement.

2.2. Sampling Materials and Support

2.2.1. Each individual Pace laboratory provides shipping containers, properly preserved sample containers, custody documents, and field quality control samples to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VII. Note that all analyses listed are not necessarily performed at all Pace laboratories and there may be additional laboratory analyses performed that are not included in these tables. Customers are encouraged to contact their local Pace Project Manager for questions or clarifications regarding sample handling. Pace may provide pick-up and delivery services to their customers when needed.

2.2.2. Some Pace facilities provide sampling support through a Field Services department. Field Services operates under the Pace Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in SOPs and Procedure Manuals.

2.3. Chain of Custody

2.3.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis.

2.3.2. Field personnel or client representatives must complete a COC for all samples that are received by the laboratory. Samplers are required to properly complete a COC. This is critical to efficient sample receipt and to ensure the requested methods are used to analyze the correct samples. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.3.3. The COC is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the initial entry and initialing and dating the change. All transfers of samples are recorded on the chain of custody in the “relinquished” and “received by” sections. All information except signatures is printed.

2.3.4. Additional information can be found in the *Sample Management* SOP or its equivalent replacement.

2.4. Sample Acceptance Policy

2.4.1. In accordance with regulatory guidelines, Pace complies with the following sample acceptance policy for all samples received.

2.4.2. If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory is required to document all non-compliances, contact the customer, and either reject the samples or fully document any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately communicated to the client.

2.4.3. Sample Acceptance Policy requirements:

- Sample containers must have unique client identification designations that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC).
- There must be clear documentation on the COC, or related documents that lists the unique sample identification, sampling site location, date and time of sample collection, and name of the sample collector.
- There must be clear documentation on the COC, or related documents that lists the requested analyses, the preservatives used, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.).
- Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval.
- Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval.
- Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval.
- Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval.
- All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2°C of the required temperature, or within the method-specified range. For samples with a required temperature of 4°C, samples with a temperature ranging from just above freezing to 6°C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
- Samples for **drinking water compliance** analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are

improperly preserved (with the exception of VOA samples- tested for pH at time of analysis and TOC- tested for pH in the field).

- Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.

Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be read and recorded to ± 0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ± 0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤ 6°C limit. Please reference the Support Equipment SOP for more information.

Note 2: Some microbiology methods allow sample receipt temperatures of up to 10°C. Consult the specific method for microbiology samples received above 6°C prior to initiating corrective action for out of temperature preservation conditions.

2.4.4. Upon sample receipt, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers;
- Sample condition: Intact, broken/leaking, bubbles in VOA samples;
- Sample holding time;
- Sample pH and residual chlorine when required;
- Appropriate containers.

2.4.5. Additional information can be found in the *Sample Management* SOP or its equivalent replacement.

2.5. Sample Log-in

2.5.1. After sample inspection, all sample information on the COC is entered into the Laboratory Information Management System (LIMS). The lab's permanent records for samples received include the following information:

- Customer name and contact
- Customer number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested
- Date and time of laboratory receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

2.5.2. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 08:00 as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

2.5.3. The LIMS automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of 50XXXXXX. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the customer's field identification; it will be a permanent reference number for all future interactions.

2.5.4. Sample labels are printed from the LIMS and affixed to each sample container.

2.5.5. Additional information can be found in the *Sample Management* SOP or its equivalent replacement.

2.6. Sample Storage

2.6.1. Additional information on sample storage can be found in the *Sample Management* SOP or its equivalent replacement and in the *Waste Handling and Management* SOP or its equivalent replacement.

2.6.2. **Storage Conditions**

2.6.2.1. Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

2.6.2.2. Storage blanks are stored with volatile samples and are used to measure cross-contamination acquired during storage. Laboratories must have documented procedures and criteria for evaluating storage blanks, appropriate to the types of samples being stored.

2.6.2.3. Additional information can be found in the *Monitoring Temperature Controlled Units* SOP or its equivalent replacement**.**

2.6.3. **Temperature Monitoring**

2.6.3.1. Samples are taken to the appropriate storage location immediately after sample receipt and check-in procedures are completed. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

2.6.3.2. The temperature of each refrigerated storage area is maintained at $\leq 6^{\circ}C$ but above freezing unless state, method or program requirements differ. The temperature of each freezer storage area is maintained at $\leq -10^{o}C$ unless state, method or program requirements differ. The temperature of each storage area is checked and documented each day of use. If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

- The temperature is rechecked after a period of time, usually two hours, to verify temperature exceedance. Corrective action is initiated and documented if necessary.
- The SQM/QM and/or laboratory management are notified if the problem persists.
- The samples are relocated to a proper environment if the temperature cannot be maintained after corrective actions are implemented.
- The affected customers are notified and/or documentation is provided on the final report, if necessary.

2.6.3.3. Additional information can be found in the *Monitoring Temperature Controlled Units* SOP or its equivalent replacement.

2.6.4. **Hazardous Materials**

2.6.4.1. Samples designated by clients upon receipt as pure product or potentially heavily contaminated samples, or samples found to be designated as such following analysis, must be labeled to indicate the hazard and stored separately from other samples.

2.6.5. **Foreign/Quarantined Soils**

2.6.5.1. Foreign soils and soils from domestic USDA quarantined areas must be adequately segregated to prevent cross-contamination and enable proper sample disposal. The USDA requires these samples and by-products to be properly identified and handled and to be treated by an approved procedure prior to disposal or as part of disposal.

2.6.5.2. Additional information regarding USDA regulations and sample handling can be found in the laboratory's *Regulated Soil Handling* SOP or its equivalent replacement.

2.7. Subcontracting Analytical Services

2.7.1. Every effort is made to perform all analyses for Pace customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory, whether inside or outside the Pace network, becomes necessary, a preliminary verbal communication with that laboratory is undertaken. Customers are notified in writing of the laboratory's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations. When possible, subcontracting will be to a TNI-accredited laboratory.

2.7.2. Potential subcontract laboratories must be approved by Pace based on the criteria listed in SOP S-IN-C-003 *Subcontracting Samples* or its equivalent revision or replacement. All sample reports from the subcontracted labs are appended to the applicable Pace final reports.

2.7.3. Any Pace work sent to other labs within the Pace network is handled as inter-regional work and all final reports are labeled clearly with the name of the laboratory performing the work. Any non-TNI work is clearly identified. Pace will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

2.7.4. Additional information can be found in the *Subcontracting Samples* SOP or its equivalent replacement.

2.8. Sample Retention and Disposal

2.8.1. Samples, extracts, digestates, and leachates must be retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

2.8.2. The minimum sample retention time is 45 days from receipt of the samples. Samples requiring thermal preservation may be moved to ambient temperature storage when the hold time is expired, when the report has been delivered, and/or when allowed by the customer, program, or contract. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

2.8.3. After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposal of **hazardous** samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires Pace to dispose of excess samples, proper arrangements will be made for disposal by an approved contractor.

2.8.4. Additional information can be found in the *Waste Handling and Management* SOP and the *Sample Management* SOP or their equivalent replacements.

3.0. QUALITY CONTROL PROCEDURES

3.1. Quality Control Samples

3.1.1. The quality control samples described in this section are analyzed per batch as applicable to the method used. Acceptance criteria must be established for all quality control samples and if the acceptance criteria are not met, corrective actions must be performed and samples reanalyzed, or the final report must be appropriately qualified.

3.1.2. Quality control samples must be processed in the same manner as associated client samples.

3.1.3. Please reference the glossary of this Quality Manual for definitions of all quality control samples mentioned in this section.

3.1.4. Any deviations to the policies and procedures governing quality control samples must be approved by the QM/SQM.

3.2. Method Blank

3.2.1. A method blank is a negative control used to assess the preparation/analysis system for possible contamination and is processed through all preparation and analytical steps with its associated client samples. The method blank is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples. Method blanks are not applicable for certain analyses (i.e., pH, flash point, temperature, etc.).

3.2.2. Each method blank is evaluated for contamination. Corrective actions for blank contamination may include the re-preparation and re-analysis of all samples (where possible) and quality control samples. Data qualifiers must be applied to results that are affected by contamination in a method blank.

3.2.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for method blanks.

3.3. Laboratory Control Sample

3.3.1. The Laboratory Control Sample (LCS) is a positive control used to assess the performance of the entire analytical system including preparation and analysis. The LCS is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples.

3.3.2. The LCS contains all analytes required by a specific method or by the customer or regulatory agency, which may not include the full list of target compounds. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
    - For methods with 1-10 target compounds, the laboratory will spike with all compounds;
    - For methods with 11-20 target compounds, the laboratory will spike with at least 10 compounds or 80%, whichever is greater;

- For methods with greater than 20 compounds, the laboratory will spike with at least 16 compounds.

3.3.3. The LCS is evaluated against the method default or laboratory-derived acceptance limits. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any sample containing a compound that was 'out-of-control' in the associated LCS must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier. When the result of the LCS exceeds the upper control limit, indicating high bias, associated samples determined to be non-detect may be reported without qualification.

3.3.4. For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary other than proper documentation. TNI has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but within than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

- >90 analytes in the LCS- 5 analytes
- 71-90 analytes in the LCS- 4 analytes
- 51-70 analytes in the LCS- 3 analytes
- 31-50 analytes in the LCS- 2 analytes
- 11-30 analytes in the LCS- 1 analyte
- <11 analytes in the LCS- no analytes allowed out)

3.3.5. A matrix spike (MS) can be used in place of a non-compliant LCS in a batch as long as the MS passes the LCS acceptance criteria. When this happens, full documentation must be made available to the data user. If this is not allowed by a customer or regulatory body, the associated samples must be rerun with a compliant LCS when possible or reported with appropriate data qualifiers.

3.3.6. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for LCSs.

3.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.4.1. A matrix spike (MS) is a positive control used to determine the effect of the sample matrix on compound recovery for a particular method. A matrix spike/matrix spike duplicate (MS/MSD) set or matrix spike/sample duplicate set is processed at a frequency specified in a particular method or as determined by a specific customer request. The MS and MSD consist of the sample matrix that is spiked with known concentrations of target analytes.

3.4.2. The MS and MSD contain all analytes required by a specific method or by the customer or regulatory agency. In the absence of specified components, the laboratory will spike the MS/MSD with the same number of compounds as previously discussed in the LCS section.

3.4.3. A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method or when limited sample volume or weight prohibits the analysis of an MS/MSD set.

3.4.4. The MS and MSD are evaluated against the method or laboratory derived limits. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance; however, is based on method blank and LCS performance, not on MS/MSD recoveries. The spike recoveries give the data user a better understanding of the final results based on their site-specific information.

3.4.5. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for MS/MSDs.

3.5. Sample Duplicate

3.5.1. A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

3.5.2. The sample and duplicate are evaluated against the method or laboratory limits for relative percent difference (RPD). Any duplicate that is outside of these limits is considered to be 'out of control' and must be qualified appropriately.

3.5.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for sample duplicates.

3.6. Surrogates

3.6.1. Surrogates are compounds that reflect the chemistry of target analytes and are added to samples for most organic analyses to measure the extraction efficiency or purge efficiency and to monitor the effect of the sample matrix on surrogate compound recovery.

3.6.2. The surrogates are evaluated against the method or laboratory derived acceptance limits. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systemic error. An exception to this would be samples that have surrogate recoveries that exceed the upper control limit but have no reportable hits for target compounds. These samples would be reported and qualified to indicate the implied high bias would not affect the final results.

3.6.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for surrogates.

3.7. Internal Standards

3.7.1. Internal Standards are method-specific analytes that are added, as applicable, to every standard, QC sample, and client sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes.

3.7.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for internal standards.

3.8. Limit of Detection (LOD)

3.8.1. Pace laboratories use a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. Unless otherwise noted in a published method, the procedure used by Pace laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B, August 28, 2017. All sample processing steps of the preparation and analytical methods are included in the LOD determination including any clean ups.

3.8.2. Additional information can be found in the *Determination of Detection and Quantitation Limits* SOP or its equivalent replacement.

3.9. Limit of Quantitation (LOQ)

3.9.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For Pace laboratories, this LOQ is referred to as the RL, or Reporting Limit. The RL may or may not be based on the lowest calibration standard concentration used in the initial calibration. Results below the lowest calibration level may not be reported without qualification since the results would not be substantiated by a calibration standard. For methods with a determined LOD, results can be reported below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

3.9.2. Additional information can be found in the *Determination of Detection and Quantitation Limits* SOP or its equivalent replacement.

3.10. Estimate of Analytical Uncertainty

3.10.1. Pace can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling or sample matrix. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, Pace laboratories base this estimation on the recovery data obtained from the Laboratory Control Samples (LCS). The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the *Estimation of Measurement Uncertainty* SOP or its equivalent replacement.

3.10.2. The measurement of uncertainty is provided only on request by the customer, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

3.11. Proficiency Testing (PT) Studies

3.11.1. Pace laboratories participate in a defined proficiency testing (PT) program. PT samples are obtained from NIST-approved providers and analyzed and reported a minimum of two times per year for the relevant fields of testing per matrix.

3.11.2. The laboratory initiates an investigation whenever PT results are determined to be ”Not Acceptable” by the PT provider. All findings and corrective actions taken are reported to the SQM/QM or their designee. A corrective action plan is initiated and, when required, this report is sent to the appropriate state accreditation agencies for their review. Additional PTs will be analyzed and reported as needed for certification purposes.

3.11.3. Additional information can be found in the *Proficiency Testing Program* SOP or its equivalent replacement.

3.12. Rounding and Significant Figures

3.12.1. In general, Pace laboratories report data to no more than three significant figures. The rounding rules listed below are descriptive of the LIMS and not necessarily of any supporting program such as Excel.

3.12.2. **Rounding:** Pace - Indianapolis follows the odd / even guidelines for rounding numbers:

- If the figure following the one to be retained is less than five, that figure is dropped and the retained ones are not changed (with three significant figures, 2.544 is rounded to 2.54).
- If the figure following the ones to be retained is greater than five, that figure is dropped and the last retained one is rounded up (with three significant figures, 2.546 is rounded to 2.55).
- If the figure following the ones to be retained is five and if there are no figures other than zeros beyond that five, then the five is dropped and the last figure retained is unchanged if it is even and rounded up if it is odd (with three significant figures, 2.525 is rounded to 2.52 and 2.535 is rounded to 2.54).

3.12.3. **Significant Figures**

3.12.3.1. Pace - Indianapolis observes the following convention for reporting to a specified number of significant figures. Unless specified by federal, state, or local requirements or on specific request by a customer, the laboratory reports:

Values > 10 – Reported to 3 significant figures
Values ≤ 10 – Reported to 2 significant figures

3.13. Retention Time Windows

3.13.1. When chromatographic conditions are changed, retention times and analytical separations are often affected. As a result, two critical aspects of any chromatographic method are the determination and verification of retention times and analyte separation. Retention time windows must be established for the identification of target analytes. The retention times of all target analytes in all calibration verification standards must fall within appropriately determined retention time windows. If an analyte falls outside the retention time window in an ICV or CCV, new absolute retention time windows must be calculated, unless instrument maintenance fixes the problem. New retention time windows must be established when column geometry is affected by maintenance.

3.13.2. Please reference method-specific SOPs for the proper procedure for establishing retention time windows.

3.14. Analytical Method Validation and Instrument Validation

3.14.1. In some situations, Pace develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods are required for specific projects or analytes of interest, when the laboratory develops or modifies a method, or when the laboratory brings new instrumentation online, the laboratory validates the method and/or instrument prior to applying it to customer samples. Method validity is established by meeting criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. The laboratory records the validation procedure, the results obtained and a statement as to the usability of the method. The minimum requirements for method or instrument validation include evaluation of sensitivity, quantitation, precision, bias, and selectivity of each analyte of interest.

3.15. Regulatory and Method Compliance

3.15.1. It is Pace policy to disclose in a forthright manner any detected noncompliance affecting the usability of data produced by our laboratories. The laboratory will notify customers within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

4.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

4.1. Document Management

4.1.1. Additional information can be found in the *Document Control and Management* SOP or its equivalent replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

4.1.2. Pace has an established procedure for managing documents that are part of the quality system.

4.1.3. A master list of managed documents is maintained at each facility identifying the current revision status and distribution of any controlled documents.

4.1.4. Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to the *Document Numbering* SOP or its equivalent replacement.

4.1.5. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for Pace. The base QAM template is distributed by the Corporate Environmental Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template then applicable lab staff will sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis and revised accordingly by the Corporate Quality office.

4.1.6. **Standard Operating Procedures (SOPs)**

4.1.6.1. SOPs are reviewed every two years at a minimum; although, a more frequent review may be required by some state or federal agencies or customers. If no revisions are made based on this review, documentation of the review itself is made by the addition of new signatures on the cover page. If revisions are made, documentation of the revisions is made in the revisions section of each SOP and a new revision number is applied to the SOP. This provides a historical record of all revisions.

4.1.6.2. All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all Pace employees use the most current version of each SOP and provides the SQM/QM with a historical record of each SOP.

4.1.6.3. Additional information can be found in the *Preparation of SOPs* SOP or its equivalent replacement.

4.2. Document Change Control

4.2.1. Additional information can be found in the *Document Control and Management* SOP or its equivalent replacement.

4.2.2. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After

revisions are approved, a revision number is assigned and the previous version of the document is officially retired.

4.2.3. All copies of the previous document are replaced with copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised that there has been a revision and any necessary training is scheduled.

5.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

5.1. Standards and Traceability

5.1.1. Each Pace facility retains pertinent information for standards, reagents, and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation, and use.

5.1.2. Upon receipt, all purchased standard reference materials are recorded into a standard logbook or database and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date, and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

5.1.3. Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, the preparation steps, preparation date, expiration dates, preparer's initials, and a unique Pace identification number. This number is used in any applicable sample preparation or analysis logs so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

5.1.4. Prepared standard or reagent containers include the Pace identification number, the standard or chemical name, and expiration date. The date of preparation, concentration with units, and the preparer's initials can be determined by tracing the standard or reagent ID through the standard log database.

5.1.5. Initial calibrations must be verified with a standard obtained from a second manufacturer or a separate lot prepared independently by the same manufacturer, unless client-specific QAPP requirements state otherwise.

5.1.6. Reference standards and reference materials must be handled, stored, and maintained in a manner that prevents contamination and/or deterioration. Reference standards and reference materials must be stored per manufacturer's recommendations to avoid degradation and stored away from other materials that could contaminate them. Handle reference standards and reference materials with care to avoid evaporation, contamination, degradation or concentration of the material. If it is necessary to package and transport or ship any reference standard or reference material, consult with the manufacturer for proper packaging, labeling and shipping instructions to prevent damage, contamination or deterioration.

5.1.7. Additional information concerning the procurement of standards and reagent and their traceability can be found in the *Standard and Reagent Management and Traceability* SOP or its equivalent replacement.

5.2. General Analytical Instrument Calibration Procedures

5.2.1. Applicable instrumentation are calibrated or checked before use to ensure proper functioning and verify that laboratory, client and regulatory requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated.

5.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in the narrative.

5.2.3. Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets the laboratory's specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported to prevent breakage and is in accordance with the vendor's recommendations.

5.2.4. In the event that recalibration of a piece of test equipment indicates the equipment may have been malfunctioning during the course of sample analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the quality manager. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or taken out of service and replaced.

5.2.5. Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

5.2.6. Please reference the *Calibration Procedures* SOP or its equivalent replacement and SOPs for specific methods for more detailed calibration information.

5.3. Support Equipment Calibration and Verification Procedures

5.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use, as applicable. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until brought back into control. Additional information regarding calibration and maintenance of support equipment can be found in the *Support Equipment* SOP or its equivalent replacement.

5.3.2. On each day of use, balances, ovens, refrigerators, incubators, freezers and water baths are checked in the expected range of use with NIST traceable references in order to ensure the equipment meets laboratory specifications. These checks are documented appropriately.

5.3.3. **Analytical Balances**

5.3.3.1. Each analytical balance is calibrated or verified annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Working calibration weights are ASTM Class 1 or other class weights that have been calibrated against a reference weight set that is re-certified every 5 years, at a minimum, by the manufacturer or other qualified vendor, against a NIST traceable reference. If balances are calibrated by an external vendor, verification of their weights must be

available upon request. All information pertaining to balance maintenance and calibration is recorded on the balance's monitoring log and/or is maintained on file in the local Quality department.

5.3.4. **Thermometers**

5.3.4.1. Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are re-certified every 3 years, at a minimum by the manufacturer or other qualified vendor with equipment directly traceable to NIST.

5.3.4.2. Working thermometers and temperature sensors that are electronic, digital or mechanical are verified against the reference thermometer quarterly according to corporate metrology procedures. Working thermometers that are liquid-in-glass are verified against the reference thermometer annually according to corporate metrology procedures. Alternatively, working thermometers may be replaced with new thermometers in lieu of verification against the reference thermometer or may be verified by the manufacturer or other qualified vendor. Each working thermometer is individually numbered and assigned a correction factor, when applicable, based on comparison with the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and when temperatures are documented.

5.3.4.3. Laboratory thermometer inventory and calibration data are maintained in the local Quality department.

5.3.5. **pH/Electrometers**

5.3.5.1. The meter is calibrated before use each day, at a minimum, using fresh buffer solutions.

5.3.5.2. The pH electrode is inspected daily and cleaned, filled or replaced as needed.

5.3.6. **Spectrophotometers**

5.3.6.1. During use, spectrophotometer performance is checked at established frequencies in analysis sequences against initial calibration verification (ICV) and continuing calibration verification (CCV) standards.

5.3.7. **Mechanical Volumetric Dispensing Devices**

5.3.7.1. Mechanical volumetric dispensing devices including bottle top dispensers dispensing critical volumes, pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis.

5.4. Instrument/Equipment Maintenance

5.4.1. The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs.

5.4.2. Department managers are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

5.4.3. To minimize downtime and interruption of analytical work, preventative maintenance may routinely performed on each analytical instrument. Up-to-date instructions on the use and maintenance of equipment are available to staff in the department where the equipment is used.

5.4.4. Department managers are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

5.4.5. All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation is, unless otherwise stated, identified as a system and not as individual pieces. The laboratory maintains equipment records that include the following:

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service
- Current location in the laboratory
- Condition when received (new, used, etc.)
- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (as applicable)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

5.4.6. All instrument maintenance is documented in maintenance logbooks that are assigned to each particular instrument or system.

5.4.7. The maintenance log entry must include a summary of the problem encountered, the maintenance performed, and an indication that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

5.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily. In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab if possible.

5.5. General Handling, Storage, Maintenance and Transport of Equipment

5.5.1. All support, measurement, and reference equipment must be handled, stored, and maintained in a manner that prevents contamination and/or deterioration. Balances, refrigerators, freezers, incubators, ovens, and hot blocks should be kept clean and free from debris inside and outside. Reference thermometers and reference weight sets must be controlled by the Quality Department, kept in pristine condition and inspected before each use. Working thermometers, weight sets, mechanical pipettes, and bottle top dispensers should be kept clean, inspected for damage before use, and handled properly. When it is necessary to package and transport or ship any support, measurement, or reference equipment to an external vendor for repair, maintenance, calibration, or certification, consult with the external vendor for proper packing, labeling and shipping to prevent damage, contamination, or deterioration.

6.0. CONTROL OF DATA

Analytical results processing, verification, and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user.

All analytical data undergo a documented multi-tier review process prior to being reported to the customer. This section describes procedures used for translating raw analytical data into accurate final sample reports as well as Pace data storage policies.

When analytical data or field data is generated, it is documented appropriately. The resulting logbooks and other laboratory records are kept in accordance with each facility's SOP for documentation storage and archival. The laboratory must ensure that there are sufficient redundant copies of electronic data so that no data is lost due to unforeseen computer issues

6.1. Primary Data Review

6.1.1. The primary analyst is responsible for initial data reduction and data review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting observations or non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. Data review checklists, either hardcopy or electronic, are used to document the primary data review process. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets, LIMS fields, and data review checklists.

6.1.2. The primary analyst compiles the initial data for secondary data review. This compilation must include sufficient documentation for secondary data review.

6.1.3. Additional information regarding data review procedures can be found in the *Data Review Process* SOP or its equivalent replacement, as well as in the *Manual Integration* SOP or its equivalent replacement.

6.2. Secondary Data Review

6.2.1. Secondary data review is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated, and that any non-conformances are properly documented.

6.2.2. The completed data from the primary analyst is sent to a designated qualified secondary data reviewer, which must be someone other than the primary analyst. The secondary data reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations, data quantitation and applicable data qualifiers. The reviewer validates the data entered into the LIMS and documents review and approval of manual integrations. Data review checklists, either hardcopy or electronic, are used to document the secondary data review process.

6.2.3. Additional information regarding data review procedures can be found in the *Data Review Process* SOP or its equivalent replacement, as well as in the *Manual Integration* SOP or its equivalent replacement.

6.3. Data Reporting

6.3.1. Data for each analytical fraction pertaining to a particular Pace project number are released in the LIMS upon validation for assembly into the final report. Anomalies encountered during technical and QC reviews are included in data qualifiers on the final report or in a separate case narrative if there is potential for data to be impacted.

6.3.2. Final reports are prepared according to the level of reporting required by the customer and can be transmitted to the customer via hardcopy or electronic deliverable. A standard Pace final report consists of the following components:

6.3.2.1. A title which designates the report as "Report of Laboratory Analysis";

6.3.2.2. Name and address of laboratory and/or subcontractor laboratories, if used;

6.3.2.3. Phone number and name of laboratory contact to whom questions can be referred;

6.3.2.4. A unique identification number for the report. The pages of the report are numbered and a total number of pages is indicated;

6.3.2.5. Name and address of customer and name of project;

6.3.2.6. Unique laboratory identification of samples analyzed as well as customer sample IDs;

6.3.2.7. Date and time of sample collection, sample receipt and sample analysis;

6.3.2.8. Identification of the test methods used;

6.3.2.9. Qualifiers to the analytical data, if applicable;

6.3.2.10. Identification of whether results are reported on a dry-weight or wet-weight basis;

6.3.2.11. Reporting limits;

6.3.2.12. Final results or measurements;

6.3.2.13. A signature and title, electronic or otherwise, of person accepting responsibility for the content of the report;

6.3.2.14. Date report was issued;

6.3.2.15. A statement clarifying that the results of the report relate only to the samples tested or to the samples as they were received by the laboratory;

6.3.2.16. A statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory;

6.3.3. Any changes made to a final report shall be designated as "Revised" or equivalent wording. The laboratory must keep sufficient archived records of all laboratory reports and revisions. For higher levels of data deliverables, a copy of all supporting raw data is sent to the customer along with a final report of results. Pace will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

6.3.4. Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

6.3.5. The following positions are the only approved signatories for Pace final reports:

- Senior General Manager
- General Manager
- Quality Manager
- Client Services Manager
- Project Manager
- Project Coordinator

6.3.6. Additional information regarding final reports and data deliverables can be found in the *Final Report and Data Deliverable Contents* SOP or its equivalent replacement.

6.4. Data Security

6.4.1. All data including electronic files, logbooks, extraction/digestion/distillation worksheets, calculations, project files and reports, and any other information used to produce the technical report are maintained secured and retrievable by the Pace facility.

6.5. Data Archiving

6.5.1. All records compiled by Pace are archived in a suitable, limited-access environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. TNI-related records will be made readily available to accrediting authorities. Access to archived data is controlled by the Quality Department.

6.5.2. Records that are computer-generated have either a hard copy or electronic backup copy. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

6.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained per the purchase agreement. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

6.6. Data Disposal

6.6.1. Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. Data disposal includes any preliminary or final reports, raw analytical data, logs or logbooks, and electronic files.

7.0. QUALITY SYSTEM AUDITS AND REVIEWS

7.1. Internal Audits

7.1.1. Responsibilities

7.1.1.1. The SQM/QM is responsible for managing, assigning and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The SQM/QM evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the effectiveness of the Quality System as outlined in this manual but may also include other quality programs applicable to an individual laboratory.

7.1.1.2. Additional information can be found in the *Internal and External Audits* SOP or its equivalent replacement.

7.1.2. Scope and Frequency of Internal Audits

7.1.2.1. The complete internal audit process consists of the following four sections, at a minimum:

- Raw Data Review audits- conducted according to a schedule per local SQM/QM. A certain number of these data review audits may be conducted per quarter to accomplish this yearly schedule;
- Quality System audits- considered the traditional internal audit function and includes analyst interviews to help determine whether practice matches method requirements and SOP language;
- Final Report reviews;
- Corrective Action Effectiveness Follow-up

7.1.2.2. Internal systems audits are conducted annually at a minimum. The scope of these audits includes evaluation of specific analytical departments or a specific quality related system as applied throughout the laboratory.

7.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible.

7.1.2.4. Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

7.1.2.5. The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the laboratory, and identification of final reports that were re-issued. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed.

7.1.3. **Internal Audit Reports and Corrective Action Plans**

7.1.3.1. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. The Quality Department auditor writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

7.1.3.2. When audit findings cast doubt on the effectiveness of the operations or on the correctness of validity of the laboratory's environmental test results, the laboratory will take timely corrective action and notify the customer in writing within three business days, if investigations show that the laboratory results may have been affected.

7.1.3.3. Additional information can be found in the *Internal and External Audits* SOP or its equivalent replacement.

7.2. External Audits

7.2.1. Pace laboratories are audited routinely by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

7.2.2. External audit teams review the laboratory to assess the effectiveness of quality systems. The SQM/QM host the external audit team and assist in facilitation of the audit process. After the audit, the external auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM, who provides a written response to the external audit team. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

7.3. Annual Managerial Review

7.3.1. A managerial review of Management and Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements. Additional information can be found in the *Review of Laboratory Management Systems* SOP or its equivalent replacement.

7.3.2. The managerial review must include the following topics of discussion:

- Suitability of policies and procedures
- Reports from managerial personnel
- Internal audit results
- Corrective and preventive actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources, staffing, and safety/waste activities.

7.3.3. This managerial review must be documented for future reference by the SQM/QM and copies of the report are distributed to laboratory staff. Results must feed into the laboratory planning system and must include goals, objectives, and action plans for the coming year. The laboratory shall ensure that any actions identified during the review are carried out within an appropriate and agreed upon timeframe.

8.0. CORRECTIVE ACTION

Additional information can be found in the *Corrective and Preventive Actions* SOP or its equivalent replacement.

During the process of sample handling, preparation, and analysis, during review of quality control records, or during reviews of non-technical portions of the lab, certain occurrences may warrant corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of Pace provides systematic procedures for the documentation, monitoring, completion of corrective actions, and follow-up verification of the effectiveness of these corrective actions. This can be done using Pace's LabTrack system or other system that lists at a minimum, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

8.1. Corrective and Preventive Action Documentation

8.1.1. The following items are examples of sources of laboratory deviations or non-conformances that may warrant some form of documented corrective action:

- Internal Laboratory Non-Conformance Trends
- Proficiency Testing Sample Results
- Internal and External Audits
- Data or Records Review
- Client Complaints
- Client Inquiries
- Holding Time violations

8.1.2. Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency or it may be a more formal documentation. This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

8.1.3. The person who discovers the deficiency or non-conformance initiates the corrective action documentation within LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and any other pertinent information. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

8.1.4. **Root Cause Analysis**: Laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within LabTrack.

8.1.5. Based on the determined root cause(s), the lab implements applicable corrective actions and verifies their effectiveness. In the event that analytical testing or results do not conform to documented

laboratory policies or procedures Project Management will notify the customer of the situation and will advise of any affect to data quality, if applicable.

8.2. Corrective Action Completion

8.2.1. Internal Laboratory Non-Conformance Trends

8.2.1.1. There are several types of non-conformance trends that may occur in the laboratory that would require the initiation of a corrective action report. Laboratories may choose to initiate a corrective action for all instances of one or more of these categories; however, the intent is that each of these would be handled according to its severity; one time instances could be handled with a footnote or qualifier whereas a systemic problem with any of these categories may require an official corrective action process. These categories, as defined in the Corrective Action SOP are as follows:

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- LCS Failure
- Calculation error
- Laboratory accident
- Instrument Failure
- Final Reporting/Data Entry error

8.2.2. PE/PT Sample Results

8.2.2.1. Any PT result assessed as "not acceptable" requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates a replacement PT sample if required. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

8.2.2.2. Additional information, such as requirements regarding time frames for reporting failures to states, makeup PTs, and notifications of investigations, can be found in the *Proficiency Testing Program* SOP or its equivalent replacement.

8.2.3. Internal and External Audits

8.2.3.1. The SQM/QM or designee is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for responding to the auditing body, the root cause of the finding, and the corrective actions needed for resolution. The SQM/QM or designee is also responsible for providing any back-up documentation used to demonstrate that a corrective action has been completed.

8.2.4. **Data Review**

8.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data review, errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

8.2.5. **Client Complaints**

8.2.5.1. Project Managers are responsible for issuing corrective action requests, when warranted, for customer complaints. As with other corrective actions, the appropriate analyst or supervisor begin an investigation to determine possible causes and corrective actions. After potential corrective actions have been determined, the Project Manager reviews the corrective action to ensure all customer needs or concerns are being adequately addressed.

8.2.6. **Client Inquiries**

8.2.6.1. When an error on the customer's final report is discovered, the Project Manager is responsible for initiating a formal corrective action form that describes the failure (e.g., incorrect analysis reported, reporting units are incorrect, or reporting limits do not meet objectives). The Project Manager is also responsible for revising the final report if necessary and submitting it to the customer.

8.2.7. **Holding Time Violations**

8.2.7.1. In the event that a holding time has been exceeded due to laboratory error, the analyst or supervisor must complete formal corrective action. The Project Manager and the SQM/QM must be made aware of all holding time violations due to laboratory error.

8.2.7.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the out-of-hold sample and the ultimate resolution is then documented and included in the customer project file.

9.0. GLOSSARY

The source of some of the definitions is indicated previous to the actual definition (e.g., TNI, DoD).

Terms and Definitions	
3P Program	The Pace continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all Pace labs.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program.
Accuracy	TNI- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	A discrete, measured, representative portion of a sample taken for analysis.
American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.

Analyst	TNI- The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed.
Analytical Method	A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Annual (or Annually)	Defined by Pace as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation).
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	TNI- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI - A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank).

Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.
Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.

Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Confirmation	TNI- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)

Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	Action taken to eliminate a detected non-conformity.
Corrective Action	The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence (1 – α) that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.
Detection Limit (DL)	The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.
Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	Deuterated compounds used as surrogates for GC/MS analysis.

Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.

Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.
Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.

Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.
In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.

Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Internal Standard	TNI - A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Laboratory Information Management System (LIMS)	The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
LabTrack	Database used by Pace to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
Linear Dynamic Range	Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
May	EPA – The word "may" is used to provide guidance on aspects of the method that are useful but not essential.
Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).
Measurement Uncertainty	An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.

Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, 1 – β, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
MintMiner	Program used by Pace to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
Must	EPA – The word "must" is used to indicate aspects of the method that are considered essential to its performance, based on sound analytical practices.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.

Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.

Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System	TNI - A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quantitation Range	The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.
Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a “standard method”, that term is equivalent to “reference method”). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.

Reporting Limit (RL)	The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL.
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.
Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a chain-of-custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.

Shall	EPA – The word "shall" is used to indicate aspects of the method that are considered essential to its performance, based on sound analytical practices.
Should	EPA – The word "should" is used to provide guidance on aspects of the method that are useful but not essential.
Signal-to-Noise Ratio (S/N)	A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

Surrogate	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where k > 1).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.
Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

10.0. REFERENCES

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11.0. REVISIONS

The Pace Corporate Environmental Quality Office files an electronic version of a Microsoft Word document with tracked changes detailing all revisions made to previous versions of the Quality Assurance Manual. This document is available upon request. All current revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual 19.0	General: made administrative edits that do not affect the policies or procedures within the document (including revising company name to Pace Analytical Services, LLC). Cover page: removed corporate approval signature lines and revised document control format. Table of Contents: added Attachment VII – Pace COC Old Section 3: moved to other sections of the QAM as applicable and deleted entire section (All section references below reflect the new section numbers). Section 1.1.2: replaced with section 3.1.1. Sections 1.3, 1.4, 1.11: removed extraneous language. Sections 1.5: added language from old section 1.6. Section 1.6: revised anonymous reporting information. Section 1.8: removed job descriptions for non-applicable personnel. Section 1.8.4: added tasks to QM job description. Section 1.8.8: added tasks to PM job description. Section 1.11.1: added keyless entry using key fobs detail. Section 2: rearranged existing sections. Section 2.4: reworded to match existing Sample Acceptance policy document. Section 2.6.3.2: added some detail regarding temperature monitoring corrective action. Section 2.6.5.1: added by-products of USDA soils. Section 3.2.2: added basic evaluation criteria. Section 3.4.3: added MS and Dup as optional alternative to MS/MSD. Section 3.5.2: added basic evaluation criteria. Section 3.9.1: added that RL may be based on calibration standard. Section 3.14: added new instrumentation as requiring validation. Section 4: in general, for each QC type, removed language regarding frequency and corrective actions and referenced lab-specific SOPs. Section 5: in general, removed extraneous language and Management of Change section. Section 5.1, 5.2: reorganized into Primary and Secondary Review sections and removed extraneous language. Section 5.3.2: specified types of support equipment to be monitored daily. Section 5.3.3.1: specified “working” weights. Section 5.3.4.2: added temperature sensors and added alternatives to annual in-house verification. Section 5.3.5: added pH electrode inspection/maintenance. Section 6: removed extraneous language including Quarterly Report section. Section 8.2.3.1: added “or designee”. Section 9 (glossary): revised and added definitions based on 2016 TNI Standard. Added “may, must, shall and should” based on SW-846 definition. Section 10: Added EPA DW Manual and revised references as applicable. Attachment III: updated corporate organizational chart. Old Attachment IV: removed floor plan attachment. Old Attachment VII: removed COC (available in SOPs). Indy added back in.	22Mar2017

Document Number	Reason for Change	Date
Quality Assurance Manual 19.1	Throughout the document, references to SOP numbers were removed leaving only SOP titles. Section 1.8.9: added for Project Coordinator position. Section 2.4.3: changed "drinking water" to "drinking water compliance" for clarity. Section 2.6.4.1: clarified hazardous sample labeling. Section 3.8.1: updated the 40 CFR Part 136 reference. Section 3.12.1: removed language that limits the use of 3 sig figs. Section 5.1.6: added section to generally cover handling, storage, and transport of reference standards and reference materials. Section 5.2: removed details and added reference to Calibration Procedures SOP. Section 5.3.4: updated to reflect quarterly digital/mechanical thermometer calibration. Section 5.5: added section to generally cover handling, storage, maintenance and transport of measurement equipment. Section 6.3.1: clarified data review anomalies will be qualified or narrated. Section 6.3.2.1: updated to include the actual name of the final report. Section 8.2.2.1: added "calculation error" as a possible type of non-conformance. Glossary: updated definition of Deuterated Monitoring Compounds, removed DoD references, and updated the definition of Reporting Limit (RL). Attachment II: updated Attachment III: updated Attachment VI: updated Attachment V: updated Attachment VI: updated	14Jun2018

ATTACHMENT I- QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:
TrueValue = Amount spiked (can also be the $\overline{CF}$ or $\overline{RF}$ of the ICAL Standards)
Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentrtion} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:
R1 = Result Sample 1
R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^{N} W_i * (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} W_i * (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} W_i * (Y_i - \overline{Y})^2\right)}}$$

With:	N	Number of standard samples involved in the calibration
	i	Index for standard samples
	Wi	Weight factor of the standard sample no. i
	Xi	X-value of the standard sample no. i
	X(bar)	Average value of all x-values
	Yi	Y-value of the standard sample no. i
	Y(bar)	Average value of all y-values

ATTACHMENT I- QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{(n-1)}}$$

where:
n = number of data points
X_i = individual data point
$\overline{X}$ = average of all data points

AVERAGE ($\overline{X}$)

$$\overline{X} = \frac{\sum_{n=1}^{i} X_i}{n}$$

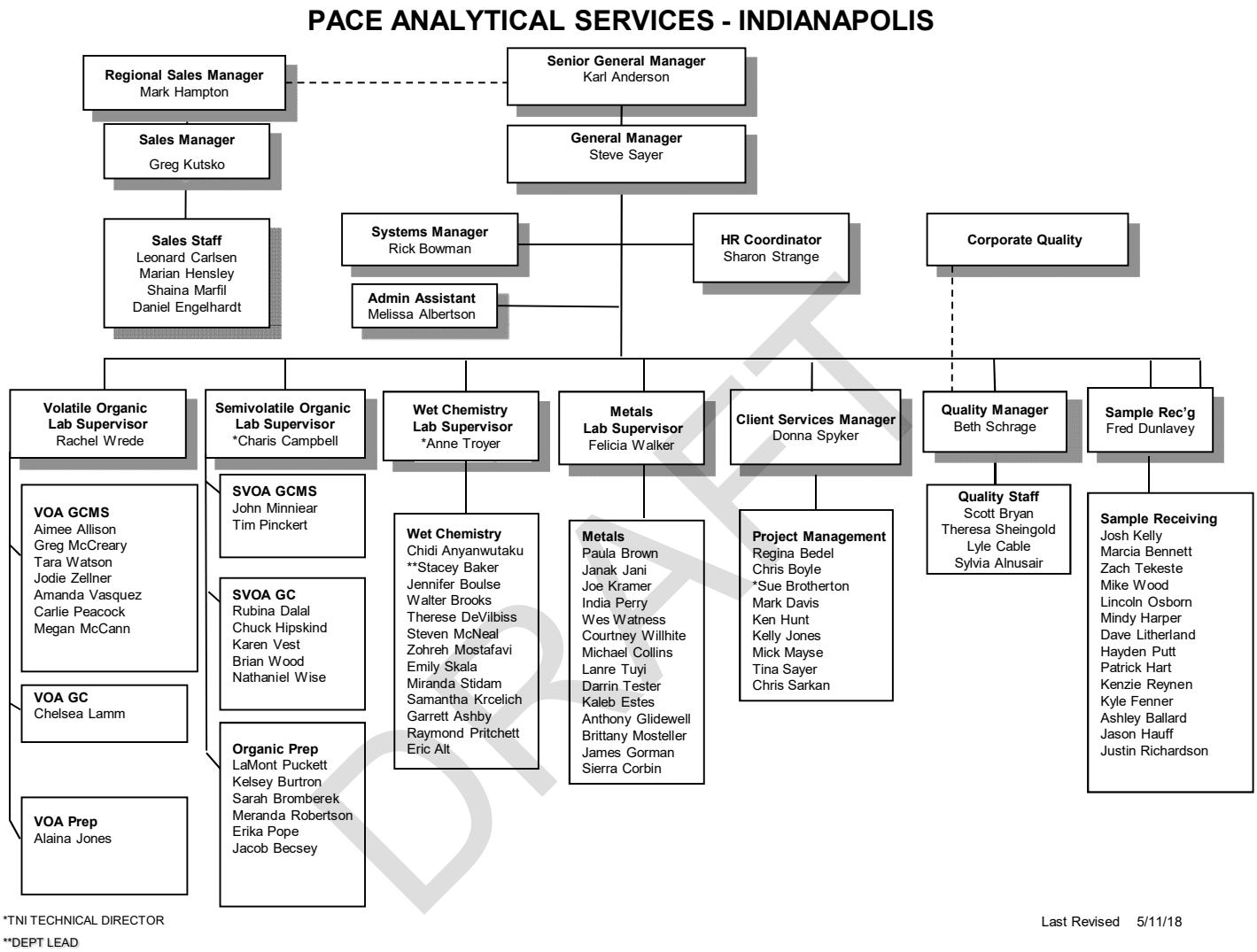
where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

$$RSD = \frac{S}{\overline{X}} * 100$$

where:
S = Standard Deviation of the data points
$\overline{X}$ = average of all data points

ATTACHMENT II- LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)

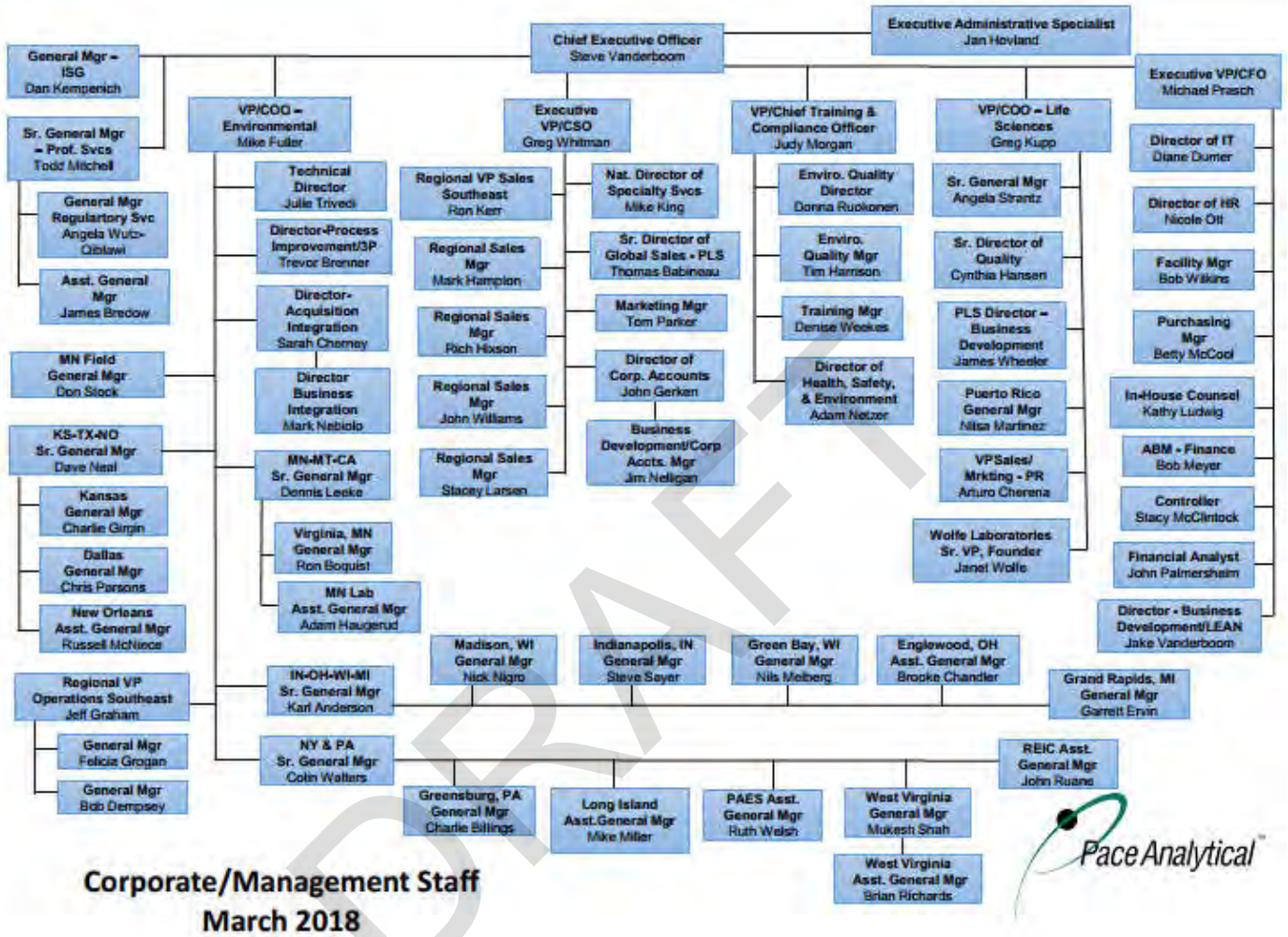


*TNI TECHNICAL DIRECTOR

**DEPT LEAD

Last Revised 5/11/18

ATTACHMENT III- CORPORATE ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



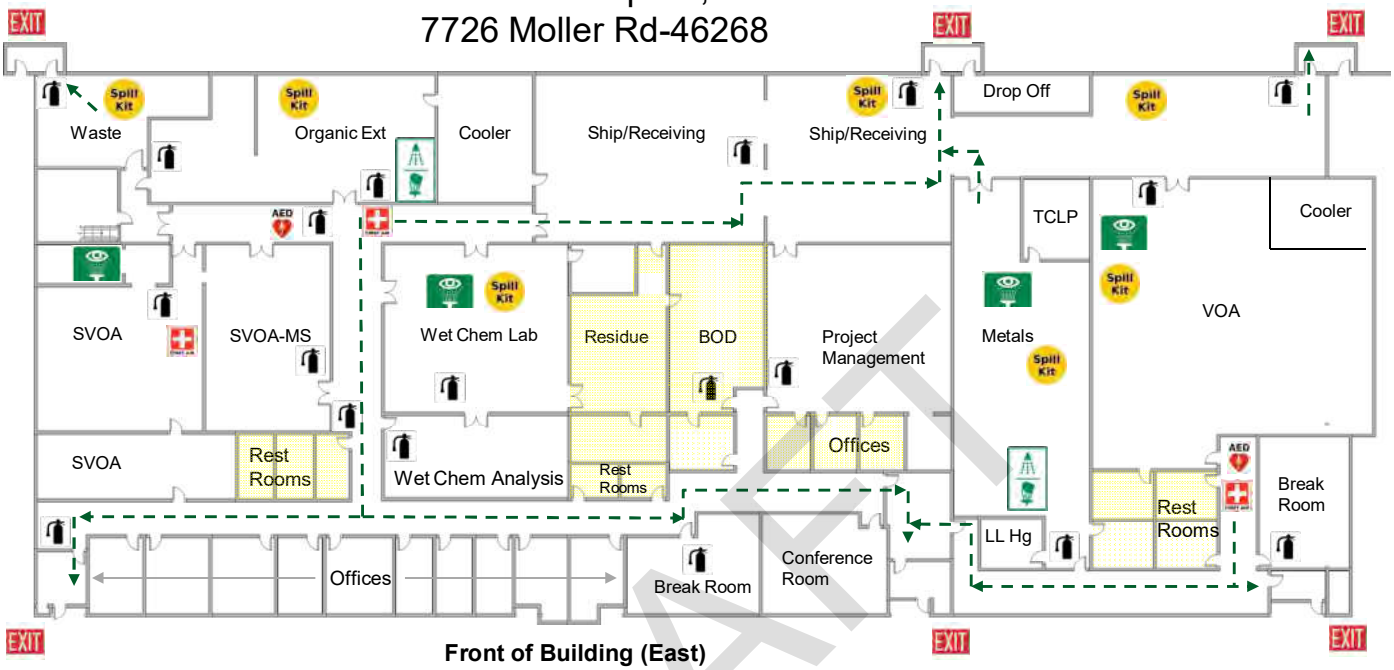
ATTACHMENT IV- EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

Pace Indianapolis Equipment/Instrumentation List

INSTRUMENT	MANUFACTURER	MODEL NUMBER	DETECTOR	AUTOSAMPLER	SERVICE ANALYSIS	YEAR
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2003
GC/MS	Agilent	6890	MS 5973	Centurion	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2003
GC/MS	Agilent	6850N	MS 5975	Centurion	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	MS 5973	Centurion W/S	8260/624 VOC	2004
GC/MS	Agilent	6850N	MS 5975	Centurion	8260/624 VOC	2010
GC/MS	Agilent	6890	MS 5973	OI	8260/624/524.2 VOC	2007
GC/MS	Agilent	7890	MS 5975C	Archon	8260	2008
GC/MS	Agilent	6890	MS 5975	OI	8260/624/524.2 VOC	2007
GC/MS	Agilent	6890	5975	Centurion	8260/624 VOC	2008
GC/MS	Hewlett-Packard	6890	MS 5973	7683	8270 PAH SIM	2000
GC/MS (2)	Agilent	7890	MS 5975	7683	8270/625 BNA	2008
GC/MS (2)	Agilent	6890	MS 5975	7683	8270 PAH SIM	2009
GC/MS (3)	Agilent	6890	MS 5973	7683	8270/625 BNA	2008
GC/MS	Agilent	7890	MS 5975	7683	8270 PAH SIM	2009
GC/MS (2)	Hewlett-Packard	5890	MS 5971	7673	Solvent Screen	2007
GC/MS	Agilent	7890B	MS 5977	7693	8270/PAH SIM	2017
GC/MS	Agilent	7890B	MS 5977	7693	8270/PAH SIM	2018
Gas Chromatograph	Agilent	6890	FID	7683	8015 Alcohols	2006
Gas Chromatograph	Hewlett-Packard	6890	FID	6890	8015 Glycols	2008
Gas Chromatograph	Agilent	7890A	FID	7693	8015 DRO/ERO	2009
Gas Chromatograph	Agilent	7890A	Dual ECD	7693	8082/608 PCBs/8011 EDB/DBCP	2009/2013
Gas Chromatograph	Hewlett-Packard	5890	FID	6890	Benzene	2006
Gas Chromatograph	Hewlett-Packard	5890	FID	8100	8015 GRO	2011
Gas Chromatograph	Hewlett-Packard	5890	FID	EST LGX50	RSK175 Dissolved gases	2006
Gas Chromatograph	Agilent	6890N	FID	8100	8015 GRO	2008
Gas Chromatograph	Agilent	6890	Dual NPD	7683	Pesticides	2008
Gas Chromatograph (2)	Agilent	6890	Dual ECD	7683	PCBs	2008
Gas Chromatograph	Hewlett-Packard	6890	Dual ECD	7683	Herbicides	2008
Gas Chromatograph	Agilent	7890	Dual ECD	7693	Pesticides	2010
Microwave Extractors (2)	CEM	230/60	n/a	n/a	soil extraction	2008/2011
Spe-Dex	Horizon	4790	n/a	n/a	1664A Oil & Grease	2008
Trace ICP (2)	Thermo Scientific	ICAP 6500	n/a	ASX520	6010/200.7 Metals	2008/2011
Trace ICP	Thermo Scientific	ICAP 6500	n/a	ESI SC-4 FAST	6010/200.7 Metals	2011
ICP/MS	Agilent	7700	n/a	ASX520	6020/200.8 Metals	2012
ICP/MS	Agilent	7800	n/a	ASX520	6020/200.8 Metals	2018
Mercury Analyzer	CETAC	M-6100	n/a	ASX520	7470/7471/245 Mercury	2012/2010
Mercury Analyzer	Teledyne Leeman	M-7600	n/a	ASX520	7470/7471/245 Mercury	2016
Low-Level Mercury Analyzer	CETAC	M-8000	n/a	ASX520	Low-Level Mercury	2015
Auto Analyzer (2)	Lachat	Quick Chem	n/a	n/a	NO3,Cl,Phenol, NH3,TKN	2010/2012
Titrosampler	Metrohm	855	n/a	n/a	Alkalinity, Acidity	2014
Automated Flash Point	Tanaka	APM-8	n/a	n/a	flash point	2010
Spectrophotometer	Spec 20	Labtronics	n/a	n/a	Sulfide	2002
Spectrophotometer	Hach	DR5000	n/a	n/a	Sulfate,Cr6+,Fe2+, PO4	2007
Spectrophotometer	Thermo	AquaMatePlus	n/a	n/a	Surfactants, COD	2005
Turbidimeter	Hach	2100P	n/a	n/a	Turbidity	2006
pH/ISE Meter (2)	Accumet	AR25/XL25	n/a	n/a	pH, Fluoride, Redox	2003/2010
pH/ISE Meter	Thermo Orion Star	A214	n/a	n/a	pH, Fluoride, Redox	2013
Conductivity Meter	Oakton	CON 700	n/a	n/a	Conductivity	2016
Dissolved Oxygen/pH Meter	Hach	HQ440d	n/a	n/a	BOD, cBOD	2014
BOD Analyzer	Thermo	AutoEz	n/a	n/a	BOD, cBOD	2013
TOC Analyzer	Shimadzu	TOC-Vwp	n/a	n/a	TOC, DOC	2008
TOC Analyzer	Teledyne	Phoenix 8000	n/a	n/a	TOC, DOC	2005
Discrete Analyzer	Smart Chem	200	n/a	n/a	Cyanide, Phosphorus	2006
Ion Chromatogram	Dionex	IC3000	n/a	AS-1	Cl-, F-, SO4-, Br-, NO3/NO2	2008
Ion Chromatogram	Dionex	ICS2100	n/a	AS-AP	Cl-, F-, SO4-, Br-, NO3/NO2	2013

ATTACHMENT V- LABORATORY FLOOR PLAN (CURRENT AS OF ISSUE DATE)

Pace Indianapolis, IN
7726 Moller Rd-46268



= Automated External defibrillator

= First Aid Station

= Spill Kits

= Shower/Eyewash Combo

= Eyewash/Drench Hose Available

= ABC Extinguisher Location

= Severe Weather Shelters

Fire Evacuation Meeting Place
(Front Parking lot. Greenway by road between main entrance and south entrance)

Effective:
3/26/18

ATTACHMENT VI- LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE)

Pace Analytical Services, LLC
Indianapolis Laboratory Certifications

Accrediting Authority	Program Category	Accrediting Agency	Accreditation #	Expiration Date
Illinois (Secondary TNI)	Hazardous Waste	IL-EPA	200074	10/12/2018
Illinois (Secondary TNI)	Non-Potable Water	IL-EPA	200074	10/12/2018
Indiana	Drinking Water	ISDH	C-49-06	12/31/2021
Kansas (Primary TNI)	Hazardous Waste	KDHE	E-10177	06/30/2018
Kansas (Primary TNI)	Non-Potable Water	KDHE	E-10177	06/30/2018
Kentucky	UST	KDEP	80226	06/30/2018
Kentucky	Wastewater	KDEP	KY98019	12/31/2018
Ohio VAP	Hazardous Waste	OH-EPA	CL0065	01/10/2020
Ohio VAP	Non-Potable Water	OH-EPA	CL0065	01/10/2020
Oklahoma	Non-Potable Water	OK DEQ	9204	08/31/2018
Oklahoma	Solids	OK DEQ	9204	08/31/2018
Texas (Secondary TNI)	Non-Potable Water	TX CEQ	T104704355	01/31/2019
Texas (Secondary TNI)	Solid Chemical Mat.	TX CEQ	T104704355	01/31/2019
USDA	Compliance Agreement	USDA	IN-16-SL-FR-002	05/04/2019
USDA	Foreign Soil Permit	USDA	P330-16-00257	08/19/2019
West Virginia	Hazardous Waste	WV-DEP	330	10/31/2018
West Virginia	Non-Potable Water	WV-DEP	330	10/31/2018
Wisconsin	Non-Potable Water	WI DNR	999788130	08/31/2018
Wisconsin	Waste, Soil, Tissue	WI DNR	999788130	08/31/2018

ATTACHMENT VII- PACE CHAIN-OF-CUSTODY (CURRENT AS OF ISSUE DATE)

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Pace Analytical® www.pacelabs.com

Section A — Required Client Information: Company: Address: Email To: Phone: Fax: Requested Due Date/TAT:

Section B — Required Project Information: Report To: Copy To: Purchase Order No.: Project Name: Project Number:

Section C — Invoice Information: Attention: Company Name: Address: Pace Quote Reference: Pace Project Manager: Pace Profile #:

Page: of

REGULATORY AGENCY: NPDES GROUNDWATER DRINKING WATER OTHER; UST RCRA; Site Location STATE:

Section D — Required Client Information

SAMPLE ID (A-Z, 0-9 / ,) Sample IDs MUST BE UNIQUE

Matrix Codes (MATRIX / CODE): Drinking Water DW; Water WT; Waste Water WW; Product P; Soil/Solid SL; Oil OL; Wipe WP; Air AR; Tissue TS; Other OT

ITEM #

MATRIX CODE (see valid codes to left)

SAMPLE TYPE (G=GRAB C=COMP)

COLLECTED: COMPOSITE START DATE / TIME; COMPOSITE END/GRAB DATE / TIME

SAMPLE TEMP AT COLLECTION

OF CONTAINERS

Preservatives: Unpreserved; H_2SO_4; HNO_3; HCl; NaOH; $Na_2S_2O_3$; Methanol; Other

Analysis Test ↓ (Y/N)

Requested Analysis Filtered (Y/N)

Residual Chlorine (Y/N)

Pace Project No./ Lab I.D.

RELINQUISHED BY / AFFILIATION — DATE — TIME

ACCEPTED BY / AFFILIATION — DATE — TIME

SAMPLE CONDITIONS: Temp in °C; Received on Ice (Y/N); Custody Sealed Cooler (Y/N); Samples Intact (Y/N)

ADDITIONAL COMMENTS

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER:

SIGNATURE of SAMPLER:

DATE Signed (MM/DD/YY):

ATTACHMENT VIII- METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE (CURRENT AS OF ISSUE DATE)

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acid Base Accounting	Sobek	Solid	Plastic/Glass	None	N/A
Acidity	SM2310B	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	$\leq 6^oC$	14 Days
Actinides	HASL-300	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid	Plastic/Glass	None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass (NY requires separate bottle filled to the exclusion of air)	$\leq 6^oC$	14 Days
Alkylated PAHs		Water	1L Amber Glass	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid	8oz Glass	$\leq 10^oC$	1 Year/40 Days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	$\leq 6^oC$; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	$\leq 6^oC$	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	$\leq 6^oC$	48 hours
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Asbestos	EPA 600/R-93/116	Solid	Plastic/Glass; bulk- 2" square; popcorn ceiling- 2tbsp; soil- 4oz	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	$\leq 6^oC$; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	$\leq 6^oC$	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; $\leq 6^oC$; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	$\leq 6^oC$; pH<2 1:1 HCl (optional)
Biomarkers		Solid	$\leq 10^oC$	1 Year/40 Days	$\leq 10^oC$
BOD/cBOD	SM5210B	Water	Plastic/Glass	$\leq 6^oC$	48 hours
Boiling Range Distribution of Petroleum Fractions	ASTM D2887-98	Product	10mL glass vials	$\leq 6^oC$	N/A
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	28 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	72 Hours
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloroacetic acid pH <3; $\leq 6^oC$	28 Days
Carbamates	8318	Water	Glass	Monochloroacetic acid pH 4-5; $\leq 6^oC$	7/40 Days
Carbamates	8318	Solid	Glass	$\leq 6^oC$	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Carbon Specific Isoptope Analysis (CSIA)	AM24	Water	40mL clear VOA vial with TLS	≤ 6°C, trisodium phosphate or HCl	N/A
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Cations (Ferrous Iron, Ferric Iron, Divalent Manganese)	7199 modified	Water	40mL clear VOA vials with mylar septum	≤ 6°C; HCl	48 Hours
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorinated Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil	≤ 6°C	48 Hours to filtration
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Fecal	SM9221E	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9221E	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Total	SM9222B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total	SM9221B	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total, Fecal and E. coli	Colilert/ Quanti-tray	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	30 Hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/ 9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present- applies to SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics- TPH DRO	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6°C	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C; pH <2 HCl	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	7/40 Days
Diquat/Paraquat	549.2	Water	Amber Plastic	≤ 6°C; $Na_2S_2O_3$	7/21 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Endothall	548.1	Water	Amber Glass	≤ 6°C; $Na_2S_2O_3$	7/14 Days
Enterococci	EPA 1600	Water	100mL Plastic	≤ 10°C	8 Hours
Enterococci	Enterolert	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Water	1L Amber Glass	pH < 2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Solid	4oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	Glass	None	Immediate

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Flashpoint/ Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	$\leq 6^oC$; pH <2 H_2SO_4 or HCl	7/40 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics (C3-C10)	8260B modified	Water	40mL vials	$\leq 6^oC$; HCl	14 Days
Gasoline Range Organics (C3-C10)	8260B modified	Solid	4oz Glass Jar	$\leq 6^oC$	14 Days
Gasoline Range Organics- Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics- Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	14 Days
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	$\leq 6^oC$; packed jars with no headspace	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	$\leq 6^oC$ in MeOH	21 Days
Glyphosate	547	Water	Glass	$\leq 6^oC$; $Na_2S_2O_3$	14 Days (18 Months frozen)
Grain Size	ASTM D422	Solid	Not specified	Ambient	N/A
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; $\leq 6^oC$	14/7 Days if extracts stored $\leq 6^oC$ or 14/14 Days if extracts stored at $\leq -10^oC$

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/ SM3500Cr-B, C	Water	Plastic/Glass	$\leq 6^oC$	24 Hours (see note 4)
Hexavalent Chromium	218.6/SM3500Cr-B, C	Water	Plastic/Glass	Ammonium Buffer pH 9.3-9.7	28 Days (see note 4)
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH >8	14 Days (see note 4)
Hexavalent Chromium	7196 (with 3060A)	Solid	Glass	$\leq 6^oC$	30 Days from collection to extraction and 7 days from extraction to analysis
Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Hydrogen by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Light Hydrocarbons by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Light Hydrocarbons in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Lipids	Pace Lipids	Tissue	Plastic/Glass	$\leq$ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	$\leq$ 6°C	28 Days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	$\leq$ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175; PM01/AM20GAx	Water	20mL vials	HCl; or trisodium phosphate or benzalkonium chloride and $\leq$ 6°C	14 Days; 7 Days unpreserved
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	28 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
Methanol, Ethanol	8015 modified	Water	40mL vials	$\leq$ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	$\leq$ 6°C	14 Days
Methyl Mercury	1630	Water	Teflon/ fluoropolymer	Fresh water- 4mL/L HCl; Saline water- 2mL/L H_2SO_4 (must be preserved within 48 hours of collection)	6 months

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Methyl Mercury	1630	Tissue	2-4oz glass jar	≤ 0°C	28 Days; ethylated distillate 48 hours
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	28 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	72 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
Oil Range Organics	8015	Solid	Glass	≤ 6°C	14/40 Days
Oil Range Organics	8015	Water	Glass	≤ 6°C	7/40 Days
Organic Matter	ASA 29-3.5.2	Solid	Plastic/Glass	None; samples air-dried and processed prior to analysis	N/A
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Oxygenates on Product (GCMS SIM)	1625 modified	Product	10mL glass vial	≤ 6°C	14 Days (7 Days from extraction)
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	Pest: 7/40 Days; PCB: 1 Year/1 Year
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3; pH<2 HCl; $\leq 6^oC$	14/30 Days
PCBs, total as Decachlorobiphenyl	508A	Water	1L Glass, TFE lined cap	$\leq 6^oC$	14/30 Days
Perchlorate	331	Water	Plastic/Glass	$\geq$0-6^oC, field filtered with headspace	28 Days
Permanent Gases (O2, N2, CO2)	RSK-175; PM01/AM20GAx	Water	40mL vials	benzalkonium chloride and $\leq 6^oC$	14 Days
Permanent Gases by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Permanent Gases in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	$\leq -10^oC$	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	$\leq 6^oC$	1 Year/1 Year
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	$\leq -10^oC$	1 Year if frozen/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particle Size	ASA 15-5 modified	Solid	Plastic/Glass (100g sample)	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	28 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	7 Days
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Purgeable Organic Halides (POX)	9021	Water	Glass; no headspace	≤ 6°C	14 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid	Plastic/Glass		
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days
Saturated Hydrocarbons		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Saturated Hydrocarbons		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/9038/375.2/ASTM D516	Water	Plastic/Glass	$\leq 6^oC$	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; $\leq 6^oC$	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid	Plastic/Glass	None	180 days
Total Inorganic Carbon (TIC)	PM01/AM20GAx	Water	40mL VOA vial with mylar septum	$\leq 6^oC$	14 Days
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; $\leq 6^oC$	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black/Lloyd Kahn	Solid	Glass	$\leq 6^oC$	14 Days
Total Organic Halogen (TOX)	SM5320/9020	Water	Glass; no headspace	$\leq 6^oC$	14 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Water	40mL vials	pH<2 HCl, no headspace, $\leq 6^oC$	7 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Solid	Glass	$\leq 6^oC$	14 days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
UCMR Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days
UCMR Hexavalent Chromium	218.7	Water	HDPE or propylene	$Na_2CO_3/NaHCO_3$/ $(NH_4)_2SO_4$; pH>8	14 Days
UCMR Chlorate	300.1	Water	Plastic or glass	EDA	28 Days
UCMR Perfluorinated Compounds	537	Water	Polypropylene	Trizma	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
UCMR 1, 4 Dioxane	522	Water	Glass	Na_2SO_3, $NaHSO_4$; pH<4	28 Days
UV254	SM5910B	Water	Glass	≤ 6°C	48 Hours
Vermiculite	EPA 600/R-93/116	Solid	Plastic/Glass	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Volatile Fatty Acids	AM21G	Water	40mL clear VOA vials	≤ 6°C	21 Days
Volatile Fatty Acids (low level)	AM23G	Water	40mL clear VOA vials	≤ 6°C with benzalkonium chloride	14 Days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C; packed jars with no headspace	7/28 Days
Volatiles	TO-14	Air	Summa Canister	None	28 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	TO-15	Air	Summa Canister or Tedlar Bag	None	28 Days
Volatiles	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Volatiles	TO-18/8260	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	8260	Solid	5035 vial kit	See note 1 (analyze for acrolein and acrylonitrile per local requirements)	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (or unpreserved if run within 7 days of collection) (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6°C; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days
Whole Oil	ASTM D3328 (prep); ASTM D5739	Product	10mL glass vials	≤ 6°C	N/A

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4°C, **and** one vial methanol preserved at ≤6°C **and** one container of unpreserved sample stored at ≤6°C.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA Method Update Rule. Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate buffer to extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR program.

ATTACHMENT A-2

QUALITY ASSURANCE MANUAL, QUALITY ASSURANCE/QUALITY CONTROL POLICIES AND PROCEDURES, PACE ANALYTICAL SERVICES, LLC-PITTSBURGH

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Pace Analytical	**Document Name:** **Quality Assurance Manual**	**Document Revised: May 18, 2018** **Effective Date of Final Signature** **Page 1 of 103**
	Document No.: **Quality Assurance Manual Rev. 19.1**	**Issuing Authorities:** **Pace Pittsburgh Quality Office**

QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures
Pace Analytical Services, LLC – Pittsburgh
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Phone: 724-850-5600

APPROVAL

05/18/18
Date

William Billings
Laboratory General Manager
724-850-5610

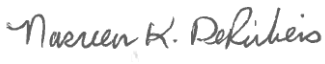

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Table of Contents

1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"
Pace Analytical Services LLC - Mission Statement

1.1. Introduction to Pace

1.1.1. Pace Analytical Services, LLC is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. Pace offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. This document defines the Quality System and Quality Assurance (QA)/Quality Control (QC) protocols.

1.1.2. Pace laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Methods are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 of this document is a representative listing of general analytical protocol references.

1.2. Statement of Purpose

1.2.1. To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. Pace management is committed to maintaining the highest possible standard of service and quality for our customers by following a documented quality system that is compliant with all current applicable state, federal, and industry standards, such as the NELAC Standard, the TNI Standard, and ISO standards and is in accordance with the stated methods and customer requirements. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the Pace network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work.

1.3.3. All personnel must comply with all current applicable state, federal, and industry standards (e.g., 2003 NELAC Standard, 2009 TNI Standards, ISO/IEC 17025 standard, DOD, etc.), and are required to perform all tests in accordance with stated methods and customer requirements. When required, lab shall also comply with the program requirements for 10CFR50, Appendix B when performing safety related tests on materials used for nuclear facilities.

1.4. Core Values

1.4.1. The following are the Pace Core Values:

- **Integrity**
- **Value Employees**

- **Know Our Customers**
- **Honor Commitments**
- **Flexible Response To Demand**
- **Pursue Opportunities**
- **Continuously Improve**

1.5. Code of Ethics and Standards of Conduct

1.5.1. **Code of Ethics**:

1.5.1.1. Each Pace employee is responsible for the propriety and consequences of his or her actions;

1.5.1.2. Each Pace employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where Pace does business or seeks to do business;

1.5.1.3. Each Pace employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

1.5.1.4. Each Pace employee must recognize and understand that our daily activities in environmental laboratories affect public health as well as the environment and that environmental laboratory analysts are a critical part of the system society depends upon to improve and guard our natural resources:

1.5.2. **Standards of Conduct:**

1.5.2.1. **Data Integrity**

1.5.2.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at Pace are the cornerstones of the company. Employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

1.5.2.1.2. Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work including commercial, financial, over-scheduling, and working condition pressures.

1.5.2.1.3. The data integrity system includes in-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.5.2.1.4. Any documentation related to data integrity issues, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.5.2.2. **Confidentiality**

1.5.2.2.1. Pace employees must not use or disclose confidential or proprietary information except when in connection with their duties at Pace. This is effective over the course of employment and for an additional period of two years thereafter.

1.5.2.2.2. Confidential or proprietary information, belonging to either Pace and/or its customers, includes but is not limited to test results, trade secrets, research and development matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and customer information, inventions, materials composition, etc.

1.5.2.3. **Conflict of Interest**

1.5.2.3.1. Pace employees must avoid situations that might involve a conflict of interest or could appear questionable to others. This includes participation in activities that conflict or appear to conflict with the employees' Pace responsibilities. This would also include offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally (such as bribes, gifts, kickbacks, or illegal payments).

1.5.2.3.2. Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other problematic activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company, or participation in any outside business during the employee's work hours.

1.5.3. Strict adherence by each Pace employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of Pace and to continue the pursuit of our common mission to protect our environment and improve our health.

1.5.4. Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

1.5.5. Compliance: all employees undergo annual Data Integrity/Ethics training which includes the concepts listed above. All employees also sign an annual Ethic Policy statement.

1.6. Anonymous Compliance Alertline

1.6.1. An ethical and safe workplace is important to the long-term success of Pace and the well-being of its employees. Pace has a responsibility to provide a work environmental where employees feel safe and can report unethical or improper behavior in complete confidence. With this in mind, Pace has engaged Lighthouse Services, Inc. to provide all employees with access to an anonymous ethics and compliance alertline for reporting possible ethics and compliance violations. The purpose of this service is to ensure that any employee can report anonymously and without fear of retaliation.

1.6.2. Lighthouse Services provides a toll-free number along with several other reporting methods, all of which are available 24 hours a day, seven days a week for use by employees and staff.

1.6.3. Telephone: English speaking USA and Canada: (844)-970-0003.

1.6.4. Telephone: Spanish speaking North America: (800)-216-1288.

1.6.5. Website: www.lighthouse-services.com/pacelabs.

1.6.6. Email: reports@lighthouse-services.com (must include company name with report).

1.7. Laboratory Organization

1.7.1. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office. See Attachment III for the Corporate Organizational structure.

1.7.2. A Senior General Manager (SGM) oversees all laboratories and service centers in their assigned region. Each laboratory or facility in the company is then directly managed by an SGM, a General Manager (GM), an Assistant General Manager (AGM), or an Operations Manager (OM). Quality Managers (QM) or Senior Quality Managers (SQM) at each laboratory report directly to the highest level of local laboratory management, however named, that routinely makes day-to-day decisions regarding that facility's operations. The QMs and SQMs will also receive guidance and direction from the corporate Director of Environmental Quality.

1.7.3. The SGM, GM, AGM or OM, or equivalent functionality in each facility, bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of these managers, the SQM/QM serves as the next in command, unless the manager in charge has assigned another designee. He or she assumes the responsibilities of the manager, however named, until the manager is available to resume the duties of their position. In the absence of both the manager and the SQM/QM, management responsibility of the laboratory is passed to the Technical Director, provided such a position is identified, and then to the most senior department manager until the return of the lab manager or SQM/QM. The most senior department manager in charge may include the Client Services Manager (CSM) or the Administrative Business Manager (ABM) at the discretion of the SGM/GM/AGM/OM.

1.7.4. A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The laboratory SGM/GM/AGM/OM or SQM/QM has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 35 consecutive calendar days, the primary accrediting authority shall be notified in writing.

1.7.5. The SQM/QM has the responsibility and authority to ensure the Quality System is implemented and followed at all times. In circumstances where a laboratory is not meeting the established level of quality or following the policies set forth in this Quality Assurance Manual, the SQM/QM has the authority to halt laboratory operations should he or she deem such an action necessary. The SQM/QM will immediately communicate the halting of operations to the SGM/GM/AGM/OM and keep them posted on the progress of corrective actions. In the event the SGM/GM/AGM/OM and the SQM/QM are not in agreement as to the need for the suspension, the Chief Operating Officer (COO) and Director of Environmental Quality will be called in to mediate the situation.

1.7.6. The lab is required to appoint deputies for key managerial personnel. These deputies must be documented for auditing purposes. The deputies, by position, are the following:

1.7.6.1. Deputy for General Manager is Customer Service Manager.

1.7.6.2. Deputy for Organics Technical Director is GC and GCMS Supervisor

1.7.6.3. Deputy for Inorganics Technical Director is senior chemist in the department.

1.7.6.4. Deputy for senior Quality Manager is the Quality Assurance Analyst.

1.7.6.5. Deputy for Client Services Manager is a senior Project Manager.

1.7.6.6. Deputy for Administrative Business Manager is designated department personnel.

1.7.6.7. Deputies for Project Managers are designated Project Managers.

1.7.7. The technical staff of the laboratory is generally organized into the following functional groups:

- Organic Sample Preparation
- Wet Chemistry Analysis
- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis
- Radiochemical Analysis
- Microbiological Analysis
- Bioassay Analysis

1.7.8. The organizational structure for Pace – Pittsburgh is listed in Attachment II. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its accrediting authorities per their individual required timeframes, not to exceed 30 days. The QAM will remain in effect until the next scheduled revision.

1.8. Laboratory Job Descriptions

1.8.1. Senior General Manager

- Oversees all functions of all the operations within their designated region;
- Oversees the development of local GMs/AGMs/OMs within their designated region;
- Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Oversees the preparation of budgets and staffing plans for all operations within their designated region;
- Ensures compliance with all applicable state, federal and industry standards;
- Works closely with Regional Sales Management.

1.8.2. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Presents the Ethics/Data Integrity training annually to all employees in their facilities as an instructor-led training.
- Ensures compliance with all applicable state, federal and industry standards.

1.8.4. Senior Quality Manager

- Provides quality oversight for multiple laboratories where there is not a local quality manager or for labs where there are multiple and separately distinct quality systems in the same facility;

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Environmental Quality;
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors QA/QC activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Environmental Quality office). The SQM is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Environmental Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews and maintains records of proficiency testing results;
- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors corrective and preventive actions;
- Maintains the currency of the Quality Manual.

1.8.5. Technical Director

- Monitors the standards of performance in quality assurance and quality control data;
- Monitors the validity of analyses performed and data generated;
- Reviews tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Serves as the manager of the laboratory in the absence of the SGM/GM/AGM/OM and SQM/QM;
- Provides technical guidance in the review, development, and validation of new methodologies.

1.8.6. Administrative Business Manager

- Responsible for financial and administrative management for the entire facility;
- Provides input relative to tactical and strategic planning activities;
- Organizes financial information so that the facility is run as a fiscally responsible business;
- Works with staff to confirm that appropriate processes are put in place to track revenues and expenses;
- Provide ongoing financial information to the SGM/GM/AGM/OM and the management team so they can better manage their business;
- Utilizes historical information and trends to accurately forecast future financial positions;

- Works with management to ensure that key measurements are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios;
- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.7. Client Services Manager

- Oversees all the day to day activities of the Client Services Department which includes Project Management and, possibly, Sample Control;
- Responsible for staffing and all personnel management related issues for Client Services;
- Serves as the primary senior consultant to customers on all project related issues such as set up, initiation, execution and closure;
- Performs or is capable of performing all duties listed for that of Project Manager.

1.8.8. Project Manager

- Coordinates daily activities including taking orders, reporting data and analytical results;
- Serves as the primary technical and administrative liaison between customers and Pace;
- Communicates with operations staff to update and set project priorities;
- Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.);
- Works with customers, laboratory staff, and other appropriate Pace staff to develop project statements of work or resolve problems of data quality;
- Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers and maintain customer records;
- Mediation of project schedules and scope of work through communication with internal resources and management;
- Responsible for preparing routine and non-routine quotations, reports and technical papers;
- Interfaces between customers and management personnel to achieve customer satisfaction;
- Manages large-scale complex projects;
- Supervises less experienced project managers and provide guidance on management of complex projects;
- Arranges bottle orders and shipment of sample kits to customers;
- Verifies login information relative to project requirements and field sample Chains-of-Custody.

1.8.9. Department Manager/Supervisor

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.10. Laboratory Analyst

- Performs detailed preparation and analysis of samples according to published methods and laboratory procedures;
- Processes and evaluates raw data obtained from preparation and analysis steps;
- Generates final results from raw data, performing primary review against method criteria;
- Monitors quality control data associated with analysis and preparation. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks;
- Reports data in LIMS, authorizing for release pending secondary approval;
- Conducts routine and non-routine maintenance of equipment as required;
- Performs or is capable of performing all duties associated with that of Laboratory Technician.

1.8.11. Laboratory Technician

- Prepares standards and reagents according to published methods or in house procedures;
- Performs preparation and analytical steps for basic laboratory methods;
- Works under the direction of a Laboratory Analyst on complex methodologies;
- Assists Laboratory Analysts on preparation, analytical or data reduction steps for complex methodologies;
- Monitors quality control data as required or directed. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

1.8.12. Field Technician

- Prepares and samples according to published methods, PASI Quality Assurance Manual and/or customer directed sampling objectives;
- Capable of the collection of representative environmental or process related air samples;
- Use computer software to compile, organize, create tables, create graphics and write test reports;
- Reviews project documentation for completeness, method compliance and contract fulfillment;
- Train less experienced environmental technicians and provide guidance on sampling and analysis;
- Responsible for project initiation and contact follow-up;
- Develop sampling plans and prepare test plan documents.

1.8.13. Field Analyst

- Analyzes field samples according to published methods, PASI Quality Assurance Manual and/or customer directed sampling objectives,
- Capable of the collection and analysis of representative environmental or process related air samples,
- Proficient in a variety of analytical tests; specifically on-site gas-phase organic and inorganic compounds by extractive fourier transform infrared spectroscopy (FTIR),
- Train less experienced staff and provide guidance on FTIR sampling and analysis,
- Assist in reporting tasks and project management responsibilities, and
- Perform back-up support for manager tasks such as reporting needs and customer concerns.

1.8.14. Sample Management Personnel

- Signs for incoming samples and verifies the data entered on the Chain of custody forms;
- Enters the sample information into the Laboratory Information Management System (LIMS) for tracking and reporting;
- Stages samples according to EPA requirements;
- Assists Project Managers and Coordinators in filling bottle orders and sample shipments.

1.8.15. Systems Administrator or Systems Manager

- Assists with the creation and maintenance of electronic data deliverables (EDDs);
- Coordinates the installation and use of all hardware, software and operating systems;
- Performs troubleshooting on all aforementioned systems;
- Trains new and existing users on systems and system upgrades;
- Maintains all system security passwords;
- Maintains the electronic backups of all computer systems.

1.8.16. Radiation Safety/Chemical Hygiene Officer

- Maintains the laboratory Radiation Safety Manual;
- Maintains the laboratory Chemical Hygiene Plan;
- Plans and implements safety policies and procedures;
- Maintains safety records;
- Organizes and/or performs safety training;
- Performs safety inspections and provides corrective/preventative actions;
- Assists personnel with safety issues.

1.8.17. Program Director/Hazardous Waste Coordinator (or otherwise named)

- Evaluates waste streams and helps to select appropriate waste transportation and disposal companies;
- Maintains complete records of waste disposal including waste manifests and state reports;
- Assists in training personnel on waste-related issues such as waste handling and storage, waste container labeling, proper satellite accumulation, secondary containment, etc.; Conducts a weekly inspection of the waste storage areas of the laboratory.

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through a web-based training system. Employees are provided with several training activities for their particular job description and scope of duties. These training activities may include:

- Hands-on training led by supervisors;
- Job-specific training checklists and worksheets;
- Lectures and instructor-led training sessions;
- Method-specific training;
- External conferences and seminars;
- Reading Standard Operating Procedures (SOPs);
- Reading the Quality Assurance Manual and Safety Manual/Chemical Hygiene Plan;
- Core training modules (basic lab skills, etc.);
- Quality system training modules (support equipment use, corrective actions/root causes, etc.);
- Data Integrity/Ethics training;

- Specialized training by instrument manufacturers;
- On-line courses.

1.9.2. All procedures and training records are maintained and available for review during laboratory audits. Additional information can be found in SOP S-ALL-Q-020 **Training and Employee Orientation** or its equivalent revision or replacement.

1.10. Laboratory Safety and Waste

1.10.1. It is the policy of Pace to make safety and waste compliance an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety as well as those working in the immediate area by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the local Safety Manual/Chemical Hygiene Plan.

1.11. Security and Confidentiality

1.11.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by Pace staff.

1.11.2. Additional security is provided where necessary, (e.g., specific secure areas for sample, data, and customer report storage), as requested by customers, or cases where national security is of concern. These areas are lockable within the facilities, or are securely offsite. Access is limited to specific individuals or their designees.

1.11.3. All information pertaining to a particular customer, including national security concerns will remain confidential. Data will be released to outside agencies only with written authorization from the customer or where federal or state law requires the company to do so.

1.12. Communications

1.12.1. Management within each lab bears the responsibility of ensuring that appropriate communication processes are established and that communication takes place regarding the effectiveness of the management/quality system. These communication processes may include email, regular staff meetings, senior management meetings, etc.

1.12.2. Corporate management bears the responsibility of ensuring that appropriate communication processes are established within the network of facilities and that communication takes place at a company-wide level regarding the effectiveness of the management/quality systems of all Pace facilities. These communication processes may include email, quarterly continuous improvement conference calls for all lab departments, and annual continuous improvement meetings for all department supervisors, quality managers, client services managers, and other support positions.

2.0. SAMPLE CUSTODY

2.1. Project Initiation

2.1.1. Prior to accepting new work, the laboratory reviews its performance capability. The laboratory confirms that sufficient personnel, equipment capacity, analytical method capability, etc., are available to complete the required work. Customer needs, certification requirements, and data quality objectives are defined and the appropriate sampling and analysis plan is developed to meet the project requirements by project managers or sales representatives. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability, and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or daily operations meetings.

2.1.2. Additional information regarding specific procedures for reviewing new work requests can be found in SOP PGH-C-033 **Review of Analytical Requests** or its equivalent revision or replacement.

2.2. Sampling Materials and Support

Each individual Pace laboratory provides shipping containers, properly preserved sample containers, custody documents, and field quality control samples to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VII. Note that all analyses listed are not necessarily performed at all Pace laboratories and there may be additional laboratory analyses performed that are not included in these tables. Customers are encouraged to contact their local Pace Project Manager for questions or clarifications regarding sample handling. Pace may provide pick-up and delivery services to their customers when needed Some Pace facilities provide sampling support through a Field Services department. Field Services operates under the Pace Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in SOPs and Procedure Manuals.

2.3. Chain of Custody

2.3.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis.

2.3.2. Field personnel or client representatives must complete a COC for all samples that are received by the laboratory. Samplers are required to properly complete a COC. This is critical to efficient sample receipt and to ensure the requested methods are used to analyze the correct samples. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.3.3. The COC is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the initial entry and initialing and dating the change. All transfers of samples are recorded on the chain of custody in the “relinquished” and “received by” sections. All information except signatures is printed.

2.3.4. Additional information can be found in SOP S-PGH-C-001 **Sample Management** or its equivalent revision or replacement.

2.4. Sample Acceptance Policy

2.4.1. In accordance with regulatory guidelines, Pace complies with the following sample acceptance policy for all samples received.

2.4.2. If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory is required to document all non-compliances, contact the customer, and either reject the samples or fully document any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately communicated to the client.

2.4.3. Sample Acceptance Policy requirements:

- Sample containers must have unique client identification designations that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC).
- There must be clear documentation on the COC, or related documents, that lists the unique sample identification, sampling site location, date and time of sample collection, and name of the sample collector.
- There must be clear documentation on the COC, or related documents, that lists the requested analyses, the preservatives used, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.).
- Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval.
- Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval.
- Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval.
- Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval.
- All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2°C of the required temperature, or within the method-specified range. For samples with a required temperature of 4°C, samples with a temperature ranging from just above freezing to 6°C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
- Samples for **drinking water** analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are improperly preserved (with the exception of VOA samples- tested for pH at time of analysis and TOC- tested for pH in the field).

- Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.

Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be read and recorded to ± 0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ± 0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤ 6°C limit. Please reference the Support Equipment SOP for more information.

Note 2: Some microbiology methods allow sample receipt temperatures of up to 10°C. Consult the specific method for microbiology samples received above 6°C prior to initiating corrective action for out of temperature preservation conditions.

Note 3: Biological Tissue Samples must be received at the following temperature based on program and contract: cooled to ≤ 6°C during the first 24 hours after collection; then samples must be kept frozen at ≤ - 10°C. TNI rules also apply if the samples are brought straight from the field; they are acceptable if evidence of cooling is present (i.e., received on ice).

2.4.4. Upon sample receipt, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers;
- Sample condition: Intact, broken/leaking, bubbles in VOA samples;
- Sample holding time;
- Sample pH and residual chlorine when required;
- Appropriate containers.

2.4.5. Additional information can be found in SOP S-PGH-C-001 **Sample Management** or its equivalent revision or replacement.

2.5. Sample Log-in

2.5.1. After sample inspection, all sample information on the COC is entered into the Laboratory Information Management System (LIMS). The lab's permanent records for samples received include the following information:

- Customer name and contact
- Customer number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested
- Date and time of laboratory receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

2.5.2. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 12:01am as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

2.5.3. For DoD work, if the time of the sample collection is not provided, the laboratory must assume the most conservative time of day. This is defined as 12:01am.

2.5.4. The Laboratory Information Management System automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of 30XXXXX-YYY. The first two numbers (30) designates the project as a PASI-Pittsburgh project, the last three digits (YYY) are used to designate the individual sample numbers, and the digits XXXXX (Where the "X's" are sequential numbers generated by the LIMS) identify the project number. This unique identification number is placed on each sample container as a durable label and becomes the link between the laboratory's sample management system and the customer's field identification; and will be a permanent reference number for all future interactions.

2.5.5. Sample labels are printed from the LIMS and affixed to each sample container.

2.5.6. Additional information can be found in SOP S-PGH-C-001 **Sample Management** or its equivalent revision or replacement.

2.6. Sample Storage

2.6.1. Additional information on sample storage can be found in SOP S-PGH-C-001 **Sample Management** or its equivalent revision or replacement and in SOP PGH-C-017 **Waste Handling and Management** or its equivalent revision or replacement.

2.6.2. **Storage Conditions**

2.6.2.1. Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

2.6.2.2. Storage blanks are stored with volatile samples and are used to measure cross-contamination acquired during storage. Laboratories must have documented procedures and criteria for evaluating storage blanks, appropriate to the types of samples being stored.

2.6.2.3. Additional information can be found in SOP PGH-Q-044 **Monitoring Temperature Controlled Units.**

2.6.3. **Temperature Monitoring**

2.6.3.1. Samples are taken to the appropriate storage location immediately after sample receipt and check-in procedures are completed.

2.6.3.2. The temperature of each refrigerated storage area is maintained at $\leq 6^{\circ}C$ (but above freezing) unless state, method or program requirements differ. The temperature of each freezer storage area is maintained at $\leq -10^{o}C$ unless state, method or program requirements differ. The temperature of each storage area is checked and documented each day of use (each calendar day). Additional information, including corrective actions for temperatures outside of acceptance limits, can be found in SOP PGH-Q-044, **Monitoring Temperature Controlled Units**.

2.6.4. **Hazardous Materials**

2.6.4.1. Samples designated by clients upon receipt as pure product or potentially heavily contaminated samples, or samples found to be designated as such following analysis, must be tagged as "hazardous" or "lab pack" and stored separately from other samples.

2.6.4.2. Clients must properly label all samples that contain radioactivity. These samples are screened by the Radiation Safety Officer and if noted to be of concern this information is communicated to the necessary laboratory personnel. Any samples with levels of radiation that are noted to be of concern will be placed into a separate storage area of the laboratory to prevent cross-contamination.

2.6.5. **Foreign/Quarantined Soils**

2.6.5.1. Foreign soils and soils from USDA regulated areas must be adequately segregated to enable proper sample disposal. The USDA requires these samples to be treated by an approved procedure. Additional information regarding USDA regulations and sample handling can be found in the laboratory's SOP for Waste Handling PGH-C-017, or its equivalent revision or replacement.

2.7. Subcontracting Analytical Services

2.7.1. Every effort is made to perform all analyses for Pace customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory, whether inside or outside the Pace network, becomes necessary, a preliminary verbal communication with that laboratory is undertaken. Customers are notified in writing of the laboratory's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations. When possible, subcontracting will be to a TNI-accredited laboratory.

2.7.2. Potential subcontract laboratories must be approved by Pace based on the criteria listed in SOP S-PGH-C-008 **Subcontracting Samples** or its equivalent revision or replacement. All sample reports from the subcontracted labs are appended to the applicable Pace final reports.

2.7.3. Any Pace Analytical work sent to other labs within the Pace network is handled as inter-regional work and all final reports are labeled clearly with the name of the laboratory performing the work. Any non-TNI work is clearly identified. Pace will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

2.7.4. Additional information can be found in SOP S-PGH-C-008 **Subcontracting Samples** or its equivalent revision or replacement.

2.7.5. Subcontracted labs used for DoD work must be accredited by DoD or its designated representatives. Subcontracted labs must receive project specific approval from the DoD client before any samples are analyzed. These requirements also apply to the use of any laboratory under the same corporate umbrella, but at a different facility or location.

2.8. Sample Retention and Disposal

2.8.1. Samples, extracts, digestates, and leachates must be retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

2.8.2. The minimum sample retention time is 45 days from receipt of the samples. Samples requiring thermal preservation may be stored at ambient temperature when the hold time is expired, the report has been delivered, and/or allowed by the customer, program, or contract. Samples

requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

2.8.3. After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of **hazardous** samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires Pace to dispose of excess samples, proper arrangements will be made for disposal by an approved contractor.

2.8.4. Additional information can be found in SOP PGH-C-017 **Waste Handling and Management** and SOP S-PGH-C-001 **Sample Management** or their equivalent revisions or replacements.

3.0. QUALITY CONTROL PROCEDURES

3.1. Quality Control Samples

3.1.1. The quality control samples described in this section are analyzed per batch as applicable to the method used. Acceptance criteria must be established for all quality control samples and if the acceptance criteria are not met, corrective actions must be performed and samples reanalyzed, or final reports must be appropriately qualified.

3.1.2. Quality control samples must be processed in the same manner as associated client samples.

3.1.3. Please reference the glossary of this Quality Manual for definitions of all quality control samples mentioned in this section.

3.1.4. Any deviations to the policies and procedures governing quality control samples must be approved by the QM/SQM.

3.2. Method Blank

3.2.1. A method blank is a negative control used to assess the preparation/analysis system for possible contamination and is processed through all preparation and analytical steps with its associated client samples. The method blank is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples. Method blanks are not applicable for certain analyses (i.e., pH, flash point, temperature, etc.).

3.2.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for method blanks.

3.2.3. For DoD samples, the method blank will be considered to be contaminated if: 1) The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit whichever is greater; 2) The concentration of any common laboratory contaminant in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit whichever is greater or 3) The blank result otherwise affects the sample results as per the test method requirements or the project-specific objectives. If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

3.3. Laboratory Control Sample

3.3.1. The Laboratory Control Sample (LCS) is a positive control used to assess the performance of the entire analytical system including preparation and analysis. The LCS is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples.

3.3.2. The LCS contains **all** analytes required by a specific method or by the customer or regulatory agency, which may include full list of target compounds, with certain exceptions. The lab must ensure that all target components are included in the spike mixture for the LCS over a two (2) year

period. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
  - For methods with 1-10 target compounds, the laboratory will spike with all compounds;
  - For methods with 11-20 target compounds, the laboratory will spike with at least 10 compounds or 80%, whichever is greater;
  - For methods with greater than 20 compounds, the laboratory will spike with at least 16 compounds.

3.3.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for LCSs.

3.3.4. For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary (except for proper documentation). TNI has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but within than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

- >90 analytes in the LCS- 5 analytes
- 71-90 analytes in the LCS- 4 analytes
- 51-70 analytes in the LCS- 3 analytes
- 31-50 analytes in the LCS- 2 analytes
- 11-30 analytes in the LCS- 1 analyte
- <11 analytes in the LCS- no analytes allowed out)

Note: the use of marginal exceedances is not approved for work from the state of South Carolina.

3.3.5. A matrix spike (MS) can be used in place of a non-compliant LCS in a batch as long as the MS passes the LCS acceptance criteria (this is a TNI allowance). Note: the use of the MS to replace a non-compliant LCS is not approved for work from the state of South Carolina. When this happens, full documentation must be made available to the data user. If this is not allowed by a customer or regulatory body, the associated samples must be rerun with a compliant LCS (if possible) or reported with appropriate data qualifiers.

3.3.6. For DoD projects, the laboratory is not allowed to have any target analytes that exceed DoD LCS control limits. In the case of LCS failures, the laboratory is required to reanalyze the associated samples with an acceptable LCS for all target compounds if there is sufficient sample remaining. The laboratory must make every effort to take the appropriate corrective actions and resolve any anomalies regarding LCSs for DoD projects. All LCS failures must be accounted for in project case narratives. See applicable method SOPs for further corrective action.

3.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.4.1. A matrix spike (MS) is a positive control used to determine the effect of the sample matrix on compound recovery for a particular method. A matrix spike/matrix spike duplicate (MS/MSD) set or matrix spike/sample duplicate set is processed at a frequency specified in a particular method or as determined by a specific customer request. The MS and MSD consist of the sample matrix that is spiked with known concentrations of target analytes.

3.4.2. The MS and MSD contain all analytes required by a specific method or by the customer or regulatory agency. In the absence of specified components, the laboratory will spike the MS/MSD with the same number of compounds as previously discussed in the LCS section. However, the lab must ensure that all targeted components are included in the spike mixture for the MS/MSD over a two (2) year period.

3.4.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for MS/MSDs.

3.4.4. For DoD work, each non-radiochemistry preparation batch of samples must contain an associated MS and MSD (or sample duplicate) using the same matrix collected for the specific DoD project. For radiochemical analyses, tests that do not incorporate the use of a carrier or tracer for yield assessment must contain an associated MS and MSD (or sample duplicate) using the same matrix collected for the specific DOD project. Gamma spectroscopy analyses are excluded from the MS/MSD requirement as the test does not require chemical processing of samples for analysis. If adequate sample material is not available, then the lack of MS/MSDs shall be noted in the case narrative. Additional MS/MSDs may be required on a project-specific basis. The MS/MSD must be spiked with all target analytes. The concentration of the spiked compounds shall be at or below the midpoint of the calibration range or at the appropriate concentration of concern. Multiple spiked samples may need to be prepared to avoid interferences.

3.5. Sample Duplicate

3.5.1. A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

3.5.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for sample duplicates.

3.6. Surrogates

3.6.1. Surrogates are compounds that reflect the chemistry of target analytes and are typically added to samples for organic analyses to measure the extraction or purge efficiency and to monitor the effect of the sample matrix on compound recovery.

3.6.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for surrogates.

3.7. Internal Standards

3.7.1. Internal Standards are method-specific analytes that are added, as applicable, to every standard, QC sample, and client sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes..

3.7.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for internal standards.

3.8. Limit of Detection (LOD)

3.8.1. Pace laboratories use a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. Unless otherwise noted in a published method, the procedure used by Pace laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. All sample processing steps of the preparation and analytical methods are included in the LOD determination including any clean ups.

3.8.2. DoD definition for LOD- The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate is 1%.

3.8.3. Additional information can be found in SOP S-PGH-Q-035 **Determination of LOD and LOQ** or its equivalent revision or replacement.

3.9. Limit of Quantitation (LOQ)

3.9.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For Pace laboratories, this LOQ is referred to as the RL, or Reporting Limit. Results reported below the reporting limit are not allowed to be reported without qualification. For methods with a determined LOD, results can be reported out below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

3.9.2. For DoD approved methods, the LOQ and LOD shall be verified quarterly and valid LOQ must be in place prior to sample analysis.

3.9.3. Additional information can be found in SOP S-PGH-Q-035 **Determination of LOD and LOQ** or its equivalent revision or replacement.

3.10. Estimate of Analytical Uncertainty

3.10.1. Pace laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, Pace laboratories base this estimation on the recovery data obtained from the Laboratory Control Samples. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP PGH-Q-046 **Estimation of Measurement Uncertainty** or its equivalent revision or replacement.

3.10.2. The measurement of uncertainty is provided only on request by the customer, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

3.10.3. Radiological tests often report uncertainty and the manner in which it is derived are in accordance with Multi-Agency Radiological Laboratories Analytical Protocols Manual (MARLAP)

and Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement (GUM). The means by which these criteria are applied can be found in the method SOPs.

3.11. Proficiency Testing (PT) Studies

3.11.1. Pace laboratories participate in a defined proficiency testing (PT) program. PT samples are obtained from NIST approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix.

3.11.2. Additional information can be found in SOP PGH-C-031 **Proficiency Testing Program** or its equivalent revision or replacement.

3.12. Rounding and Significant Figures

3.12.1. In general, the Pace laboratories report data to no more than three significant figures. Therefore, all measurements made in the analytical process must reflect this level of precision. In the event that a parameter that contributes to the final result has less than three significant figures of precision, the final result must be reported with no more significant figures than that of the parameter in question. The rounding rules listed below are descriptive of the LIMS and not necessarily of any supporting program such as Excel.

3.12.2. **Rounding:** Pace-Pittsburgh follows the odd / even guidelines for rounding numbers:

- If the figure following the one to be retained is less than five, that figure is dropped and the retained ones are not changed (with three significant figures, 2.544 is rounded to 2.54).
- If the figure following the ones to be retained is greater than five, that figure is dropped and the last retained one is rounded up (with three significant figures, 2.546 is rounded to 2.55).
- If the figure following the ones to be retained is five and if there are no figures other than zeros beyond that five, then the five is dropped and the last figure retained is unchanged if it is even and rounded up if it is odd (with three significant figures, 2.525 is rounded to 2.52 and 2.535 is rounded to 2.54).

3.12.3. **Significant Figures**

3.12.3.1. Pace-Pittsburgh follows the following convention for reporting to a specified number of significant figures. Unless specified by federal, state, or local requirements or on specific request by a customer, the laboratory reports:

Values > 10 – Reported to 3 significant figures

Values ≤ 10 – Reported to 2 significant figures

3.13. Retention Time Windows

3.13.1. When chromatographic conditions are changed, retention times and analytical separations are often affected. As a result, two critical aspects of any chromatographic method are the determination and verification of retention times and analyte separation. Retention time windows must be established for the identification of target analytes. The retention times of all target analytes in all calibration verification standards must fall within the retention time windows. If an analyte falls outside the retention time window in an ICV or CCV, new absolute retention time windows

must be calculated, unless instrument maintenance fixes the problem. When a new column is installed, a new retention time window study must be performed.

3.13.2. Please reference method-specific SOPs for the proper procedure for establishing retention time windows.

3.14. Analytical Method Validation and Instrument Validation

3.14.1. In some situations, Pace develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods are required for specific projects or analytes of interest, or when the laboratory develops or modifies a method, the laboratory validates the method prior to applying it to customer samples. Method validity is established by meeting criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. The laboratory records the validation procedure, the results obtained and a statement as to the usability of the method. The minimum requirements for method validation include evaluation of sensitivity, quantitation, precision, bias, and selectivity of each analyte of interest.

3.15. Regulatory and Method Compliance

3.15.1. It is Pace policy to disclose in a forthright manner any detected noncompliance affecting the usability of data produced by our laboratories. The laboratory will notify customers within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

3.15.2. For DoD QSM the laboratory shall upon discovery, notify all affected customers of potential data quality issues resulting from nonconforming work within 15 business days. Notification shall be performed according to a written procedure. Records of corrections taken or proposed corrective actions to resolve the nonconformance shall be submitted to the customer(s) within 30 business days of discovery.

4.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

4.1. Document Management

4.1.1. Additional information can be found in SOP S-PGH-Q-043 **Document Control and Management** or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

4.1.2. Pace has an established procedure for managing documents that are part of the quality system.

4.1.3. A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. Copies of all quality systems documentation provided to DoD for review must be in English.

4.1.4. Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to SOP S-ALL-Q-003 **Document Numbering**.

4.1.5. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for Pace. The base QAM template is distributed by the Corporate Environmental Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template and submit those modifications to the Corporate Director of Environmental Quality for review. Once approved, all applicable lab staff sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis and revised accordingly by the Corporate Quality office.

4.1.6. **Standard Operating Procedures (SOPs)**

4.1.6.1. SOPs are reviewed every two years at a minimum although a more frequent review may be required by some state or federal agencies or customers. If no revisions are made based on this review, documentation of the review itself is made by the addition of new signatures on the cover page. If revisions are made, documentation of the revisions is made in the revisions section of each SOP and a new revision number is applied to the SOP. This provides a historical record of all revisions.

4.1.6.2. All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all Pace employees use the most current version of each SOP and provides the SQM/QM with a historical record of each SOP.

4.1.6.3. Additional information can be found in SOP S-PGH-Q-001 **Preparation of SOPs** or its equivalent revision or replacement.

4.1.6.4. For DoD approval, all technical SOPs are reviewed for accuracy and adequacy annually and whenever method procedures change and updated as appropriate. All such reviews are documented and made available for assessment. Non-technical SOPs that are not required elements of the quality system are considered administrative SOPs and are not required to be reviewed annually.

4.2. Document Change Control

4.2.1. Additional information can be found in SOP S-PGH-Q-043 **Document Control and Management** or its equivalent revision or replacement.

4.2.2. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After revisions are approved, a revision number is assigned and the previous version of the document is officially retired.

4.2.3. All copies of the previous document are replaced with copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised that there has been a revision and any necessary training is scheduled.

5.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

5.1. Standards and Traceability

5.1.1. Each Pace facility retains pertinent information for standards, reagents, and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation, and use.

5.1.2. Upon receipt, all purchased standard reference materials are recorded into a standard logbook or database and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date, and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

5.1.3. Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, the preparation steps, preparation date, expiration dates, preparer's initials, and a unique Pace identification number. This number is used in any applicable sample preparation or analysis logbook so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

5.1.4. All prepared standard or reagent containers include the Pace identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials, unless the container is too small to hold all of this information. This ensures traceability back to the standard preparation logbook or database.

5.1.5. All initial calibrations must be verified with a standard obtained from a second manufacturer or a separate lot prepared independently by the same manufacturer, unless client-specific QAPP requirements state otherwise.

5.1.6. Additional information concerning the procurement of standards and reagent and their traceability can be found in the SOP PGH-C-037 **Standard and Reagent Management and Traceability** or its equivalent revision or replacement.

5.2. General Analytical Instrument Calibration Procedures

5.2.1. All applicable instrumentation are calibrated or checked before use to ensure proper functioning and verify that laboratory, client and regulatory requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated.

5.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in the narrative.

5.2.3. Radiological calibrations may follow one of several methodologies based on technology of the counting; these can include efficiency curves, energy calibrations and quench curves. The various calibrations should ensure that the range chosen encompasses the activities expected in the client samples.

5.2.4. Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets the laboratory's specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported to prevent breakage and is in accordance with the calibration laboratory's recommendations.

5.2.5. In the event that recalibration of a piece of test equipment indicates the equipment may have been malfunctioning during the course of sample analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the quality manager. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or taken out of service and replaced.

5.2.5.1. For DoD QSM, the laboratory shall upon discovery, notify all affected customers of potential data quality issues resulting from nonconforming work within 15 business days. Notification shall be performed according to a written procedure. Records of corrections taken or proposed corrective actions to resolve the nonconformance shall be submitted to the customer(s) within 30 business days of discovery Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

5.2.6. Radiological Equipment Calibration

5.2.6.1. Radiological Equipment should be calibrated at the appropriate frequency and whenever the equipment undergoes maintenance. In the case of liquid scintillation counters the equipment shall be recalibrated when a significant move has taken place.

5.2.6.2. Calibrations can vary with equipment; in the case of gas flow proportional counters standards that range the expected residue range for gross alpha and beta shall be used, with efficiency curves developed to encompass the range of client sample residues. Any samples outside of this range shall be evaluated and the aliquot changed to accommodate the curve if necessary. Beta emitters, or isotopes that are shown to have less than a 2% efficiency change with residue that are known to not experience self attenuation may be calibrated by using a least 3 standards of known activity and comparing the efficiency results to ensure all agree to a relative standard deviation of less than 5%.

5.2.6.3. Quench factors for liquid scintillation counters shall be prepared by adding varied amounts of quenching agent. Any sample displaying a quench factor outside of the curve shall be evaluated. If the quench factors are shown to not vary in efficiency by greater than 2% then an efficiency calibration can be established using at least 3 standards of known activity and comparing the efficiency results to ensure all agree to a relative standard deviation of less than 5%.

5.2.6.4. Cross talk factors must also be evaluated when samples are known to contain more than one beta or an alpha and beta emitter.

5.2.6.5. All detectors must pass various daily tests depending upon the technology. The criteria of these various tests should be known to the analyst. Any detector that does not pass the daily check must be re-checked. If the daily test fails a second time the detector must be taken out of service for that day. Any detector that fails two daily checks must be evaluated and serviced if required. In most instances two passing daily checks are required to put a detector back into service.

5.3. Support Equipment Calibration and Verification Procedures

5.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use, as applicable. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until brought back into control. Additional information regarding calibration and maintenance of support equipment can be found in SOP PGH-C-032 **Support Equipment** or its equivalent revision or replacement.

5.3.2. On each day the support equipment is used, it is verified, as applicable, in the expected range of use with NIST traceable references in order to ensure the equipment meets laboratory specifications. These checks are documented appropriately. This applies mainly to thermometers within temperature-controlled units and balances.

5.3.3. **Analytical Balances**

5.3.3.1. Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Calibration weights are ASTM Class 1 or other class weights that have been calibrated against a NIST standard weight and are re-certified every 5 years at a minimum against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the local Quality department.

5.3.4. Thermometers

5.3.4.1. Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are re-certified, at a minimum, every 5 years with equipment directly traceable to NIST.

5.3.4.2. Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures (working digital thermometers are calibrated quarterly). Each thermometer is individually numbered and assigned a correction factor based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

5.3.4.3. Laboratory thermometer inventory and calibration data are maintained in the local Quality department.

5.3.5. pH/Electrometers

5.3.5.1. The meter is calibrated before use each day, using fresh buffer solutions. The range of pH that is used for calibration should bracket the pH measurements of the samples analyzed.

5.3.6. Spectrophotometers

5.3.6.1. During use, spectrophotometer performance is checked at established frequencies in analysis sequences against initial calibration verification (ICV) and continuing calibration verification (CCV) standards.

5.3.7. Mechanical Volumetric Dispensing Devices

5.3.7.1. Mechanical volumetric dispensing devices including bottle top dispensers (those that are critical in measuring a required amount of reagent), pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis.

5.3.7.2. Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP PGH-C-032 **Support Equipment** or its equivalent revision or replacement.

5.4. Instrument/Equipment Maintenance

5.4.1. The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs. Further details can be found in SOP S-PGH-Q-038 **Laboratory Equipment** or its equivalent revision or replacement.

5.4.2. The Operations Manager and/or department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

5.4.3. To minimize downtime and interruption of analytical work, preventative maintenance may routinely performed on each analytical instrument. Up-to-date instructions on the use and maintenance of equipment are available to staff in the department where the equipment is used.

5.4.4. Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

5.4.5. All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation is, unless otherwise stated, identified as a system and not as individual pieces. The laboratory maintains equipment records that include the following:

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service
- Current location in the laboratory
- Condition when received (new, used, etc.)
- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

5.4.6. All instrument maintenance is documented in maintenance logbooks that are assigned to each particular instrument or system.

5.4.7. The maintenance log entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

5.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily. In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab if possible.

6.0. CONTROL OF DATA

Analytical results processing, verification, and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user.

All analytical data undergo a documented multi-tier review process prior to being reported to the customer. This section describes procedures used for translating raw analytical data into accurate final sample reports as well as Pace data storage policies.

When analytical, field, or product testing data is generated, it is documented appropriately. These logbooks and other laboratory records are kept in accordance with each facility's SOP for documentation storage and archival In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues.

6.1. Primary Data Review

6.1.1. The primary analyst is responsible for initial data reduction and data review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. Data review checklists, either hardcopy or electronic, are used to document the primary data review process. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets, LIMS fields, and data review checklists.

6.1.2. The primary analyst compiles the initial data for secondary data review. This compilation must include sufficient documentation for secondary data review.

6.1.3. Additional information regarding data review procedures can be found in SOP PGH-Q-037 **Data Review** or its equivalent revision or replacement, as well as in SOP S-ALL-Q-016 **Manual Integration** or its equivalent revision or replacement.

6.2. Secondary Data Review

6.2.1. Secondary data review is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated, and that any non-conformances are properly documented.

6.2.2. The completed data from the primary analyst is sent to a designated qualified secondary data reviewer (this cannot be the primary analyst). The secondary data reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer validates the data entered into the LIMS and documents approval of manual integrations. Data review checklists, either hardcopy or electronic, are used to document the secondary data review process.

6.2.3. Additional information regarding data review procedures can be found in SOP PGH-Q-037 **Data Review** or its equivalent revision or replacement, as well as in SOP S-All-Q-016 **Manual Integration** or its equivalent revision or replacement.

6.2.4. Some reports and/or data packages may be reviewed by the QM or SQM or designee based on program requirements (e.g., DoD) or client requirements. In this case a thorough review for completeness and accuracy may include a compilation of raw data and QC summaries in addition to the final report to produce a full deliverable package. In the case of DoD, 100% of all packages must have a final administrative review (to confirm that primary and secondary reviews were completed and documented and that data packages are complete) and 10% of all data packages must be reviewed by the Quality Manager for technical completeness/accuracy. This 10% review can be done after the data packages have been submitted to the clients. See SOP PGH-Q-040 **Internal and External Audits**, for full Quality department final report and raw data review requirements.

6.3. Data Reporting

6.3.1. Data for each analytical fraction pertaining to a particular Pace project number are delivered to the Project Manager for assembly into the final report. All points mentioned during technical and QC reviews are included in data qualifiers on the final report or in a separate case narrative if there is potential for data to be impacted.

6.3.2. Final reports are prepared according to the level of reporting required by the customer and can be transmitted to the customer via hardcopy or electronic deliverable.

6.3.3. For DoD labs, both date and time of preparation and analysis are considered essential information, regardless of the length of the holding time, and shall be included as part of the laboratory report.

6.3.4. Any changes made to a final report shall be designated as “Revised” or equivalent wording. The laboratory must keep sufficient archived records of all laboratory reports and revisions. For higher levels of data deliverables, a copy of all supporting raw data is sent to the customer along with a final report of results. Pace will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

6.3.5. Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

6.3.6. The following positions are the only approved signatories for Pace final reports:

- Senior General Manager
- General Manager
- Assistant General Manager
- Senior Quality Manager
- Quality Manager
- Client Services Manager
- Project Manager
- Project Coordinator

6.4. Data Security

6.4.1. All data including electronic files, logbooks, extraction/digestion/distillation worksheets, calculations, project files and reports, and any other information used to produce the technical report are maintained secured and retrievable by the Pace facility.

6.5. Data Archiving

6.5.1. All records compiled by Pace are archived in a suitable, limited-access environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. TNI-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the SQM/QM or a designated Data Archivist.

6.5.2. Records that are computer-generated have either a hard copy or electronic backup copy. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

6.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained per the purchase agreement. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

6.6. Data Disposal

6.6.1. Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. Data disposal includes any preliminary or final reports that are disposed.

7.0. QUALITY SYSTEM AUDITS AND REVIEWS

7.1. Internal Audits

7.1.1. Responsibilities

7.1.1.1. The SQM/QM is responsible for managing and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The SQM/QM evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the effectiveness of the Quality System as outlined in this manual but may also include other quality programs applicable to an individual laboratory.

7.1.1.2. Additional information can be found in SOP PGH-Q-040 **Internal and External Audits** or its equivalent revision or replacement.

7.1.2. Scope and Frequency of Internal Audits

7.1.2.1. The complete internal audit process consists of the following four sections: 1) Raw Data Reviews, 2) traditional Quality Systems internal audits (including SOP and method compliance), 3) Final Report Reviews, and 4) Corrective Action Effectiveness Follow-up.

7.1.2.2. Internal systems audits are conducted yearly at a minimum. The scope of these audits includes evaluation of specific analytical departments or a specific quality related system as applied throughout the laboratory.

7.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible.

7.1.2.4. Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

7.1.2.5. The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the laboratory, and which final reports had to be re-issued. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed.

7.1.2.5.1. For DoD QSM the laboratory shall upon discovery, notify all affected customers of potential data quality issues resulting from nonconforming work within 15 business days. Notification shall be performed according to a written procedure. Records of corrections taken or proposed corrective actions to resolve the nonconformance shall be submitted to the customer(s) within 30 business days of discovery.

7.1.3. Internal Audit Reports and Corrective Action Plans

7.1.3.1. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the SQM/QM writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

7.1.3.2. When audit findings cast doubt on the effectiveness of the operations or on the correctness of validity of the laboratory's environmental test results, the laboratory will take timely corrective action and notify the customer in writing within three business days, if investigations show that the laboratory results may have been affected.

7.1.3.3. Additional information can be found in SOP PGH-Q-040 **Internal and External Audits** or its equivalent revision or replacement.

7.2. External Audits

7.2.1. Pace laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

7.2.2. External audit teams review the laboratory to assess the effectiveness of quality systems. The SQM/QM host the external audit team and assist in facilitation of the audit process. After the audit, the external auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM, who provides a written response to the external audit team. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

7.3. Annual Managerial Review

7.3.1. A managerial review of Management and Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements. Additional information can be found in SOP S-ALL-Q-015 **Review of Laboratory Management System** or its equivalent revision or replacement.

7.3.2. The managerial review must include the following topics of discussion:

- Suitability of quality management policies and procedures
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventive actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources, and staffing.

7.3.3. This managerial review must be documented for future reference by the SQM/QM and copies of the report are distributed to laboratory staff. Results must feed into the laboratory planning system and must include goals, objectives, and action plans for the coming year. The laboratory shall ensure

that any actions identified during the review are carried out within an appropriate and agreed upon timescale.

8.0. CORRECTIVE ACTION

Additional information can be found in SOP PGH-Q-039 **Corrective and Preventive Actions** or its equivalent revision or replacement.

During the process of sample handling, preparation, and analysis, or during review of quality control records, or during reviews of non-technical portions of the lab, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of Pace provides systematic procedures for the documentation, monitoring, completion of corrective actions, and follow-up verification of the effectiveness of these corrective actions. This can be done using Pace's LabTrack system or other system that lists at a minimum, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

8.1. Corrective and Preventive Action Documentation

8.1.1. The following items are examples of sources of laboratory deviations or non-conformances that may warrant some form of documented corrective action:

- Internal Laboratory Non-Conformance Trends
- Proficiency Testing Sample Results
- Internal and External Audits
- Data or Records Review
- Client Complaints
- Client Inquiries
- Holding Time violations

8.1.2. Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g., matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

8.1.3. The person who discovers the deficiency or non-conformance initiates the corrective action documentation within the lab's corrective action system. The documentation must include (as applicable): the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

8.1.4. **Root Cause Analysis**: Laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if

the condition is significant, and investigating the root cause of the nonconformance. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within the lab's corrective action system.

8.1.5. Based on the root cause(s) determined, the lab implements applicable corrective actions and verifies their effectiveness. In the event that analytical testing or results do not conform to documented laboratory policies or procedures Project Management will notify the customer of the situation and will advise of any ramifications to data quality if impacted (with the possibility of work being recalled).

8.2. Corrective Action Completion

8.2.1. Internal Laboratory Non-Conformance Trends

8.2.1.1. There are several types of non-conformance trends that may occur in the laboratory that would require the initiation of a corrective action report. Laboratories may choose to initiate a corrective action for all instances of one or more of these categories if they so choose, however the intent is that each of these would be handled according to its severity; one time instances could be handled with a footnote or qualifier whereas a systemic problem with any of these categories may require an official corrective action process. These categories, as defined in the Corrective Action SOP are as follows:

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting error

8.2.2. PE/PT Sample Results

8.2.2.1. Any PT result assessed as "not acceptable" requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

8.2.2.2. Additional information, such as requirements regarding time frames for reporting failures to states, makeup PTs, and notifications of investigations, can be found in SOP PGH-C-031 **Proficiency Testing Program** or its equivalent revision or replacement.

8.2.3. Internal and External Audits

8.2.3.1. The SQM/QM is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for responding to the auditing body, the root cause of the finding, and the corrective actions needed for resolution. The SQM/QM is also responsible for providing any back-up documentation used to demonstrate that a corrective action has been completed.

8.2.4. **Data Review**

8.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data reviews (e.g., by the SQM/QM), errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

8.2.5. **Client Complaints**

8.2.5.1. Project Managers are responsible for issuing corrective action forms, when warranted, for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor for investigation. After potential corrective actions have been determined, the Project Manager reviews the corrective action form to ensure all customer needs or concerns are being adequately addressed.

8.2.6. **Client Inquiries**

8.2.6.1. When an error on the customer report is discovered, the Project Manager is responsible for initiating a formal corrective action form that describes the failure (e.g., incorrect analysis reported, reporting units are incorrect, or reporting limits do not meet objectives). The Project Manager is also responsible for revising the final report if necessary and submitting it to the customer.

8.2.7. **Holding Time Violations**

8.2.7.1. In the event that a holding time has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the SQM/QM must be made aware of all holding time violations.

8.2.7.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the hold time excursion and the ultimate resolution is then documented and included in the customer project file.

9.0. GLOSSARY

The source of some of the definitions is indicated previous to the actual definition (e.g., TNI, DoD).

Terms and Definitions	
3P Program	The Pace continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all Pace labs.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies*. The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.

Terms and Definitions	
American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.
Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by Pace as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Terms and Definitions	
Batch	TNI- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Terms and Definitions	
Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100

Terms and Definitions	
Confirmation	TNI- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)

Terms and Definitions	
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence $(1-\alpha)$ that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

Terms and Definitions	
Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.

Terms and Definitions	
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.

Terms and Definitions	
Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).

Terms and Definitions	
Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.
In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.

Terms and Definitions	
Internal Standard	TNI and DoD- A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
LabTrack	Database used by Pace to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Terms and Definitions	
Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.

Terms and Definitions	
Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.

Terms and Definitions	
Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, 1 – β, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
MintMiner	Program used by Pace to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).

Terms and Definitions	
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.

Terms and Definitions	
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Terms and Definitions	
Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Terms and Definitions	
Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.

Terms and Definitions	
Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a chain-of-custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.

Terms and Definitions	
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.

Terms and Definitions	
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

Terms and Definitions	
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where k > 1).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

Terms and Definitions	
Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

10.0. REFERENCES

10.1. "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, most current version.

10.2. "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.

10.3. "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.

10.4. U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis.

10.5. U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis.

10.6. "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.

10.7. "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.

10.8. "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.

10.9. "NIOSH Manual of Analytical Methods", U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health, most current version.

10.10. "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (Sep 1986).

10.11. Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.

10.12. Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.

10.13. Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.

10.14. Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.

10.15. Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.

10.16. Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, most current version.

10.17. National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.

10.18. ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories- most current version.

10.19. Department of Defense Quality Systems Manual (QSM), most current version.

10.20. TNI (The NELAC Institute) Standard- most current version applicable to each lab.

10.21. UCMR Laboratory Approval Requirements and Information Document, most current version.

10.22. US EPA Drinking Water Manual, most current version.

11.0. REVISIONS

The Pace Corporate Environmental Quality Office files an electronic version of a Microsoft Word document with tracked changes detailing all revisions made to previous versions of the Quality Assurance Manual. This document is available upon request. All current revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual 19.0	General: made administrative edits that do not affect the policies or procedures within the document (including revising company name to Pace Analytical Services, LLC). Cover page: removed corporate approval signature lines. Old Section 3: moved to other sections of the QAM as applicable and deleted entire section (All section references below reflect the new section numbers). Section 1.1.2: replaced with section 3.1.1. Sections 1.3, 1.4, 1.11: removed extraneous language. Sections 1.5: added language from old section 1.6. Section 1.6: revised anonymous reporting information. Section 1.7.6: added deputies per position and deleted DoD language from old section 1.7.7. Section 1.8: removed non-key personnel job descriptions. Section 2: rearranged existing sections. Section 2.4: reworded to match existing Sample Acceptance policy document. Section 4: in general, for each QC type, removed language regarding frequency and corrective actions and referenced lab-specific SOPs. Section 5: in general, removed extraneous language and Management of Change section. Section 5.1, 5.2: reorganized into Primary and Secondary Review sections and removed extraneous language. Section 6: removed extraneous language including Quarterly Report section. Section 9 (glossary): revised and added definitions based on 2016 TNI Standard. Section 10: Added EPA DW Manual and revised references as applicable. Attachment III: updated corporate organizational chart. Old Attachment IV: removed floor plan attachment. Old Attachment VII: removed COC (available in SOPs).	10Mar2017
Quality Assurance Manual 19.1	1. Updated SOP references. 2. Update lab org chart. 3. Updated equipment list 4. Updated SOP list. 5. Updated certification list.	18May2018

ATTACHMENT I- QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:
TrueValue = Amount spiked (can also be the $\overline{CF}$ or $\overline{RF}$ of the ICAL Standards)
Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentrtion} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:
R1 = Result Sample 1
R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^{N} W_i * (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} W_i * (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} W_i * (Y_i - \overline{Y})^2\right)}}$$

With:	N	Number of standard samples involved in the calibration
	i	Index for standard samples
	Wi	Weight factor of the standard sample no. i
	Xi	X-value of the standard sample no. i
	X(bar)	Average value of all x-values
	Yi	Y-value of the standard sample no. i
	Y(bar)	Average value of all y-values

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ATTACHMENT I- QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{(n-1)}}$$

where:
n = number of data points
X_i = individual data point
$\overline{X}$ = average of all data points

AVERAGE ($\overline{X}$)

$$\overline{X} = \frac{\sum_{n=1}^{i} X_i}{n}$$

where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

$$RSD = \frac{S}{\overline{X}} * 100$$

where:
S = Standard Deviation of the data points
$\overline{X}$ = average of all data points

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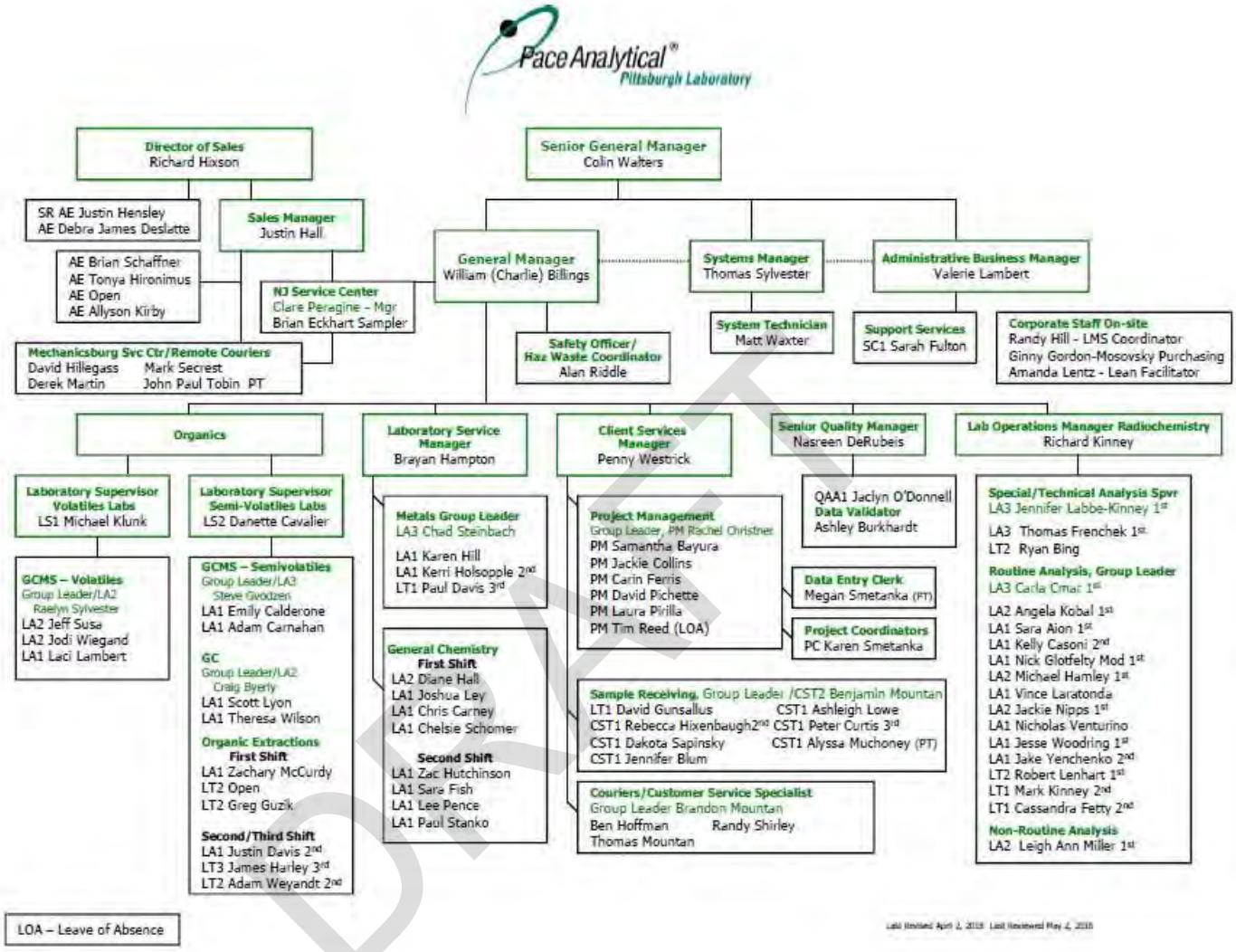
ATTACHMENT II- LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



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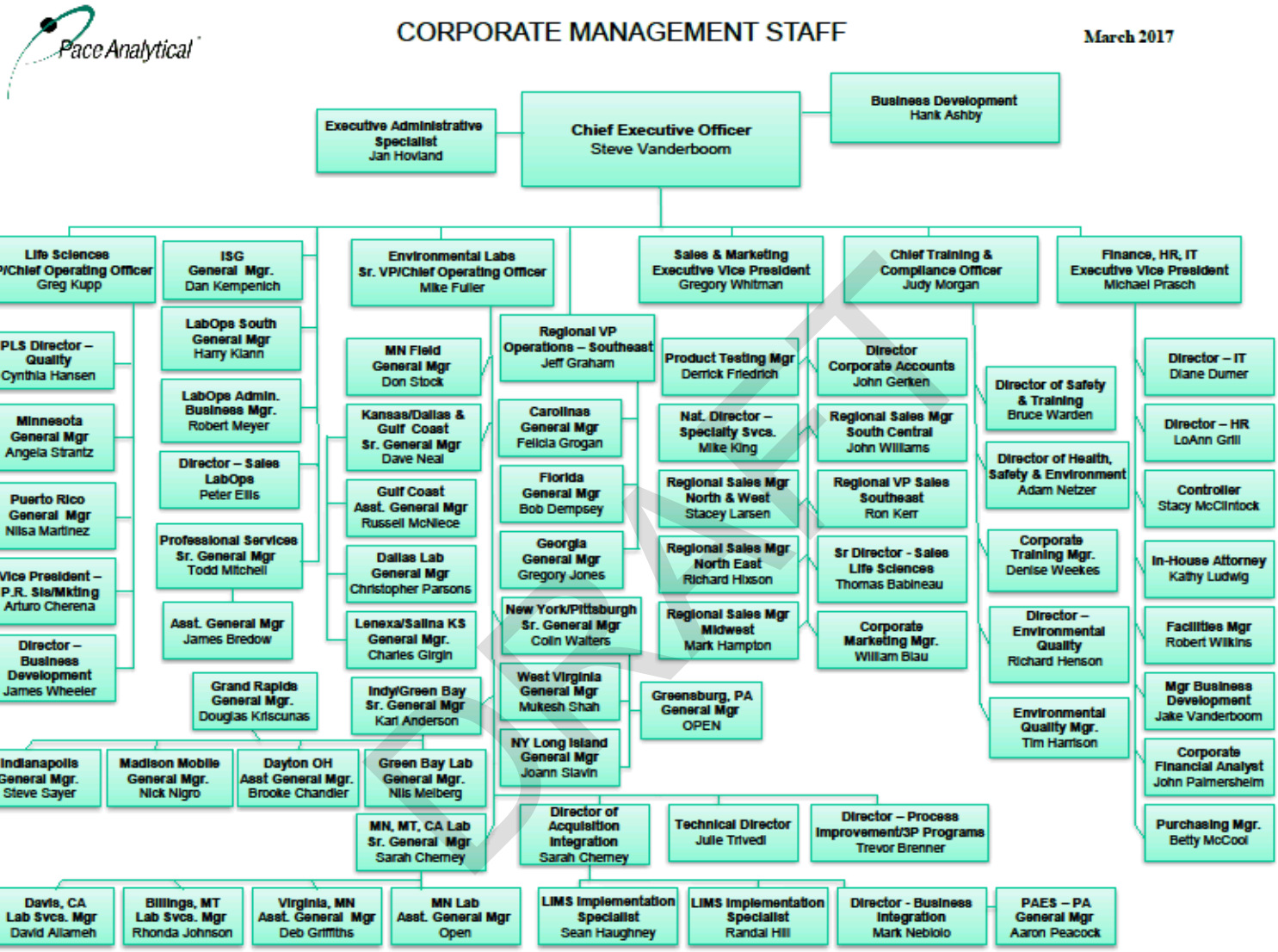
	Document No.: **Quality Assurance Manual Rev. 19.1**	**Issuing Authorities:** **Pace Pittsburgh Quality Office**

ATTACHMENT III- CORPORATE ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



ATTACHMENT IV- EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co Re
ICP	Iteva V2.8.096	CETAC	6500	20090207	ICP-2	ICP-2	PMT's	Metals	Trace Metals	
ICP	Iteva V2.8.097	CETAC	6500	1665DC132619	ICP-3	ICP-3	PMT's	Metals	Trace Metals	
Mercury Analysis	Quick Trace V1.7.6	CETAC	Cetac	Quicktrace M-6100	HG-1	NA	30HG1	Metals	Metals	
Microwave	NA	CEM Corp	MDS2100	ZR8160	NA	NA	NA	Metals	Metals	U
Balance	NA	Mettler-Toledo	XS203S	B08050503		30BAL6	NA	Metals	Metals	U
IC	Chromeleon 7	Dionex	ICS 1100	98100641E991001	IC	30WTA4	IC	WC	Anions	
Automated Spectrometer	Omnion V 4.0	Lachat	8500	150700001870	NA	30WTA7	UV	WC	Wet Chemistry	
Automated Spectrometer	Omnion V3.0	Lachat	8500	120400001408	NA	30WTA5	UV	WC	Wet Chemistry	
Automated Spectrometer	SmartChem V3.1.14	SmartChem	Discrete Analyzer	W0602083	NA	30WTA1	UV	WC	Wet Chemistry	
Spectrophotometer	NA	Milton-Roy	SPEC 21D	3156129024	NA	30WET2	UV	WC	Thiocyanate, MBAS, OrthoP(in EPIC)	
Spectrophotometer	Hach Lange:66	Milton-Roy	DR 5000	1259771	NA	30WETF/ 30WET9	UV	WC	HexCr W/S, Phenol, OrthoP, Res chlorine, COD, MBAS - (EPIC 30WETF is associated with Soil DR-5000 HexCr SEPC - Soil)	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co Re
Rotary Piston Vacuum Pump for O&G	NA	ANVER	PMP-4.3HP	NA	NA	NA	NA	WC	O & G (1664)	
Solid Phase Extractor	NA	Horizon	SPE-Dex 3000XL	00241	NA	NA	NA	WC	O & G (1664)	
Infrared Spectrometer	NA	Perkin Elmer	1310	132724	NA	30WET7	NA	WC	TPH	
Solvent Extractor	NA	Dionex	ASE-200	99090116	ASE-200	NA	NA	WC	Soil Extraction	
Dissolved O2 Meter	Hach Lab V2.1.0.713	Hach	Sension 8	110100050689	NA	30WETA	Meter	WC	BOD/CBOD	U
pH/Ion/Conductivity	NA	Accumet	50	C0021230	NA		Meter	WC	Fluoride	
pH/Ion/Conductivity	NA	Accumet	Acuumet AB250	AB92350833	30WETG	30WETG	Meter	WC	pH	
pH/Ion/Conductivity	NA	Orion Star	A215	X05092	NA	30WETC	Meter	WC	pH, Conductance	
pH/Ion/Conductivity	NA	Precision	Flash Alert	NA	NA	30WET6	NA	WC	Flash Point	
MARS 230/60	NA	CEM	907501	MD9413	1		NA	Oprep	O-Prep	
TOC	NA	OI Analytical	1030	D750788365	NA	30WTA2	UV	WC	TOC	
TOC	NA	Shimadzu	TOC-V WP	638-91064-12 (autosample S/N: 638-93141-08	30WTA8	30WTA8	UV	WC	TOC	
TKN block (new)	SCP V1.4	SCP SCIENTIFIC	HTC 1014220373	TSA1014061434	NA		NA	WC	TKN	
Turbidimeter	NA	Hf Scientific	Micro 100	200802069	NA	30WET8	NA	WC	Turbidity	
COD block	NA	HACH	45600-00	910605052	NA	COD001	NA	WC	COD	
COD block	NA	HACH	45600-00	940100010288	NA	COD002	NA	WC	COD	
Distillation block	NA	NA	NA	NA	NA	DIST003	NA	WC	CN	U
Distillation block	NA	NA	NA	NA	NA	DIST004	NA	WC	CN	U
Distillation block	NA	NA	NA	NA	NA	DIST005	NA	WC	Cyanide	
Distillation block	NA	NA	NA	NA	NA	DIST006	NA	WC	Cyanide	
Pressure Cooker	NA	NA	NA	NA	NA	AC001	NA	WC	Phos	U
Pressure Cooker	NA	NA	NA	NA	NA	AC002	NA	WC	Phos	U
Balance	NA	Mettler	ML802	B329563586	PJ3600	30BAL2	NA	WC	Wet Chemistry	U
Balance	NA	Sartorius	BS 2105	40248175	NA	30BAL7	NA	WC	Wet Chemistry	U

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co R
Balance	NA	Mettler	MS204S/03	B510684209	B510684209	30BAL8	NA	WC	Extra Wet Chemistry	U
Oven #3	NA	Fisher Scientific	NA	NA	OVN003	OVN003	NA	WC	Wet Chemistry	U
Oven #9	NA	Thelco	NA	NA	OVN009	OVN009	NA	WC	Wet Chemistry	U
Oven #10	NA	Fisher Scientific	NA	NA	OVN010	OVN010	NA	WC	Wet Chemistry	U
Incubator #1	NA	NA	NA	NA	INC001	INC001	NA	WC	Wet Chemistry	U
Incubator #2	NA	NA	NA	NA	INC002	INC002	NA	WC	Wet Chemistry	U
Incubator #5	NA	NA	NA	NA	INC005	INC005	NA	WC	Wet Chemistry	U
Incubatory #6	NA	NA	NA	NA	INC006	INC006	NA	WC	Wet Chemistry	U
Incubatory #7	NA	NA	NA	NA	INC007	INC007	NA	WC	Wet Chemistry	U
Refrigerator #10	NA	Kenmore	253.607210 1	WA14800568	NA	NA	NA	WC	Wet Chemistry	U
Refrigerator # 26	NA	Beverage Air	C134	1515739	NA	NA	NA	WC	Wet Chemistry	U
Refrigerator # 27	NA	Kool IT	KSM42	1200WAB2014092 6010	NA	NA	NA	WC	Wet Chemistry	
Refrigerator # 29	NA	Kool IT	KSM42	1200WAB2014122 8055	NA	NA	NA	WC	Wet Chemistry	
Refrigerator # 30	NA	Kool IT	KSM42	1200WAB2014122 8056	NA	NA	NA	WC	Wet Chemistry	
Refrigerator # 33	NA	Kool IT	KSM42	KSM42150723008	NA	NA	NA	WC	Wet Chemistry	
Refrigerator # 39	NA	Avantco	178GDS47	621231171607150 0	NA	NA	NA	WC	Wet Chemistry	
GC/MS	Chemstation 1701BA Rev2.0/Targ et RC-10	Hewlett-Packard	6890/5973	US82321858; oven US00024152	MSS1	30MSS1	MSD	GCMS Semi	GCMS Semivolatil e	/R

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co R
GC/MS	Chemstation 1701EA Rev2.0/Target RC-10	Hewlett-Packard	6890N/5973Network	US01150089; oven US00035050	MSS2	30MSS2	MSD	GCMS Semi	GCMS Semivolatile	/R
GC/MS	Chemstation 1701EA Rev2.0/Target RC-10	Agilent	6890N/5973Network	US43146815; oven CN10435024	MSS3	30MSS3	MSD	GCMS Semi	GCMS Semivolatile	/R
GC/MS	Chemstation 1701EA Rev2.0/Target RC-10	Hewlett-Packard	6890/5975	US52420703; oven US10248098	MSS4	30MSS4	MSD	GCMS Semi	GCMS Semivolatile	/R
GC/MS	Chemstation 1701EA Rev2.0/Target RC-10	Hewlett-Packard	6890A/5975B	US62744417; oven US00037743	MSS5	30MSS5	MSD	GCMS Semi	GCMS Semivolatile	/R
GC/MS	Chemstation 1701EA Rev2.0/Target RC-10	Hewlett-Packard	oven model 7890A; MSD model 5975C	oven SN: CN11281016; MSD SN: US11483920	MSS6	30MSS6	MSD	GCMS Semi	GCMS Semivolatile	/R
Refrigerator #1	NA	Fisher Scientific	13-988-450RW	30330074	NA	NA	NA	GCMS Semi	GC & GCMS Semivolatiles	U
Refrigerator #2	NA	Fisher Scientific	TDX155NSBRHW	GH750959	NA	NA	NA	GCMS Semi	GC & GCMS Semivolatiles	U
Refrigerator / Freezer #41	NA	Isotemp	10FCEEFSA	1162937201170920	NA	NA	NA	GCMS Semi	GC & GCMS Semivolatiles	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co R
Refrigerator/ new walk-in #42	NA	Cold Vault	3678-3-L	DX1801915-01	NA	N	NA	Receiving	Sample Receiving	
GC	Chemstation Rev B.04.02(118)	Hewlett-Packard	7890A	CN10041083	7	30GCS7	FID	GC Semi	DRO	/R
GC	Chemstation Rev B.03.01(317)	Hewlett-Packard	5890A	2643A11529	8	30GCS8	Dual ECD	GC Semi	PCB, 8011	/R
GC	Chemstation Rev B.03.01(317)	Hewlett-Packard	5890 Ser. II	3029A0193	9	30GCS9	Dual ECD	GC Semi	Pest	/R
GC	Chemstation Rev C.00.00	Agilent	6890	US10250070	A	30GCSA	Dual ECD	GC Semi	PCB	/R
GC	Chemstation Rev C.00.00	Agilent	7890B	SN: CN14173047	B	30GCSB	Dual FID	GC Semi	ORO	/R
GC	Chemstation Rev C.00.00	Agilent	7890B	SN: CN17253167 (Autosample tray SN: CN17240018, injector SN: CN17240077)	C	30GCSC	Dual uECD	GC Semi	Pest	
GC	Chemstation Rev C.00.00	Agilent	7890B	SN: CN17313056 (Autosampler SN: CN8174787)	D	30GCSD	Dual uECD	GC Semi	PCB	
Balance	NA	Mettler-Toledo	PL6001E	B651482025	B651482025	30BA26	NA	GC Semi	8011	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co Re
GC/MS (Out of service)	Chemstation 1701BA Rev2.0/Target RC-10	Hewlett-Packard	6890/5973	US00007782/US70820584	HPMS1	30MV1A-B	MSD	GCMS VOA	Volatiles	/R
GC/MS	Chemstation 1701BA Rev2.0/Target RC-10	Hewlett-Packard	6890/5973	DE00004512/US72821154	HPMS2	30MV2A-B	MSD	GCMS VOA	Volatiles	/R
GC/MS	Chemstation 1701BA Rev2.0/Target RC-10	Hewlett-Packard	6890/5973	US00032703/US94223089	HPMS3	30MV3A-B	MSD	GCMS VOA	Volatiles	/R
GC/MS	Chemstation 1701BA Rev2.0/Target RC-10	Hewlett-Packard	6890/5973	US00007768/US70820610	HPMS4	30MV4A-B	MSD	GCMS VOA	Volatiles	/R
GC/MS	Chemstation 1701EA Rev2.0/Target RC-10	Hewlett-Packard	6850/5975	CN11004009/US10050001	HPMS5	30MV5A-B	MSD	GCMS VOA	Volatiles	/R
GC/MS	Chemstation 1701EA Rev2.0/Target RC-10	Hewlett-Packard	6890A/5973N MS	US00040634/US10360153	HPMS6	30MV6A-B	MSD	GCMS VOA	Volatiles	/R
GC/MS	Chemstation 1701EA Rev2.0/Target RC-10	NA	6890N/5975 MS	CN10539039/US53921127	HPMS7	30MV7A-B	MSD	GCMS VOA	Volatiles	/R
P&T Autosampler 30MSV1	NA	Archon	8100	11856-196A	HPMS1	30MSV1	NA	GCMS VOA	Volatiles	/R
P&T Concentrator 1-A	NA	Tekmar	3000	94348006	HPMS1	30MSV1	NA	GCMS VOA	Volatiles	/R
P&T Concentrator 1-B	NA	Tekmar	3000	98082011	HPMS1	30MSV1	NA	GCMS VOA	Volatiles	/R

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co Re
P&T Autosampler 30MSV2	NA	EST	Centurion	144061104	HPMS2	30MSV2	NA	GCMS VOA	Volatiles	/R
P&T Concentrator 2-A	NA	EST	Evolution	EV674060115	HPMS2	30MSV2	NA	GCMS VOA	Volatiles	
P&T Concentrator	NA	EST	Evolution	EV675060115	HPMS2	30MSV2	NA	GCMS VOA	Volatiles	
P&T Autosampler 30MSV3	NA	EST	Centurion	CENT233040307	HPMS3	30MSV3	NA	GCMS VOA	Volatiles	/R
P&T Concentrator 3-A	NA	Tekmar	3000	9924010	HPMS3	30MSV3	NA	GCMS VOA	Volatiles	
P&T Concentrator 3-B	NA	Tekmar	3000	00060005	HPMS3	30MSV3	NA	GCMS VOA	Volatiles	
P&T Autosampler 30MSV4	NA	EST	Centurion	CENT214101206	HPMS4	30MSV4	NA	GCMS VOA	Volatiles	/R
P&T Concentrator 4-A	NA	Tekmar	3000	94259003	HPMS4	30MSV4	NA	GCMS VOA	Volatiles	/R
P&T Concentrator 4-B	NA	Tekmar	3000	94264003	HPMS4	30MSV4	NA	GCMS VOA	Volatiles	/R
P&T Autosampler 30MSV5	NA	EST	Centurion	CENTS397112514	HPMS5	30MSV5	NA	GCMS VOA	Volatiles	
P&T Concentrator 5-A	NA	EST	Evolution	EV234122809	HPMS5	30MSV5	NA	GCMS VOA	Volatiles	
P&T Concentrator 5-B	NA	EST	Evolution	EV623100614	HPMS5	30MSV5	NA	GCMS VOA	Volatiles	
P&T Autosampler 30MSV6	NA	EST	Centurion	CENTS397112514	HPMS6	30MSV6	NA	GCMS VOA	Volatiles	/R
P&T Concentrator 6-A	NA	EST	Evolution	EV690070915	HPMS6	30MSV6	NA	GCMS VOA	Volatiles	/R
P&T Concentrator 6-B	NA	EST	Evolution	EV620092414	HPMS6	30MSV6	NA	GCMS VOA	Volatiles	/R
P&T Autosampler 30MSV7	NA	EST	Centurion	CENTS460063019	HPMS7	30MSV7	NA	GCMS VOA	Volatiles	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co Re
P&T Concentrator 7-A	NA	EST	Evolution	EV758063016	HPMS7	30MSV7	NA	GCMS VOA	Volatiles	
P&T Concentrator 7-B	NA	EST	Evolution	EV757063016	HPMS7	30MSV7	NA	GCMS VOA	Volatiles	
Balance	NA	HRB	1002TL	HR1409140	HR1409140	30BAL15	NA	GCMS VOA	GCMS Volatiles	U
Balance	NA	Mettler	AE240	K89959	AE240	30BAL1	NA	GCMS VOA	GCMS Volatiles	U
GC	Chemstation 1701BA Rev2.0/Target RC-10	Hewlett-Packard	5890 Ser. II	3033A31116	GCV-1	30GCV1	PID/FID	GCMS VOA	GC Volatiles	/R
GC	Chemstation 1701DA Rev2.0/Target RC-10	Hewlett-Packard	6890 N	US10608040	GCV-2	30GCV2	FID	GCMS VOA	GRO	/R
GC	Chemstation 1701BA Rev2.0/Target RC-10	Hewlett-Packard	3890 Ser. II	3121A35926	Screen	No ID	Dual FID	GCMS VOA	GC Volatiles	/R
Refrigerator #4	NA	Fisher Scientific	K89959/ TB15SPFR	MG732472	NA	NA	NA	GCMS VOA	GCMS Volatiles	U
Refrigerator #18	NA	TRUE	GDM-47	4503580	NA	NA	NA	GCMS VOA	GCMS Volatiles	U
Refrigerator #20 (Walk-in)	NA	American Cooler Tech	NA	NA	NA	NA	NA	Samp Receiving	Receiving	
Refrigerator #38 (walk in)	NA	Cold Vault	3678-3-L	DX1604122	NA	NA	NA	GCMS VOA	GCMS Volatiles	
Freezer Chest #7	NA	Kelvinator	NA	NA	NA	NA	NA	GCMS VOA	GCMS Volatiles	U
Muffle Furnace	NA	Fisher Scientific	NA	NA	OVN001	OVN001	NA	OPrep	O-Prep	U
Refrigerator #15	NA	Fisher Scientific	NA	NA	NA	NA	NA	OPrep	O-Prep	U
Refrigerator #40	NA	Frigidaire	FFPA33L2 SM	71601377	NA	NA	NA	OPrep	O-Prep	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co R
Mechanical Shaker	NA	J-Kem Scientific	Max-Q	NA	NA	NA	NA	OPrep	O-Prep	U
Rotator #1	NA	NA	NA	NA	NA	NA	NA	OPrep	O-Prep	U
Rotator #2	NA	NA	NA	NA	NA	NA	NA	OPrep	O-Prep	U
Rotator #3	NA	NA	NA	NA	NA	NA	NA	OPrep	O-Prep	U
ZHE Tumbler #1	NA	Millipore	34R4BFC1-23	0455NZML0008	NA	NA	NA	OPrep	O-Prep	
ZHE Tumbler #2	NA	Millipore	34R4BFC1-23	4555N4003	NA	NA	NA	OPrep	O-Prep	
Hot Plate	NA	Thermolyne	Cimarec3	NA	NA	NA	NA	OPrep	O-Prep	U
TurboVap II #6	NA	Biotage	NA	NA	NA	NA	NA	OPrep	O-Prep	/R
TurboVap II #5	NA	Caliper Life Sciences	NA	NA	NA	NA	NA	OPrep	O-Prep	/R
TurboVap II #3	NA	Zymark	NA	TV9937N9099	NA	NA	NA	OPrep	O-Prep	/R
TurboVap II #4	NA	Zymark	NA	TV9941N9146	NA	NA	NA	OPrep	O-Prep	/R
TurboVap II #7?	NA	Zymark	TurboVap II	NA	NA	NA	NA	OPrep	O-Prep	/R
TurboVap II #9	NA	Zymark	TurboVap II	TV0431N12480	NA	NA	NA	OPrep	O-Prep	Us r
Centrifuge	NA	Thermo Fisher Scientific	Survall ST-8	720016022706 Cat#: 75007200	NA	NA	NA	OPrep	O-Prep	
Oven #6 (dry wghts)	NA	Fisher Scientific	Isotherm 500 Series	NA	OVN006	OVN006	NA	OPrep	O-Prep	U
Oven #11	NA	Fisher Scientific	NA	NA	OVN011	OVN011	NA	OPrep	O-Prep	U
Microwave 1	NA	Mars Xpress	MARS 230/60/ 907501	MD9413	NA	NA	NA	OPrep	O-Prep	U

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co R
Microwave 2	NA	CEM Mars	MARS 6 230/60/ 910900	MJ5218	NA	NA	NA	OPrep	O-Prep	U
Multi-tube Vortexer	NA	Fisher Scientific	NA	170620003	NA	NA	NA	GC & GCMS Semi	GC & GCMS Semivolatiles	
Ultrasonic Bath	NA	Fisher Scientific	Ultrasonic Bath 9.5L	BX0041461710	NA	NA	NA	OPrep	O-Prep	U
pH/Ion/Conductivity	NA	Accumet TCLP#1	AB15	NA	NA	NA	Meter	OPrep	O-Prep	U
pH/Ion/Conductivity	NA	Accumet TCLP#2	AB150	AB92350773	NA	NA	Meter	OPrep	O-Prep	
Balance	NA	Mettler	PL6001E	B614292857		30BA16	NA	OPrep	O-Prep	U
Balance	NA	Mettler	ML802	B435978730		30BA12	NA	OPrep	O-Prep	
Balance	NA	Mettler	PL602E	B615334699		30BA17	NA	OPrep	O-Prep	
Balance	NA	Mettler	PL6001E	B725267510	B725267510	30BA25	NA	OPrep	O-Prep	
Oven #6B	NA	Isotemp	NA	NA	NA	NA	NA	Rad	Radiological	U
Oven #7	NA	Fisher Scientific	NA	NA	NA	NA	NA	Rad	Radiological	U
Oven 13	NA	Grieve	LR-271C	NA	NA	NA	NA	Rad	Radiological	U
Refrigerator #22 (out of service)	NA	Haier	10954	Not on Site	NA	NA	NA	Rad	Radiological	
Refrigerator #37	NA	Haier	HC17SW20RB	BA0A6VM0100TRFSW0983	NA	NA	NA	Rad	Radiological	
Balance	NA	Denver Instruments	XP300	990366	XP300	30BA24	NA	Rad	Radiological	
Balance	NA	Mettler	AE163	88919	88919	NA	NA	Rad	Radiological	
Balance	NA	Mettler-Toledo	MS3002S/03	B435969938	B435969938	30BA23	NA	Rad	Radiological	
Balance (AS Area)	NA	AND	GX8K	14900809	14900809	30BA18	NA	Rad	Radiologic	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co
									al	
Balance (Sample Prep)	NA	AND	GX8K	14900804	14900804	30BA19	NA	Rad	Radiologic al	
Balance	NA	Denver Instrument Corp	A160	B034176	B034176	NA	NA	Rad	Radiologic al	
Balance	NA	Mettler	ME204E	B614292248	B614292248	30BA20	NA	Rad	Radiologic al	
Balance (Ra-228)	NA	Mettler	ME204E	B610197175	B610197175	30BA21	NA	Rad	Radiologic al	
Balance (Ra-228)	NA	Radwag	AS82220R2 WIFI	501949	501949	NA	NA	Rad	Radiologic al	
Balance (Spare)	NA	Mettler	AE240	NA	NA	NA	NA	Rad	Radiologic al	
Balance	NA	Mettler	ME4002E	B618430806	B618430806	30BA22	NA	Rad	Radiologic al	
Liquid Scintillation Counter	Quantasmart V1.31	Packard	Tricarb 2900TR/LL	4CLC01	#2	NA	#2	Rad	Radiologic al	
Alpha/Beta Counter	UMS V 1.09o	Berthold	LB-770	145103-1058	Det 1-10	NA	Det 1-10	Rad	Radiologic al	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	MPC9604	236529-BO	Det 11-14	NA	Det 11-14	Rad	Radiologic al	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	MPC9604	236528-BO	Det 15-18	NA	Det 15-18	Rad	Radiologic al	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	MPC9604	236527-BO	Det 19-22	NA	Det 19-22	Rad	Radiologic al	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	MPC9604	521665	Det 23-26	NA	Det 23-26	Rad	Radiologic al	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	MPC9604	521664	Det 27-30	NA	Det 27-30	Rad	Radiologic al	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	MPC9604	521663	Det 31-34	NA	Det 31-34	Rad	Radiologic al	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	MPC9604	521662	Det 35-38	NA	Det 35-38	Rad	Radiologic al	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument	NPC9604	15289409	Det 39-42	NA	Det 39-42	Rad	Radiologic al	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co
		Corp								
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	NPC9605	15289410	Det 43-46	NA	Det 43-46	Rad	Radiological	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	NPC9606	15289411	Det 47-50	NA	Det 47-50	Rad	Radiological	
Alpha/Beta Counter	PIC Vista 2000 V1.007	Protean Instrument Corp	NPC9607	15289412	Det 51-54	NA	Det 51-54	Rad	Radiological	
Alpha/Beta Counter	PIC Vista 2000 V1.008	Protean Instrument Corp	MPC9604	16147442	Det 55-58	NA	Det 55-58	Rad	Radiological	
Alpha/Beta Counter	PIC Vista 2000 V1.009	Protean Instrument Corp	MPC9604	16147443	Det 59-62	NA	Det 59-62	Rad	Radiological	
Alpha/Beta Counter	PIC Vista 2000 V1.010	Protean Instrument Corp	MPC9604	16147444	Det 63-66	NA	Det 63-66	Rad	Radiological	
Alpha/Beta Counter	PIC Vista 2000 V1.011	Protean Instrument Corp	MPC9604	16147445	Det 67-70	NA	Det 67-70	Rad	Radiological	
Alpha/Beta Counter	PIC Vista 2000 V1.012	Protean Instrument Corp	MPC9604	16147446	Det 71-74	NA	Det 71-74	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Ludlum Measurements Inc.	Model 2200 Scaler	No tag (Detector A)	A	NA	A	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Ludlum Measurements Inc.	Model 2200 Scaler	245722 (Detector B)	B	NA	B	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Ludlum Measurements Inc.	Model 2200 Scaler	No tag (Detector C)	C	NA	C	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Ludlum Measurements Inc.	Model 2200 Scaler	245744 (Detector D)	D	NA	D	Rad	Radiological	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co R
Radium Analysis (Radon Emenation)	NA	Ludlum Measurements Inc.	Model 2200 Scaler	326506 (Detector E)	E	NA	E	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Ludlum Measurements Inc.	Model 2200 Scaler	326509 (Detector F)	F	NA	F	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Ludlum Measurements Inc.	Model 2200 Scaler	324512 (Detector G)	G	NA	G	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Ludlum Measurements Inc.	Model 2200 Scaler	326498 (Detector H)	H	NA	H	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Meter	Model 182	PR227468 (Detector A)	A	NA	A	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Meter	Model 182	PR083007 (Detector B)	B	NA	B	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Meter	Model 182	PR083010 (Detector C)	C	NA	C	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Meter	Model 182	PR261260 (Detector D)	D	NA	D	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Meter	Model 182	PR083011 (Detector E)	E	NA	E	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Meter	Model 182	PR083008 (Detector F)	F	NA	F	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Meter	Model 182	PR083005 (Detector G)	G	NA	G	Rad	Radiological	
Radium Analysis (Radon Emenation)	NA	Meter	Model 182	PR083006 (Detector H)	H	NA	H	Rad	Radiological	
KPA	KPA Win 128	ChemChek	KPA-11	92-45050031	NA	NA	NA	Rad	Radiological	
Gamma Counter	Canberra VAX	Canberra	IGC-4019	2676	Detector 40% A	NA	A	Rad	Radiological	
Gamma Counter (out of service)	Canberra VAX	Canberra	GX 5019	9005136	Detector 50% B	NA	B	Rad	Radiological	
Gamma Counter (out of service)	Canberra VAX	Canberra	GC 6020	9983922	Detector 60% C	NA	C	Rad	Radiological	

Instrument	Software & Version #	Manufacturer	Model No.	Serial No.	Instrument ID	Epic Pro Instr. ID	Detector	Dept	Analysis/ Location	Co
Gamma Counter	Canberra VAX	Canberra	GR 3521	2016166	Detector 35% D	NA	D	Rad	Radiological	
Gamma Counter	Gammavision Windows XP/7	Ortec	GEM-100-S	46-P41426A	Detector #2	NA	#2	Rad	Radiological	
Gamma Counter	Gammavision Windows XP/7	Ortec	GEM-100P4-ST	46-TP41365A	Detector #5	NA	#5	Rad	Radiological	
Gamma Counter (out of service)	Gammavision Windows XP/7	Ortec Module	DSPEC Jr 2.0 V.046	10071606	Detector #3	NA	#3	Rad	Radiological	
Gamma Counter (out of service)	Gammavision Windows XP/7	Ortec Module	DSPEC Jr 2.0 V.046	06116387	Detector #2	NA	#2	Rad	Radiological	
Gamma Counter (out of service)	Gammavision Windows XP/7	Ortec Module	DSPEC Jr 2.0 V.046	06053268	Detector #5	NA	#5	Rad	Radiological	
Alpha Spec	Ortec Alphavision 5.3	Oxford Tennelec	S5HP	37959	Detectors 25 through 40	NA	25-40	Rad	Radiological	
Alpha Spec (out of service)	Canberra VAX	Canberra	7200-04	6972152	Detectors 1 through 24, and 25C through 36C	NA	1-24, 25C-36C	Rad	Radiological	
Sodium Iodide Detectors	Maestro V 7.01	Ortec	Digibase Unispec	14346852 (Detector 1)	#1	NA	1	Rad	Radiological	
Sodium Iodide Detectors	Maestro V 7.01	Ortec	Digibase Unispec	14346844 (Detector 2)	#2	NA	2	Rad	Radiological	
Sodium Iodide Detectors	Maestro V 7.01	Ortec	Digibase Unispec	14346843 (Detector 3)	#3	NA	3	Rad	Radiological	
Sodium Iodide Detectors	Maestro V 7.01	Ortec	Digibase Unispec	14346847 (Detector 4)	#4	NA	4	Rad	Radiological	
Survey Meter	NA	Ludlum Measurements Inc.	3019	25014380	25014380	NA	NA	Rad	Radiological	

ATTACHMENT V- LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

Lab Area	Pace SOP No.	Revision	Document Name	Effective/Review Date	Review Date	Review Date
AD	S-PGH-C-001	14	Sample Management	3/1/2018		
AD	S-PGH-C-008	7	Subcontracting Samples	5/2/2018		
AD	PGH-C-012	3	Customer Complaints	8/29/2017		
AD	PGH-C-016	5	Data Packages	3/10/2017		
AD	PGH-C-017	4	Waste Handling and Management	8/29/2017		
AD	S-PGH-L-027	3	DI Water Quality & Suitability	3/19/2018		
AD	S-PGH-C-028	6	Bottle Prep	12/4/2017		
AD	PGH-C-033	2	Review of Analytical Requests	4/12/2017		
AD	WI-PGH-C-039	2	Documentation of Non-Compliances for Sample Receipt and Handling	12/26/2016		
AD	WI-PGH-C-040	0	Project Login Review of Workorders for Drinking Water Samples	2/10/2017		
AD	S-PGH-F-004	1	Collection of Environmental Samples by Pace Personnel	3/15/2018		
AD	S-ALL-Q-030	5	Data Checker	10/16/2014	10/28/2016	
GC	S-PGH-O-004	12	Diesel Range Organics (DRO) by EPA 8015B & 8015D	3/20/2018		
GC	PGH-O-006	8	Polychlorinated Biphenyls (608)	12/23/2016		
GC	PGH-O-009	13	Polychlorinated Biphenyls (8082-8082A)	12/21/2016		
GC	PGH-O-010	5	Sulfur Cleanup, Method 3660B	2/2/2017		
GC	PGH-O-017	7	Sulfuric Acid Cleanup, Method 3665A	2/21/2017		
GC	PGH-O-019	5	ETPH (Connecticut Method)	3/20/2018		
GC	PGH-O-021	7	OC Pesticide Analysis by GC (608)	12/23/2016		
GC	S-PGH-O-024	15	EDB & DBCP by Method 8011	4/26/2018		
GC	PGH-O-026	10	OC Pesticide Analysis by GC (8081A-8081B)	4/18/2017		
GC	WI-PGH-O-001	1	Materials Cleanliness Protocol	4/18/2017		
GC	WI-PGH-O-002	1	DRO (8015B) - Sparrows Point	4/17/2018		
GC	WI-PGH-O-038	1	Polychlorinated Biphenyls (8082-8082A) Sparrows Point	4/17/2018		
MET	PGH-M-008	17	Determination of Metals by ICP (200.7 and 6010B)	12/23/2016		
MET	PGH-M-011	8	Mercury Prep (Aq)	12/23/2016		
MET	PGH-M-012	10	Mercury Prep (Solid & Semi-solid)	3/21/2018		
MET	PGH-M-013	10	Preparation Solid/Semisolid Samples for ICP Analysis, Method 3050B	3/21/2018		
MET	PGH-M-014	9	Microwave Digestion of Organic Wastes	12/23/2016		
MET	PGH-M-015	11	Preparation of Aqueous Samples for ICP Analysis, Methods 3005A & 200.7	12/23/2016		
MET	PGH-M-017	6	Mercury Analysis by CVAA Cetac	12/23/2016		
OPrep	PGH-M-003	9	TCLP/ZHE Extraction Procedure	8/23/2017		
OPrep	S-PGH-O-016	5	Percent Moisture in Soils ASTM D2974-87	1/22/2018		
OPrep	PGH-O-002	5	Extraction of PCBs from Wipes	7/12/2014	12/28/2016	
OPrep	S-PGH-O-007	8	Solid Phase Extraction of TCLP for SemiVoa Compounds.	4/25/2018		
OPrep	PGH-O-011	8	Extraction of Organic Waste	1/26/2017		
OPrep	PGH-O-020	6	CT-ETPH - Extraction of Aqueous and Solid	4/12/2017		

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			Samples			
OPrep	S-PGH-O-022	8	Microwave Extraction of Solid Samples for Organics	5/17/2018		
OPrep	PGH-O-028	7	Separatory Funnel Extraction	3/26/2018		
OPrep	PGH-O-034	4	SPLP & ZHE Extraction (1312)	5/24/2017		
OPrep	S-PGH-O-036	3	ASTM Leach Extraction	4/25/2018		
OPrep	WI-PGH-O-039	1	Microwave Extraction of Solids - Sparrows Point	2/22/2016	2/28/2018	
QA	S-PGH-Q-020	5	Logbook of Logbooks	3/15/2018		
QA	S-PGH-Q-023	6	Archiving Laboratory Documents	3/15/2018		
QA	PGH-C-031	2	PT Program	7/17/2017		
QA	PGH-C-032	4	Support Equipment	2/27/2017		
QA	S-PGH-L-036	2	Purchase of Laboratory Supplies	3/1/2018		
QA	PGH-C-037	0	Standard and Reagent Management and Traceability	9/9/2013	6/1/2017	
QA	PGH-C-038	2	Receipt and Storage of Laboratory Supplies	8/31/2017		
QA	PGH-Q-022		Spreadsheet Validation	12/26/2016		
QA	S-PGH-Q-025	5	Reporting SDWA MCL Violations	5/9/2018		
QA	S-PGH-Q-035	3	MDL/LOD/LOQ	12/14/2017		
QA	PGH-Q-037	3	Data Review Process	12/10/2014	10/28/2016	
QA	S-PGH-Q-038	1	Laboratory Equipment	5/16/2018		
QA	PGH-Q-039	2	Corrective And Preventative Action	3/10/2017		
QA	PGH-Q-040	0	Internal and External Audits	9/23/2014	10/28/2016	
QA	PGH-Q-041	0	Evaluation and Qualification of Vendors	9/23/2014	10/28/2016	
QA	PGH-Q-042	0	Regulatory Limit Notification	10/20/2014	10/28/2016	
QA	S-PGH-Q-043	2	Document Control and Management	3/9/2018		
QA	PGH-Q-044	0	Monitoring Storage Units	11/21/2014	10/28/2016	
QA	PGH-Q-045	0	Control Charts & Acceptance Limits	7/16/2015	6/1/2017	
QA	PGH-Q-046	0	Estimation of Measurement Uncertainty	7/20/2015	6/1/2017	
QA	PGH-Q-047	0	Management of Change	8/14/2015	6/1/2017	
QA	PGH-Q-048	0	Sample Homogenization and Sub-sampling	8/18/2015	6/1/2017	
QA	S-PGH-L-009	6	Glassware Washing	4/5/2018		
QA	S-PGH-Q-001	11	Preparation of Standard Operating Procedures	3/9/2018		
QA	S-All-Q-003	11	Document Numbering Procedure	3/9/2018		
QA	S-All-Q-009	8	General Documentation Requirements	3/9/2018		
QA	S-All-Q-014	8	Quarterly Quality Report	5/3/2018		
QA	S-All-Q-015	3	Review of Laboratory Management System	3/9/2018		
QA	S- All-Q-016	8	Manual Integration	4/17/2017		
QA	S-All-Q-020	6	Orientation and Training Procedures	7/20/2015	6/1/2017	
QA	S-All-Q-028	4	Use and Operations of Lab Track System	3/16/2018		
QA	S-All-Q-029	3	MintMiner Data File Review for Data Integrity Monitoring	3/16/2018		
QA	S-All-Q-035	3	Data Recall	3/16/2018		
QA	S-All-Q-047	0	Method Validation and Instrument Verification	3/8/2018		
QA	WI-PGH-Q-001	0	Marathon Analysis Guide	6/11/2014	3/22/2016	5/1/2018

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QA	WI-PGH-Q-002	0	NJ Data of Known Quality Guide	4/15/2015	4/18/2016	5/1/2018
QA	QA Manual	19	Quality Assurance Manual	3/10/2017		
RAD	S-PGH-R-001	19	Analysis of samples for Gross Alpha and Gross Beta - 900.0 & SM 7110C	2/8/2018		
RAD	S-PGH-R-002	6	Gas Flow Proportional Counter Instrument Operations	3/21/2018		
RAD	S-PGH-R-003	19	Analysis of Water Samples for Ra-228 Content - 904.0	2/8/2018		
RAD	S-PGH-R-004	14	Analysis of Water Samples for Total Alpha Radium - 903.0, SM7500	2/8/2018		
RAD	S-PGH-R-005	14	Analysis of Water Samples for Sr90 Content - 905.0	2/8/2018		
RAD	S-PGH-R-007	18	Analysis of Water Samples for Ra-226 Content - 903.1	2/8/2018		
RAD	S-PGH-R-008	13	Analysis of Samples for Alpha Emitting Actinides and Pu-241	2/8/2018		
RAD	S-PGH-R-010	9	Sr-89/90 by Extraction Chromatography ASTM D5811-08 (2013)	3/15/2018		
RAD	S-PGH-R-013	6	Ni-59/Ni-63 Analysis Eichrom	3/27/2018		
RAD	PGH-R-014	3	Analysis of Iron-55	2/15/2017		
RAD	PGH-R-015	3	Analysis of samples for Technetium-99	2/15/2017		
RAD	S-PGH-R-017	6	Glassware Cleaning	2/28/2018		
RAD	S-PGH-R-018	4	Radioactive Standards Preparation	3/15/2018		
RAD	S-PGH-R-020	10	Alpha Spectroscopy Instrument Operation	3/1/2018		
RAD	S-PGH-R-021	17	Tritium in Water - Distillation - 906.0	2/8/2018		
RAD	S-PGH-R-022	5	Liquid Scintillation Counting	3/15/2018		
RAD	S-PGH-R-023	12	Gamma Spec Instrument Operations - 901.1	2/2/2018		
RAD	PGH-R-024	4	Rad Sample Preparation	3/2/2016	3/6/2017	
RAD	S-PGH-R-027	4	Neutron Dosimeter Wires by Gamma Spec	2/15/2018		
RAD	S-PGH-R-028	4	Neutron Dosimeter Capsules for Cs-137	2/15/2018		
RAD	S-PGH-R-030	4	Analysis of samples for I-129	2/13/2018		
RAD	S-PGH-R-031	12	Total Uranium by KPA	2/8/2018		
RAD	S-PGH-R-032	11	State of NJ 48Hr Gross Alpha Analysis	2/9/2018		
RAD	PGH-R-034	3	Analysis of C-14	2/15/2017		
RAD	PGH-R-037	10	Radon in Water	4/17/2017		
RAD	PGH-R-038	2	Dosimetry Foils for Niobium	11/21/2014	3/6/2017	
RAD	S-PGH-R-040	7	Gamma Spectroscopy Analysis - Prep - 901.1	3/1/2018		
RAD	S-PGH-R-041	4	Analysis of Polonium-210	2/9/2018		
RAD	PGH-R-042	5	Analysis of samples for Pb-210	7/13/2017		
RAD	WI-PGH-R-063	1	Radioactive Calibrations	2/28/2018		
RAD	PGH-R-064	0	Isotopic Radium Analysis in Water; Ra-226 and Ra-223/224 by Alpha Spec -Eichrom	10/27/2014	3/6/2017	
RAD	PGH-R-065	0	Alpha Scintillation Counter Operations	11/25/2015	2/6/2017	2/18/2018
RAD	S-PGH-R-066	1	Analysis of Gaseous Samples for Radon by Alpha Scintillation Counting	2/9/2018		
RAD	WI-PGH-R-067	1	Rad Sample pH checks	2/8/2018		
RAD	WI-PGH-R-068	1	Receipt of Sample Packages Marked Radioactive (UN2910)	11/7/2017		

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RAD	WI-PGH-R-069	0	Work Instruction for Treatment of Aqueous Waste for Isotopic Radium Analyses Utilizing Ba-133	10/12/2017		
RAD	WI-PGH-R_070	0	Work Instruction for Sample Management of Westinghouse Dosimetry projects	10/13/2017		
RAD	PGH-R-069	1	Analysis of Drinking Water Samples for Gross Alpha and Gross Beta Content, Method EPA 900.0	5/8/2017	2/28/2018	
RAD	PGH-R-071	1	Analysis of Drinking Water Samples for Ra-228, Method 904.0	11/20/2017		
RAD	PGH-R-072	0	Analysis of Drinking Water Samples for Total Alpha Radium, Method: EPA 903.0	2/27/2017	2/28/2018	
RAD	PGH-R-074	0	Total Uranium Content of Drinking Water Samples by Laser Kinetic Phosphorimetry Analysis (KPA) Method: ASTM D5174	2/24/2017	2/28/2018	
RAD	PGH-R-075	0	Coprecipitation Method for Gross Alpha Radioactivity in Drinking Water Methods: SM 7110C-00	2/27/2017	2/28/2018	
SVOA	PGH-O-001	14	Semivolatiles by GC/MS (8270C & 8270D)	3/21/2018		
SVOA	PGH-O-003	8	Semivolatiles by GC/MS (625)	5/24/2017		
SVOA	PGH-O-023	8	PAH's by SIM	3/20/2018		
SVOA	WI-PGH-O-004	1	Initial Calibration Procedure for GC/MS Methods	3/27/2018		
Safety	SOP-All-S-005-0	0	Air Quality Monitoring and Fume Hood Monitoring	11/22/2017		
Safety	PGH-S-001	3	Rescue Alert System Operation	4/17/2017		
Safety	PGH-S-002	0	Radiation Safety Compliance	6/7/2017		
Safety	S-ALL-S-001	5	Hazard Assessment	4/17/2017		
Safety	Rad Safety Manual	4	Radiation Safety Manual	4/7/2017		
VOA	PGH-O-012	3	Preparation of EnCore Solid Samples and Terracore solid samples by EPA Method 5035A	7/27/2016		
VOA	PGH-O-015	14	Volatile Organic Compounds by EPA Methods 8260B & 8260C	3/23/2018		
VOA	PGH-O-016	12	Gasoline Range Organics (GRO) by EPA Method 8015B & 8015D	3/20/2018		
VOA	PGH-O-033	8	Volatile Organic Compounds by EPA Method 624	3/20/2018		
VOA	WI-PGH-O-003	1	GRO (8015B) - Sparrows Point	3/20/2018		
VOA	S-All-O-038	2	Processing TICs for GCMS	3/9/2018		
WC	PGH-I-003	8	pH in Water, Soil & Waste	8/22/2017		
WC	PGH-I-004	11	Phenolics	11/1/2017		
WC	S-PGH-I-009	12	BOD/CBOD - SM 5210-B-2011	2/14/2018		
WC	PGH-I-010	8	Sulfide	8/8/2017		
WC	S-PGH-I-011	12	Orthophosphate	4/20/2018		
WC	S-PGH-I-012	14	Hexavalent Chromium	5/9/2018		

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WC	S-PGH-I-013	14	Non-Filterable Residue (Total Suspended Solids, TSS) - 2540D-1997	5/9/2018		
WC	S-PGH-I-015	10	Alkalinity	4/18/2018		
WC	PGH-I-016	6	Acidity - Titrimetric	11/8/2017		
WC	PGH-I-017	6	Reactive Cyanide and Sulfide	8/14/2017		
WC	PGH-I-019	6	Paint Filter Liquids Test	4/24/2017		
WC	S-PGH-I-020	12	Filterable Residue (TDS), SM 2540C-1997	5/9/2018		
WC	S-PGH-I-021	6	Pensky-Martens Closed-Cup Method for Determining Ignitability	5/17/2018		
WC	PGH-I-024	8	Turbidity	12/22/2016		
WC	S-PGH-I-025	9	Fluoride	5/1/2018		
WC	S-PGH-I-027	8	Total Kjeldahl Nitrogen (TKN)	4/19/2018		
WC	PGH-I-028	10	Color	8/9/2017		
WC	PGH-I-030	9	Nitrate/Nitrite	12/12/2016		
WC	S-PGH-I-031	11	Chloride (by Lachat)	5/9/2018		
WC	S-PGH-I-033	9	Chemical Oxygen Demand	3/2/2018		
WC	S-PGH-I-035	12	Ammonia	4/20/2018		
WC	PGH-I-037	5	Sulfite	12/20/2016		
WC	S-PGH-I-038	8	Residual Chlorine	5/9/2018		
WC	S-PGH-I-039	16	Total Solids (TS) and Total Volatile Solids (TVS)	5/9/2018		
WC	PGH-I-042	12	Oil & Grease in water by SPE (EPA 1664)	12/20/2016		
WC	PGH-I-045	8	Dissolved Oxygen	8/7/2017		
WC	S-PGH-I-047	7	Settleable Material	5/11/2018		
WC	PGH-I-050	9	Methylene Blue Activated Substances (MBAS)	12/21/2016		
WC	PGH-I-052	8	O&G/TPH Soxhlet (hexane)	12/20/2016		
WC	PGH-I-053	16	Cyanide: Total and Amenable	9/11/2017		
WC	PGH-I-054	7	Nitrite - Smartchem	12/16/2016		
WC	PGH-I-055	8	Thiocyanate	5/18/2017		
WC	S-PGH-I-056	12	Sulfate - Smartchem - ASTM D516-11 and EPA 9038	1/22/2018		
WC	S-PGH-I-057	14	Phosphorus - SmartChem, 4500-P B (5)-11, 4500-P E-11	1/25/2018		
WC	S-PGH-I-058	5	Ferrous Iron -SmartChem	4/18/2018		
WC	S-PGH-I-059	11	Anions by Ion Chromatography	3/2/2018		
WC	PGH-I-060	8	Total Organic Carbon	12/16/2016		
WC	S-PGH-I-062	5	Specific Conductance	4/18/2018		
WC	S-PGH-I-065	1	Fluoroborate	4/20/2018		
WC	S-PGH-I-066	3	Alkaline Digestion for Cr+6 (3060A)	4/4/2018		
WC	S-PGH-I-067	1	Free Cyanide	4/20/2018		
SOM	S-SOM-C-001	0	Support Equipment	3/20/2017		
SOM	S-SOM-F-001	0	Measuring Temperature in the Field	3/20/2017	3/22/2018	
SOM	S-SOM-F-002	0	Measuring pH in the Field	3/20/2017	3/22/2018	
SOM	S-SOM-F-003	0	DO in the Field	3/20/2017	3/22/2018	
SOM	S-SOM-F-004	0	Specific Conductance in the Field	4/21/2017	5/2/2018	
SOM	S-SOM-F-005	0	Turbidity in the Field	4/25/2017	5/2/2018	
SOM	S-SOM-F-006	1	Total Residual Chlorine in the Field	5/2/2018		
SOM	S-SOM-F-007	0	Field Sampling	9/25/2017		

ATTACHMENT VI- LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE)

SCOPE AND APPLICATION CERTIFICATES ARE MAINTAINED AND FILED IN THE LOCAL QUALITY DEPARTMENT

Laboratory: Pittsburgh Environmental Certifications

Accrediting Authority	Program Category	Accrediting Agency	Certification #/ Lab ID
Connecticut	Waste Water & Hazardous Waste - Solid	DOPH	PH-0694
Maine	Waste Water	DOH&HS	PA01457
New Hampshire	Waste Water & Hazardous Waste - Solid	DES	2976
New Jersey	Waste Water & Hazardous Waste - Solid	DEP	PA-051
New Jersey	Drinking Water and Waste Water	DEP	11050 (Pace Somerset NJ)
New York	Waste Water & Hazardous Waste - Solid	DOH - ELAP	10888
Pennsylvania	Drinking Water (RAD)	DEP	65-00282
Pennsylvania	Waste Water & Hazardous Waste - Solid	DEP	65-00282
PA Rad License	Materials License	NRC	PA-1057
USDA	Soil Permit	USDA	P330-17-00091
USDA	Compliance Agreement PPQ form 519	USDA	P-SOIL-03
West Virginia	Waste Water & Hazardous Waste - Solid	DEP	143
Laboratory: Pittsburgh Radiological Certifications			
Alabama	Drinking Water	DEM	41590
Arizona	Drinking Water	DOHS	AZ0734
Arkansas	Drinking Water	DEQ	NA
California	Drinking Water & Hazardous Waste	DOH	04222CA
Colorado	Drinking Water	DPH&E	NA
Connecticut	Drinking Water, Waste Water and Hazardous Waste	DPH	PH-0694
EPA Region 4	Drinking Water	EPA	NA
EPA Region 5	Drinking Water	US EPA	NA
Delaware	Drinking Water	H&SS	NA
DoD	Waste Water & Hazardous Waste	ANAB	L2417
Florida	Drinking Water & Waste Water	DOH	E87683

Laboratory: Pittsburgh Environmental Certifications

Accrediting Authority	Program Category	Accrediting Agency	Certification #/ Lab ID
Georgia	Drinking Water	DNR	C040
Guam	Drinking Water	EPA	NA
Hawaii	Drinking Water	DOH	NA
Idaho	Drinking Water	DOH&W	NA
Illinois	Drinking Water	DEP	NA
Indiana	Drinking Water	DEP	NA
Iowa	Drinking Water	DNR	391
Kansas	Drinking Water	DOH&EC	E-10358
Kentucky DW	Drinking Water	DEP	90133
Kentucky WW	Waste Water	DEP	90133
Los Angeles Sanitation	Waste Water	Sanitation District	10257
Louisiana	Drinking Water	DHH	LA170007
Louisiana	Waste Water & Hazardous Waste - Solid	DEQ	04086
Maine	Drinking Water & Waste Water	DH & HS	PA01457
Maryland	Drinking Water	DOH&MH	308
Massachusetts	Drinking Water	DEP	M-PA1457
Michigan	Drinking Water	DEQ	NA
Missouri	Drinking Water	DONR	235
Montana	Drinking Water	DOPH&HS	Cert0082
Nebraska	Drinking Water	DOH&HS	NE-OS-29-14
Nevada	Drinking Water, Waste Water & Hazardous Waste	DOC&NR	PA014572017-1
New Hampshire	Drinking Water, Waste Water	DES	2976
New Jersey	Drinking Water	DEP	PA051
New Mexico	Drinking Water, Waste Water and Hazardous Waste	DPNR	PA01457
New York	Drinking Water, Waste Water	DOH	10888
North Carolina	Drinking Water	DOH&HS	42706
North Dakota	Drinking Water, Waste Water & Hazardous Waste	ND DOH	R-190
Ohio	Drinking Water	OH EPA	41249
Oregon	Drinking Water, Waste Water and Hazardous Waste	ORELAP	PA200002
Pennsylvania	Drinking Water, Waste Water and Hazardous Waste	DEP	65-00282
Pennsylvania	Rad License	DEP - BRP	PA-1057

Laboratory: Pittsburgh Environmental Certifications

Accrediting Authority	Program Category	Accrediting Agency	Certification #/ Lab ID
Puerto Rico	Drinking Water	DOH	PA01457
Rhode Island	Drinking Water	DOH	65-00282
South Dakota	Drinking Water	DOE&NR	PA01457
Tennessee	Drinking Water	DEC	02867
Texas	Drinking Water	COEQ	T104704188-16-11
US Virgin Islands	Drinking Water	DPNR	NA
Utah	Drinking Water, Waste Water and Hazardous Waste	DOH	PA014572017-9
Vermont	Drinking Water	DOH	VT-0282
Virginia (VELAP)	Drinking Water, Waste Water and Hazardous Waste	DGS	460198
Washington	Drinking Water	DOE	C868
West Virginia	Drinking Water	DOH	9964C
West Virginia	Waste Water & Hazardous Waste	DEP	143
Wisconsin	Drinking Water	DOH	NA
Wyoming	Drinking Water	DEP	8TMS-L

ATTACHMENT VII- METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE (CURRENT AS OF ISSUE DATE)

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acid Base Accounting	Sobek	Solid	Plastic/Glass	None	N/A
Acidity	SM2310B	Water	Plastic/Glass	≤ 6ºC	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	≤ 6ºC	14 Days
Actinides	HASL-300	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid	Plastic/Glass	None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass (NY requires separate bottle filled to the exclusion of air)	≤ 6ºC	14 Days
Alkylated PAHs		Water	1L Amber Glass	≤ 6ºC; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid	8oz Glass	≤ 10ºC	1 Year/40 Days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4 , bromate, chlorite, chlorate)	300.0/300.1/SM4110 B	Water	Plastic/Glass	≤ 6ºC; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4 , bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	≤ 6ºC	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	≤ 6ºC	48 hours
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; ≤ 6ºC; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Asbestos	EPA 600/R-93/116	Solid	Plastic/Glass; bulk- 2" square; popcorn ceiling- 2tbsp; soil- 4oz	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤ 6ºC; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	≤ 6ºC	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; ≤ 6ºC; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	≤ 6ºC; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6ºC; pH<2 1:1 HCl (optional)
Biomarkers		Solid	≤ 10ºC	1 Year/40 Days	≤ 10ºC
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6ºC	48 hours
Boiling Range Distribution of Petroleum Fractions	ASTM D2887-98	Product	10mL glass vials	≤ 6ºC	N/A
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	28 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	72 Hours
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloroacetic acid pH <3; ≤ 6ºC	28 Days
Carbamates	8318	Water	Glass	Monochloroacetic acid pH 4-5; ≤ 6ºC	7/40 Days
Carbamates	8318	Solid	Glass	≤ 6ºC	7/40 Days
Carbon Specific Isoptope Analysis (CSIA)	AM24	Water	40mL clear VOA vial with TLS	≤ 6ºC, trisodium phosphate or HCl	N/A
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Cations (Ferrous Iron, Ferric Iron, Divalent Manganese)	7199 modified	Water	40mL clear VOA vials with mylar septum	≤ 6ºC; HCl	48 Hours
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorinated Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil	≤ 6ºC	48 Hours to filtration
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	24 Hours
Coliform, Fecal	SM9221E	Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9221E	Solid	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	24 Hours
Coliform, Total	SM9222B	Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Coliform, Total	SM9221B	Solid	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Coliform, Total, Fecal and E. coli	Colilert/ Quanti-tray	Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	30 Hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6ºC	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/ 9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6ºC; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present- applies to SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	≤ 6ºC	14/40 Days
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6ºC	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10ºC	1 Year if frozen/40 Days
Diesel Range Organics- TPH DRO	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6ºC but above freezing	28 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6ºC	14/40 Days; 7 Days from collection to

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
					extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6ºC	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6ºC; pH <2 HCl	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6ºC	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	1 year
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6ºC	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6ºC	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10ºC	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	7/40 Days
Diquat/Paraquat	549.2	Water	Amber Plastic	≤ 6ºC; $Na_2S_2O_3$	7/21 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6ºC; $Na_2S_2O_3$ if Cl present	14 Days
Endothall	548.1	Water	Amber Glass	≤ 6ºC; $Na_2S_2O_3$	7/14 Days
Enterococci	EPA 1600	Water	100mL Plastic	≤ 10ºC	8 Hours
Enterococci	Enterolert	Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Explosives	8330/8332	Water	1L Amber Glass	≤ 6ºC	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Water	1L Amber Glass	pH < 2 HCl; ≤ 6ºC	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Solid	4oz Glass Jar	≤ 6ºC	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6ºC	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6ºC	7/40 Days
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	Glass	None	Immediate
Flashpoint/ Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	≤ 6ºC; pH <2 H_2SO_4 or HCl	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics (C3-C10)	8260B modified	Water	40mL vials	≤ 6ºC; HCl	14 Days
Gasoline Range Organics (C3-C10)	8260B modified	Solid	4oz Glass Jar	≤ 6ºC	14 Days
Gasoline Range Organics- Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics- Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤ 6ºC	14 Days
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6ºC	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	≤ 6ºC; packed jars with no headspace	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; ≤ 6ºC	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	≤ 6ºC in MeOH	21 Days
Glyphosate	547	Water	Glass	≤ 6ºC; $Na_2S_2O_3$	14 Days (18 Months frozen)
Grain Size	ASTM D422	Solid	Not specified	Ambient	N/A
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤ 6ºC	14/7 Days if extracts stored ≤ 6ºC or 14/14 Days if extracts stored at ≤ -10ºC
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	≤ 10ºC; $Na_2S_2O_3$	8 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days
Herbicides,	8151	Water	1L Amber	≤ 6ºC; $Na_2S_2O_3$ if Cl	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Chlorinated			Glass	present	
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/ SM3500Cr-B, C, D	Water	Plastic/Glass	≤ 6ºC	24 Hours (see note 4)
Hexavalent Chromium	218.6/SM3500Cr-B, C, D	Water	Plastic/Glass	Ammonium Buffer pH 9.3-9.7	28 Days (see note 4)
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH >8	14 Days (see note 4)
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6ºC	30 Days from collection to extraction and 7 days from extraction to analysis
Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Hydrogen by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Light Hydrocarbons by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Light Hydrocarbons in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Lipids	Pace Lipids	Tissue	Plastic/Glass	≤ -10ºC	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	≤ - 10ºC	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	≤ 6ºC	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	≤ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175; PM01/AM20GAx	Water	20mL vials	HCl; or trisodium phosphate or benzalkonium chloride and ≤ 6°C	14 Days; 7 Days unpreserved
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	28 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
Methanol, Ethanol	8015 modified	Water	40mL vials	≤ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	≤ 6°C	14 Days
Methyl Mercury	1630	Water	Teflon/ fluoropolymer	Fresh water- 4mL/L HCl; Saline water- 2mL/L H_2SO_4 (must be preserved within 48 hours of collection)	6 months
Methyl Mercury	1630	Tissue	2-4oz glass jar	≤ 0°C	28 Days; ethylated distillate 48 hours
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	28 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	72 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6ºC	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6ºC	28 Days
Oil Range Organics	8015	Solid	Glass	≤ 6ºC	14/40 Days
Oil Range Organics	8015	Water	Glass	≤ 6ºC	7/40 Days
Organic Matter	ASA 29-3.5.2	Solid	Plastic/Glass	None; samples air-dried and processed prior to analysis	N/A
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Oxygenates on Product (GCMS SIM)	1625 modified	Product	10mL glass vial	≤ 6ºC	14 Days (7 Days from extraction)
PBDEs	1614	Water	1L Amber Glass	≤ 6ºC	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6ºC	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10ºC	1 Year/1 Year
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	Pest: 7/40 Days; PCB: 1 Year/1 Year
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3; pH<2 HCl; ≤ 6ºC	14/30 Days
PCBs, total as Decachlorobiphenyl	508A	Water	1L Glass, TFE lined cap	≤ 6ºC	14/30 Days
Perchlorate	331	Water	Plastic/Glass	≥0-6ºC, field filtered with headspace	28 Days
Permanent Gases (O2, N2, CO2)	RSK-175; PM01/AM20GAx	Water	40mL vials	benzalkonium chloride and ≤ 6ºC	14 Days
Permanent Gases by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Permanent Gases in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	≤ -10ºC	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; ≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6ºC	1 Year/1 Year
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	≤ -10ºC	1 Year if frozen/1 Year
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6ºC but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6ºC but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10ºC	1 Year/1 Year
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particle Size	ASA 15-5 modified	Solid	Plastic/Glass (100g sample)	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	28 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	7 Days
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	≤ 6ºC	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6ºC	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6ºC but above freezing	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6ºC	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10ºC	1 Year if frozen/40 Days
Purgeable Organic Halides (POX)	9021	Water	Glass; no headspace	≤ 6ºC	14 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note	9320	Solid	Plastic/Glass		

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
3)					
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	≤ 6ºC	14/40 Days
Saturated Hydrocarbons		Water	≤ 6ºC; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6ºC; pH<2 1:1 HCl (optional)
Saturated Hydrocarbons		Solid	≤ 10ºC	1 Year/40 Days	≤ 10ºC
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6ºC	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6ºC	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6ºC	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6ºC	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6ºC	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6ºC	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6ºC	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6ºC	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6ºC	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6ºC	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6ºC	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; $Zn(OAc)_2$; ≤ 6ºC	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6ºC	48 Hours
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid	Plastic/Glass	None	180 days
Total Inorganic Carbon (TIC)	PM01/AM20GAx	Water	40mL VOA vial with mylar septum	≤ 6ºC	14 Days
Total Organic Carbon	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
(TOC)				HCl; ≤ 6ºC	
Total Organic Carbon (TOC)	9060/Walkley Black/Lloyd Kahn	Solid	Glass	≤ 6ºC	14 Days
Total Organic Halogen (TOX)	SM5320/9020	Water	Glass; no headspace	≤ 6ºC	14 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Water	40mL vials	pH<2 HCl, no headspace, ≤ 6ºC	7 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Solid	Glass	≤ 6ºC	14 days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6ºC	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
UCMR Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days
UCMR Hexavalent Chromium	218.7	Water	HDPE or propylene	Na_2CO_3/$NaHCO_3$/($NH_4)_2SO_4$; pH>8	14 Days
UCMR Chlorate	300.1	Water	Plastic or glass	EDA	28 Days
UCMR Perfluorinated Compounds	537	Water	Polypropylene	Trizma	14 Days
UCMR 1, 4 Dioxane	522	Water	Glass	Na_2SO_3, $NaHSO_4$; pH<4	28 Days
UV254	SM5910B	Water	Glass	≤ 6ºC	48 Hours
Vermiculite	EPA 600/R-93/116	Solid	Plastic/Glass	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Volatile Fatty Acids	AM21G	Water	40mL clear VOA vials	≤ 6ºC	21 Days
Volatile Fatty Acids (low level)	AM23G	Water	40mL clear VOA vials	≤ 6ºC with benzalkonium chloride	14 Days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6ºC	14 Days preserved
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6ºC; packed jars with no headspace	7/28 Days
Volatiles	TO-14	Air	Summa Canister	None	28 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	TO-15	Air	Summa Canister or Tedlar Bag	None	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Volatiles	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6ºC but above freezing	28 Days
Volatiles	TO-18/8260	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	8260	Solid	5035 vial kit	See note 1 (analyze for acrolein and acrylonitrile per local requirements)	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6ºC; $Na_2S_2O_3$ if Cl present (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6ºC	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6ºC; $Na_2S_2O_3$ if Cl present (or unpreserved if run within 7 days of collection) (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6ºC; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days
Whole Oil	ASTM D3328 (prep); ASTM D5739	Product	10mL glass vials	≤ 6ºC	N/A

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4ºC, **and** one vial methanol preserved at ≤6ºC **and** one container of unpreserved sample stored at ≤6ºC.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA Method Update Rule. Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate buffer to extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR program.

ATTACHMENT A-3

QUALITY ASSURANCE MANUAL, QUALITY ASSURANCE/QUALITY CONTROL POLICIES AND PROCEDURES, PACE ANALYTICAL ENERGY SERVICES, LLC-PITTSBURGH

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QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures

Pace Analytical Energy Services, LLC – Pittsburgh
220 William Pitt Way Pittsburgh, PA 15238

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Table of Contents

1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"
Pace Analytical Services LLC - Mission Statement

1.1. Introduction to Pace

1.1.1. Pace Analytical Services, LLC is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. Pace offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. This document defines the Quality System and Quality Assurance (QA)/Quality Control (QC) protocols.

1.1.2. Pace laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. Methods are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies.

1.1.3. Pace Analytical Energy Services, LLC. (PAES) recognizes its crucial role in providing reliability and excellence in the environmental analytical industry. The laboratory provides information necessary for engineering, industrial, and regulatory clients to make informed judgments and applicable policy decisions. PAES' management acknowledges that uncompromising dedication to quality is fundamental to remaining a competitive force in the analytical services market. The scope of services includes:

Wastewater and storm water

- Ion analysis via Ion Chromatography
- Wet Chemistry analyses for pH and TOC/DOC

In Situ Remediation Analyses

- Dissolved Gas (Oxygen, Nitrogen, Carbon Dioxide, Carbon Monoxide, Hydrogen, Acetylene, Methane, Ethane, Ethene, Propane, Propene, Iso-Butane, n-Butane, Total Inorganic Carbon)
- Volatile Fatty Acids (Lactic, Pyruvic, Formic, Acetic Propionic, Pentanoic, Hexanoic and Butyric Acids)
- Ion Chromatography Analyses of chloride, nitrate, nitrite, sulfate, ferric iron, ferrous iron and divalent manganese
- Compound Specific Isotope Analysis (CSIA) of VOCs in groundwater and vapor

Soil Vapor Extraction Analyses

- VOC's in vapor
- Oxygen, Nitrogen, Carbon Dioxide, Carbon Monoxide, Hydrogen, Acetylene, Methane, Ethane, Ethene, Propane, Propene, Iso-Butane, and n-Butane in vapor

Shale Gas Analyses

- Compositional Analysis (nitrogen, oxygen, argon, carbon dioxide, C1-C6)

- Carbon isotopic ratio ($^{13}C/^{12}C$ or $\delta^{13}C$) of methane, ethane
- Hydrogen isotopic ratio ($^{2}H/^{1}H$ or $\delta^{2}H$) of methane

Petroleum Forensics

- C3-C12 Gasoline Range Hydrocarbons
- Oxygenates in product
- GC/MS Full Scan analyses
- Whole oil analyses
- Boiling Range Distribution of Petroleum Fractions
- EDB and Organic Lead

1.2. Statement of Purpose

1.2.1. To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. Pace management is committed to maintaining the highest possible standard of service and quality for our customers by following a documented quality system that is compliant with all current applicable state, federal, and industry standards, such as the NELAC Standard, the TNI Standard, and ISO standards and is in accordance with the stated methods and customer requirements. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the Pace network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work.

1.4. Core Values

1.4.1. The following are the Pace Core Values:

- **Integrity**
- **Value Employees**
- **Know Our Customers**
- **Honor Commitments**
- **Flexible Response To Demand**
- **Pursue Opportunities**
- **Continuously Improve**

1.5. Code of Ethics and Standards of Conduct

1.5.1. **Code of Ethics**:

1.5.1.1. Each Pace employee is responsible for the propriety and consequences of his or her actions;

1.5.1.2. Each Pace employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where Pace does business or seeks to do business;

1.5.1.3. Each Pace employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

1.5.1.4. Each Pace employee must recognize and understand that our daily activities in environmental laboratories affect public health as well as the environment and that environmental laboratory analysts are a critical part of the system society depends upon to improve and guard our natural resources:

1.5.2. **Standards of Conduct:**

1.5.2.1. **Data Integrity**

1.5.2.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at Pace are the cornerstones of the company. Employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

1.5.2.1.2. Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work including commercial, financial, over-scheduling, and working condition pressures.

1.5.2.1.3. The data integrity system includes in-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.5.2.1.4. Any documentation related to data integrity issues, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.5.2.2. **Confidentiality**

1.5.2.2.1. Pace employees must not use or disclose confidential or proprietary information except when in connection with their duties at Pace. This is effective over the course of employment and for an additional period of two years thereafter.

1.5.2.2.2. Confidential or proprietary information, belonging to either Pace and/or its customers, includes but is not limited to test results, trade secrets, research and development matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and customer information, inventions, materials composition, etc.

1.5.2.3. **Conflict of Interest**

1.5.2.3.1. Pace employees must avoid situations that might involve a conflict of interest or could appear questionable to others. This includes participation in activities that conflict or appear to conflict with the employees' Pace responsibilities. This would also include

offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally (such as bribes, gifts, kickbacks, or illegal payments).

1.5.2.3.2. Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other problematic activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company, or participation in any outside business during the employee's work hours.

1.5.3. Strict adherence by each Pace employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of Pace and to continue the pursuit of our common mission to protect our environment and improve our health.

1.5.4. Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

1.5.5. Compliance: all employees undergo annual Data Integrity/Ethics training which includes the concepts listed above. All employees also sign an annual Ethic Policy statement.

1.6. Anonymous Compliance Alertline

1.6.1. An ethical and safe workplace is important to the long-term success of Pace and the well-being of its employees. Pace has a responsibility to provide a work environmental where employees feel safe and can report unethical or improper behavior in complete confidence. With this in mind, Pace has engaged Lighthouse Services, Inc. to provide all employees with access to an anonymous ethics and compliance alertline for reporting possible ethics and compliance violations. The purpose of this service is to ensure that any employee can report anonymously and without fear of retaliation.

1.6.2. Lighthouse Services provides a toll-free number along with several other reporting methods, all of which are available 24 hours a day, seven days a week for use by employees and staff.

1.6.3. Telephone: English speaking USA and Canada: (844)-970-0003.

1.6.4. Telephone: Spanish speaking North America: (800)-216-1288.

1.6.5. Website: www.lighthouse-services.com/pacelabs.

1.6.6. Email: reports@lighthouse-services.com (must include company name with report).

1.7. Laboratory Organization

1.7.1. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office. See Attachment III for the Corporate Organizational structure.

1.7.2. A Senior General Manager (SGM) oversees all laboratories and service centers in their assigned region. Each laboratory or facility in the company is then directly managed by an SGM, a General Manager (GM), an Assistant General Manager (AGM), or an Operations Manager (OM). Quality Managers (QM) or Senior Quality Managers (SQM) at each laboratory report directly to the highest

level of local laboratory management, however named, that routinely makes day-to-day decisions regarding that facility's operations. The QMs and SQMs will also receive guidance and direction from the corporate Director of Environmental Quality.

1.7.3. The SGM, GM, AGM or OM, or equivalent functionality in each facility, bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of these managers, the SQM/QM serves as the next in command, unless the manager in charge has assigned another designee. He or she assumes the responsibilities of the manager, however named, until the manager is available to resume the duties of their position. In the absence of both the manager and the SQM/QM, management responsibility of the laboratory is passed to the Technical Director, provided such a position is identified, and then to the most senior department manager until the return of the lab manager or SQM/QM. The most senior department manager in charge may include the Client Services Manager (CSM) or the Administrative Business Manager (ABM) at the discretion of the SGM/GM/AGM/OM.

1.7.4. A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The laboratory SGM/GM/AGM/OM or SQM/QM has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 35 consecutive calendar days, the primary accrediting authority shall be notified in writing.

1.7.5. The SQM/QM has the responsibility and authority to ensure the Quality System is implemented and followed at all times. In circumstances where a laboratory is not meeting the established level of quality or following the policies set forth in this Quality Assurance Manual, the SQM/QM has the authority to halt laboratory operations should he or she deem such an action necessary. The SQM/QM will immediately communicate the halting of operations to the SGM/GM/AGM/OM and keep them posted on the progress of corrective actions. In the event the SGM/GM/AGM/OM and the SQM/QM are not in agreement as to the need for the suspension, the Chief Operating Officer (COO) and Director of Environmental Quality will be called in to mediate the situation.

1.7.6. The technical staff of the laboratory is generally organized into the following functional groups:

- Monitored Natural Attenuation (MNA)
- Compound Specific Isotopes (CSIA)
- Petroleum Forensics

1.7.7. The organizational structure for Pace Analytical Energy Services, LLC is listed in Attachment II. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its accrediting authorities per their individual required timeframes, not to exceed 30 days. The QAM will remain in effect until the next scheduled revision.

1.8. Laboratory Job Descriptions

1.8.1. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;

- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Presents the Ethics/Data Integrity training annually to all employees in their facilities as an instructor-led training.
- Ensures compliance with all applicable state, federal and industry standards.

1.8.2. Assistant General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Presents the Ethics/Data Integrity training annually to all employees in their facilities as an instructor-led training.
- Ensures compliance with all applicable state, federal and industry standards.
- Oversees the daily production and quality activities of all departments;
- Manages all departments and works with staff to ensure department objectives are met;
- Works with all departments to ensure capacity and customer expectations are accurately understood and met;
- Works with SGM to prepare appropriate budget and staffing plans for all departments;
- Responsible for prioritizing personnel and production activities within all departments;
- Performs formal and informal performance reviews of departmental staff.

1.8.3. Quality Manager

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Environmental Quality.
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors QA/QC activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Environmental Quality office). The QM is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Environmental Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews and maintains records of proficiency testing results;
- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;

- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors corrective and preventive actions;
- Maintains the currency of the Quality Manual.

1.8.4. Technical Director

- Monitors the validity of analyses performed and data generated as needed;
- Provides technical guidance in the review, development, and validation of new methodologies.
- Assists clients evaluate their analytical needs.
- Prepares and presents written or oral interpretations of data.

1.8.5. Administrative Business Manager

- Responsible for financial and administrative management for the entire facility;
- Provides input relative to tactical and strategic planning activities;
- Organizes financial information so that the facility is run as a fiscally responsible business;
- Works with staff to confirm that appropriate processes are put in place to track revenues and expenses;
- Provide ongoing financial information to the SGM/GM/AGM/OM and the management team so they can better manage their business;
- Utilizes historical information and trends to accurately forecast future financial positions;
- Works with management to ensure that key measurements are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios;
- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.
- Ensures that vendor invoices are properly coded and posted for payment
- Ensures that invoices are properly coded and posted for revenue

1.8.6. Client Services Manager

- Oversees all the day to day activities of the Client Services Department which includes Project Management and, possibly, Sample Control;
- Responsible for staffing and all personnel management related issues for Client Services;
- Serves as the primary senior consultant to customers on all project related issues such as set up, initiation, execution and closure;
- Performs or is capable of performing all duties listed for that of Project Manager.

1.8.7. Project Manager

- Coordinates daily activities including taking orders, reporting data and analytical results;
- Serves as the primary technical and administrative liaison between customers and Pace;

- Communicates with operations staff to update and set project priorities;
- Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.);
- Works with customers, laboratory staff, and other appropriate Pace staff to develop project statements of work or resolve problems of data quality;
- Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers and maintain customer records;
- Mediation of project schedules and scope of work through communication with internal resources and management;
- Responsible for preparing routine and non-routine quotations, reports and technical papers;
- Interfaces between customers and management personnel to achieve customer satisfaction;
- Manages large-scale complex projects;
- Supervises less experienced project managers and provide guidance on management of complex projects;
- Arranges bottle orders and shipment of sample kits to customers;
- Verifies login information relative to project requirements and field sample Chains-of-Custody.

1.8.8. CSIA Department Manager/Supervisor

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Ensures compliance with all applicable state, federal and industry standards;
- Works with SGM/GM to prepare appropriate budget and staffing plans;
- Responsible for prioritizing personnel and production activities;
- Performs formal and informal performance reviews of departmental staff.
- Works with all departments to ensure capacity and customer expectations are accurately understood and met.

1.8.9. IC Department Manager/Supervisor

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Ensures compliance with all applicable state, federal and industry standards;
- Works with SGM/GM to prepare appropriate budget and staffing plans;
- Responsible for prioritizing personnel and production activities ;
- Performs formal and informal performance reviews of departmental staff;
- Works with all departments to ensure capacity and customer expectations are accurately understood and met.

1.8.10. MNA Department Manager/Supervisor

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Works with SGM/GM to prepare appropriate budget and staffing plans;
- Responsible for prioritizing personnel and production activities ;
- Performs formal and informal performance reviews of departmental staff;
- Works with the department staff to ensure capacity and customer expectations are accurately understood and met;
- Works with staff to ensure department objectives are met;
- Review/approve all department generated data/results from staff with LIMS; apply results and/or quality assigned qualifiers manually, as needed; apply narrative information, as needed;
- Apply narrative information related to sample receipt; review final reports, as needed;
- Review and/or establish SOP's; adjustment to bench related QA protocols; data management.

1.8.11. Additional job descriptions are available upon request from the laboratory ABM.

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through a web-based training system. Employees are provided with several training activities for their particular job description and scope of duties. These training activities may include:

- Hands-on training led by supervisors;
- Job-specific training checklists and worksheets;
- Lectures and instructor-led training sessions;
- Method-specific training;
- External conferences and seminars;
- Reading Standard Operating Procedures (SOPs);
- Reading the Quality Assurance Manual and Safety Manual/Chemical Hygiene Plan;
- Core training modules (basic lab skills, etc.);
- Quality system training modules (support equipment use, corrective actions/root causes, etc.);
- Data Integrity/Ethics training;
- Specialized training by instrument manufacturers;
- On-line courses.

1.9.2. All procedures and training records are maintained and available for review during laboratory audits. Additional information can be found in SOP S-PAE-Q-015 **Administering and Documenting Training in Laboratory Procedures and Instrumentation** or its equivalent revision or replacement.

1.10. Laboratory Safety and Waste

1.10.1. It is the policy of Pace to make safety and waste compliance an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety as well as those working in the immediate area by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the local Safety Manual/Chemical Hygiene Plan.

1.11. Security and Confidentiality

1.11.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by Pace staff.

1.11.2. Employee access into the UPARC complex is controlled through key-card turn-styles where each individual that works in the complex has a unique code for entry. UPARC Security is aware of who is on-site or off-site at any given time. PAES laboratory areas are controlled through keyed entry to prevent employees from other firms housed in the complex from gaining access to PAES laboratories. Each employee is issued a key that will open doors to rooms occupied by PAES. During normal working hours, the laboratory areas are kept unlocked. After normal business hours the rooms are locked to prevent unauthorized personnel entry.

1.11.3. A UPARC security force monitors the facility twenty-four hours a day with a series of video cameras. The guards also make rounds by foot and vehicle during afternoon and night shifts. Visitors cannot gain access to the complex except through the Main Security Gate. All visitors are required to register at the main gate and obtain a visitor's pass before entering the complex. UPARC Security notifies PAES upon the visitor's arrival to verify admittance. Visitors are directed to PAES Reception Office to sign the visitor's log. The visitor is then escorted, by a PAES employee to the employee or laboratory they intend to visit.

All information pertaining to a particular customer, including national security concerns will remain confidential. Data will be released to outside agencies only with written authorization from the customer or where federal or state law requires the company to do so.

1.12. Communications

1.12.1. Management within each lab bears the responsibility of ensuring that appropriate communication processes are established and that communication takes place regarding the effectiveness of the management/quality system. These communication processes may include email, regular staff meetings, senior management meetings, etc.

1.12.2. Corporate management bears the responsibility of ensuring that appropriate communication processes are established within the network of facilities and that communication takes place at a company-wide level regarding the effectiveness of the management/quality systems of all Pace facilities. These communication processes may include email, quarterly continuous improvement conference calls for all lab departments, and annual continuous improvement meetings for all department supervisors, quality managers, client services managers, and other support positions.

2.0. SAMPLE CUSTODY

2.1. Project Initiation

2.1.1. Prior to accepting new work, the laboratory reviews its performance capability. The laboratory confirms that sufficient personnel, equipment capacity, analytical method capability, etc., are available to complete the required work. Customer needs, certification requirements, and data quality objectives are defined and the appropriate sampling and analysis plan is developed to meet the project requirements by project managers or sales representatives. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability, and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or operations meetings.

2.1.2. All contract documents are forwarded to Corporate Counsel for review. Any exceptions with the contract language noted by the Counsel will be forwarded to the client for their approval. Once mutually acceptable agreement is reached, the General Manager or their designee with approval, will sign the contract and /or purchase order. In the event that the contract needs amended after work has commenced, the same contract review procedures shall be repeated and any changes shall be communicated to all affected personnel.

2.2. Sampling Materials and Support

2.2.1. Each individual Pace laboratory provides shipping containers, properly preserved sample containers, custody documents, and field quality control samples to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VII. Note that all analyses listed are not necessarily performed at all Pace laboratories and there may be additional laboratory analyses performed that are not included in these tables. Customers are encouraged to contact their local Pace Project Manager for questions or clarifications regarding sample handling. Pace may provide pick-up and delivery services to their customers when needed.

2.2.2. Some Pace facilities provide sampling support through a Field Services department. Field Services operates under the Pace Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in SOPs and Procedure Manuals.

2.3. Chain of Custody

2.3.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis.

2.3.2. Field personnel or client representatives must complete a COC for all samples that are received by the laboratory. Samplers are required to properly complete a COC. This is critical to efficient sample receipt and to ensure the requested methods are used to analyze the correct samples. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.3.3. The COC is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the initial entry and initialing and dating the change. All transfers of samples are recorded on the chain of custody in the "relinquished" and "received by" sections. All information except signatures is printed.

2.3.4. Additional information can be found in SOP S-PAE-C-003 **Sample Receiving** or its equivalent revision or replacement.

2.4. Sample Acceptance Policy

2.4.1. In accordance with regulatory guidelines, Pace complies with the following sample acceptance policy for all samples received.

2.4.2. If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory is required to document all non-compliances, contact the customer, and either reject the samples or fully document any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately communicated to the client.

2.4.3. Sample Acceptance Policy requirements:

- Sample containers must have unique client identification designations that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC).
- There must be clear documentation on the COC, or related documents, such as the Cooler Receipt form, that lists the unique sample identification, sampling site location, date and time of sample collection, and name of the sample collector.
- There must be clear documentation on the COC, or related documents that lists the requested analyses, the preservatives used, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.).
- Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval.
- Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval.
- Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval. Clients are requested to notify a PAES Project Manager if samples with short hold times are being shipped.
- Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval.
- All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2°C of the required temperature, or within the method-specified range. For samples with a required temperature of 4°C, samples with a temperature ranging from just above freezing to 6°C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice.

A cooler receipt form is generated and placed in the project file along with the chain of custody and other related documentation.

2.5.3. All documentation relating to the project is maintained in the project file and retained in the laboratory for five years following the date the project is completed.

2.5.4. Additional information can be found in SOP S-PAE-C-003 **Sample Receiving** or its equivalent revision or replacement.

2.6. Sample Storage

2.6.1. Additional information on sample storage can be found in SOP S-PAE-C-003 **Sample Receiving** or its equivalent revision or replacement and in SOP S-PAE-W-002 **Waste Handling and Management** or its equivalent revision or replacement.

2.6.2. Storage Conditions

2.6.2.1. Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

2.6.2.2. Additional information can be found in SOP S-PAE-Q-008 **Monitoring Temperature Controlled Units** or its equivalent revision or replacement.

2.6.3. Temperature Monitoring

2.6.3.1. Samples are taken to the appropriate storage location immediately after sample receipt and check-in procedures are completed.

2.6.3.2. The temperature of each refrigerated storage area is maintained at $\leq 6^{\circ}C$ (but above freezing) unless state, method or program requirements differ. The temperature of each freezer storage area is maintained at $\leq -10^{\circ}C$ unless state, method or program requirements differ. The temperature of each storage area is checked and documented each working day using Min/Max thermometers. Additional information, including corrective actions for temperatures outside of acceptance limits, can be found in SOP S-PAE-Q-008 **Monitoring Temperature Controlled Units** or its equivalent revision or replacement.

2.6.4. Hazardous Materials

2.6.4.1. Samples designated by clients upon receipt as pure product or potentially heavily contaminated samples, or samples found to be designated as such following analysis, will be handled according to SOP S-PAE-W-002 **Waste Handling and Management** or its equivalent revision or replacement.

2.6.5. Foreign/Quarantined Soils

2.6.5.1. Foreign soils and soils from USDA regulated areas must be adequately segregated to enable proper sample disposal. The USDA requires these samples to be treated by an approved procedure. Additional information regarding USDA regulations and sample handling can be found in the laboratory's SOP for **Regulated Soil Handling** S-PAE-S-002, or its equivalent revision or replacement.

Any samples that are not received at the required temperature will not be processed without prior client approval.

- Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.
- Samples that fail the radiation screen according to the criteria set forth in SOP S-PAE-C-003, **Sample Receiving,** or its equivalent revision or replacement, will not be accepted.
- Coolers that arrive with hazard labels on them for which PAES is not equipped or certified will not be accepted. A chart listing these hazards is posted in Sample Receiving.

When problems with samples or documentation are found during the sample receiving process, a Non-Conformance Form is completed by sample receiving personnel and forwarded to a Project Manager. The Project manager will make every attempt to contact the client as soon as possible to make decisions concerning the discrepancies. The Non-Conformance Form is kept as a permanent part of the project file.

2.4.4. Upon sample receipt, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers;
- Sample condition: Intact, broken/leaking, bubbles in VOA samples;
- Sample holding time;
- Sample pH
- Appropriate containers.

2.4.5. Additional information can be found in SOP S-PAE-C-003 **Sample Receiving** or its equivalent revision or replacement.

2.5. Sample Log-in

2.5.1. After sample inspection, all sample information on the COC is entered into the Laboratory Information Management System (LIMS). The lab's permanent records for samples received include the following information:

- Customer name and contact
- Customer number
- PAES Analytical project number
- PAES Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested
- Date and time of laboratory receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

2.5.2. Specific sample log in procedures are outlined in PAES Standard Operating Procedure for HORIZON LIMS. PAES LIMS assigns a unique internal project number and sequential sample numbers. These numbers are used to track the project through the laboratory. The sample numbers are transferred to each sample container using a computer-generated label. These numbers are documented on the chain of custody form and verified by the Sample Receiving Client Service Tech.

2.7. Subcontracting Analytical Services

2.7.1. Every effort is made to perform all analyses for Pace customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory, whether inside or outside the Pace network, becomes necessary, a preliminary verbal communication with that laboratory is undertaken. Customers are notified in writing of the laboratory's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations. When possible, subcontracting will be to a TNI-accredited laboratory.

2.7.2. Potential subcontract laboratories must be approved by Pace based on the criteria listed in SOP S-PAE-C-001 **Subcontracting** or its equivalent revision or replacement. All sample reports from the subcontracted labs are appended to the applicable Pace final reports.

2.7.3. Any Pace Analytical work sent to other labs within the Pace network is handled as inter-regional work and all final reports are labeled clearly with the name of the laboratory performing the work. Any non-TNI work is clearly identified. Pace will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

2.7.4. Additional information can be found in SOP S-PAE-C-001 **Subcontracting** or its equivalent revision or replacement.

2.8. Sample Retention and Disposal

2.8.1. Samples, extracts, digestates, and leachates must be retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

2.8.2. The minimum sample retention time is 30 days after final report generation. Samples requiring thermal preservation may be stored at ambient temperature when the hold time is expired, the report has been delivered, and/or allowed by the customer, program, or contract. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

2.8.3. After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of hazardous samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires Pace to dispose of excess samples, proper arrangements will be made for disposal by an approved contractor.

2.8.4. Additional information can be found in SOP S-PAE-W-002 **Waste Handling and Management** and SOP S-PAE-C-003 **Sample Receiving** or their equivalent revisions or replacements.

3.0 QUALITY CONTROL PROCEDURES

3.1. Quality Control Samples

3.1.1. The quality control samples described in this section are analyzed per batch as applicable to the method used. Acceptance criteria must be established for all quality control samples and if the acceptance criteria are not met, corrective actions must be performed and samples reanalyzed, or final reports must be appropriately qualified.

3.1.2. Quality control samples must be processed in the same manner as associated client samples.

3.1.3. Please reference the glossary of this Quality Manual for definitions of all quality control samples mentioned in this section.

3.1.4. Any deviations to the policies and procedures governing quality control samples must be approved by the QM/SQM.

3.2. Method Blank

3.2.1. A method blank is a negative control used to assess the preparation/analysis system for possible contamination and is processed through all preparation and analytical steps with its associated client samples. The method blank is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples. Method blanks are not applicable for certain analyses (i.e., pH, flash point, temperature, etc.).

3.2.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for method blanks.

3.3. Laboratory Control Sample

3.3.1. The Laboratory Control Sample (LCS) is a positive control used to assess the performance of the entire analytical system including preparation and analysis. The LCS is processed at a minimum frequency of one per preparation batch and is comprised of a matrix similar to the associated client samples.

3.3.2. The LCS contains **all** analytes required by a specific method or by the customer or regulatory agency, which may include full list of target compounds, with certain exceptions. The lab must ensure that all target components are included in the spike mixture for the LCS over a two (2) year period. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
    - For methods with 1-10 target compounds, the laboratory will spike with all compounds;
    - For methods with 11-20 target compounds, the laboratory will spike with at least 10 compounds or 80%, whichever is greater;

- For methods with greater than 20 compounds, the laboratory will spike with at least 16 compounds.

3.3.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for LCSs.

3.3.4. For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary (except for proper documentation). TNI has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but within than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

- >90 analytes in the LCS- 5 analytes
- 71-90 analytes in the LCS- 4 analytes
- 51-70 analytes in the LCS- 3 analytes
- 31-50 analytes in the LCS- 2 analytes
- 11-30 analytes in the LCS- 1 analyte
- <11 analytes in the LCS- no analytes allowed out)

Note: the use of marginal exceedances is not approved for work from the state of South Carolina.

3.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.4.1. A matrix spike (MS) is a positive control used to determine the effect of the sample matrix on compound recovery for a particular method. A matrix spike/matrix spike duplicate (MS/MSD) set or matrix spike/sample duplicate set is processed at a frequency specified in a particular method or as determined by a specific customer request. The MS and MSD consist of the sample matrix that is spiked with known concentrations of target analytes.

3.4.2. The MS and MSD contain all analytes required by a specific method or by the customer or regulatory agency. In the absence of specified components, the laboratory will spike the MS/MSD with the same number of compounds as previously discussed in the LCS section. However, the lab must ensure that all targeted components are included in the spike mixture for the MS/MSD over a two (2) year period.

3.4.3. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for MS/MSDs.

3.5. Sample Duplicate

3.5.1. A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

3.5.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for sample duplicates.

3.6. Surrogates

3.6.1. Surrogates are compounds that reflect the chemistry of target analytes and are typically added to samples for organic analyses to measure the extraction or purge efficiency and to monitor the effect of the sample matrix on compound recovery.

3.6.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for surrogates.

3.7. Internal Standards

3.7.1. Internal Standards are method-specific analytes that are added, as applicable, to every standard, QC sample, and client sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes.

3.7.2. Please reference method-specific SOPs for acceptance criteria and associated corrective actions for internal standards.

3.8. Limit of Detection (LOD)

3.8.1. Pace laboratories use a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. Unless otherwise noted in a published method, the procedure used by Pace laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B, the TNI Standard and the MUR. All sample processing steps of the preparation and analytical methods are included in the LOD determination including any clean ups.

3.8.2. Additional information can be found in SOP S-PAE-Q-010 **Determination of Detection Limits and Reporting Limits** or its equivalent revision or replacement.

3.9. Limit of Quantitation (LOQ)

3.9.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For Pace laboratories, this LOQ is referred to as the RL, or Reporting Limit. Results reported below the reporting limit are not allowed to be reported without qualification. For methods with a determined LOD, results can be reported out below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

3.9.2. Additional information can be found in SOP S-PAE-Q-010 **Determination of Detection Limits and Reporting Limits** or its equivalent revision or replacement.

3.10. Estimate of Analytical Uncertainty

3.10.1. Pace laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, Pace laboratories base this estimation on the recovery data obtained from the Laboratory Control Samples. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence.

3.10.2. The measurement of uncertainty is provided only on request by the customer, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

3.11. Proficiency Testing (PT) Studies

3.11.1. Pace laboratories participate in a defined proficiency testing (PT) program. PT samples are obtained from NIST approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix.

3.12. Rounding and Significant Figures

3.12.1. In general, the Pace laboratories report data to no more than three significant figures. Therefore, all measurements made in the analytical process must reflect this level of precision. In the event that a parameter that contributes to the final result has less than three significant figures of precision, the final result must be reported with no more significant figures than that of the parameter in question. The rounding rules listed below are descriptive of the LIMS and not necessarily of any supporting program such as Excel.

3.12.2. **Rounding:** Pace-PAES follows the odd / even guidelines for rounding numbers:

- If the figure following the one to be retained is less than five, that figure is dropped and the retained ones are not changed (with three significant figures, 2.544 is rounded to 2.54).
- If the figure following the ones to be retained is greater than five, that figure is dropped and the last retained one is rounded up (with three significant figures, 2.546 is rounded to 2.55).
- If the figure following the ones to be retained is five and if there are no figures other than zeros beyond that five, then the five is dropped and the last figure retained is unchanged if it is even and rounded up if it is odd (with three significant figures, 2.525 is rounded to 2.52 and 2.535 is rounded to 2.54).

3.12.3. **Significant Figures**

- Unless specified by federal, state, or local requirements or on specific request by a customer, PAES reports all analytical results to at least 2 significant figures, regardless of the magnitude of the value reported.

3.13. Retention Time Windows

3.13.1. When chromatographic conditions are changed, retention times and analytical separations are often affected. As a result, two critical aspects of any chromatographic method are the determination and verification of retention times and analyte separation. Retention time windows must be established for the identification of target analytes. The retention times of all target analytes in all calibration verification standards must fall within the retention time windows. If an analyte falls outside the retention time window in an ICV or CCV, new absolute retention time windows must be calculated, unless instrument maintenance fixes the problem. When a new column is installed, a new retention time window study must be performed.

3.13.2. Please reference method-specific SOPs for the proper procedure for establishing retention time windows.

3.14. Analytical Method Validation and Instrument Validation

3.14.1. In some situations, Pace develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods are required for specific projects or analytes of interest, or when the laboratory develops or modifies a method, the laboratory validates the method prior to applying it to customer samples. Method validity is established by meeting criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. The laboratory records the validation procedure, the results obtained and a statement as to the usability of the method. The minimum requirements for method validation include evaluation of sensitivity, quantitation, precision, bias, and selectivity of each analyte of interest.

3.15. Regulatory and Method Compliance

3.15.1. It is Pace policy to disclose in a forthright manner any detected noncompliance affecting the usability of data produced by our laboratories. The laboratory will notify customers within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

4.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

4.1. Document Management

4.1.1. Additional information can be found in SOP S-PAE-Q-002 **Document Control and Management** or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

4.1.2. Pace has an established procedure for managing documents that are part of the quality system.

4.1.3. A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents.

4.1.4. Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to SOP S-ALL-Q-003 **Document Numbering** or its equivalent revision or replacement

4.1.5. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for Pace. The base QAM template is distributed by the Corporate Environmental Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template and then all applicable staff sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis and revised accordingly by the Corporate Quality office.

4.1.6. **Standard Operating Procedures (SOPs)**

4.1.6.1. SOPs are reviewed every two years at a minimum although a more frequent review may be required by some state or federal agencies or customers. If no revisions are made based on this review, documentation of the review itself is made by the addition of new signatures on the cover page. If revisions are made, documentation of the revisions is made in the revisions section of each SOP and a new revision number is applied to the SOP. This provides a historical record of all revisions.

4.1.6.2. All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all Pace employees use the most current version of each SOP and provides the SQM/QM with a historical record of each SOP.

4.1.6.3. Additional information can be found in SOP S-PAE-Q-001 **Preparation of SOPs** or its equivalent revision or replacement.

4.2. Document Change Control

4.2.1. Additional information can be found in SOP S-PAE-Q-002 **Document Control and Management** or its equivalent revision or replacement.

4.2.2. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After

revisions are approved, a revision number is assigned and the previous version of the document is officially retired.

4.2.3. All copies of the previous document are replaced with copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised that there has been a revision and any necessary training is scheduled.

5.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

5.1. Standards and Traceability

5.1.1. Each Pace facility retains pertinent information for standards, reagents, and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation, and use.

5.1.2. Upon receipt, all purchased standard reference materials are recorded into a standard logbook or database and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date, and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

5.1.3. Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, the preparation steps, preparation date, expiration dates, preparer's initials, the concentration values and units, and a unique Pace identification number. This number is used in any applicable sample preparation or analysis logbook so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

5.1.4. All prepared standard or reagent containers include the Pace identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials, unless the container is too small to hold all of this information. This ensures traceability back to the standard preparation logbook or database.

5.1.5. All initial calibrations must be verified with a standard obtained from a second manufacturer or a separate lot prepared independently by the same manufacturer, unless client-specific QAPP requirements state otherwise.

5.1.6. Additional information concerning the procurement of standards and reagent and their traceability can be found in the SOP S-PAE-Q-011**Standard and Reagent Management and Traceability** or its equivalent revision or replacement.

5.2. General Analytical Instrument Calibration Procedures

5.2.1. All applicable instrumentation are calibrated or checked before use to ensure proper functioning and verify that laboratory, client and regulatory requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated.

5.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in the narrative.

5.2.3. Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets the laboratory's specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported to prevent breakage and is in accordance with the calibration laboratory's recommendations.

5.2.4. In the event that recalibration of a piece of test equipment indicates the equipment may have been malfunctioning during the course of sample analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the quality manager. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or taken out of service and replaced.

5.2.5. Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

5.3. Support Equipment Calibration and Verification Procedures

5.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use, as applicable. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until brought back into control. Additional information regarding calibration and maintenance of support equipment can be found in SOP S-PAE-Q-009 **Support Equipment** or its equivalent revision or replacement.

5.3.2. On each day the support equipment is used, it is verified, as applicable, in the expected range of use with NIST traceable references in order to ensure the equipment meets laboratory specifications. These checks are documented appropriately. This applies mainly to thermometers within temperature-controlled units and balances.

5.3.3. **Analytical Balances**

5.3.3.1. Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Calibration weights are ASTM Class 1 or other class weights that have been calibrated against a NIST standard weight and are re-certified annually at a minimum against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the local Quality department.

5.3.4. **Thermometers**

5.3.4.1. Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are re-certified, annually with equipment directly traceable to NIST.

5.3.4.2. Working thermometers are compared with the reference thermometers annually. Each thermometer is individually numbered and assigned a correction factor based on the NIST reference

source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

5.3.4.3. Laboratory thermometer inventory and calibration data are maintained in the local Quality department.

5.3.5. **pH/Electrometers**

5.3.5.1. The meter is calibrated before use each day, using fresh buffer solutions.

5.3.6. **Mechanical Volumetric Dispensing Devices**

5.3.6.1. Mechanical volumetric dispensing devices including bottle top dispensers (those that are critical in measuring a required amount of reagent), pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis.

5.3.6.2. Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-PAE-Q-009 **Support Equipment** or its equivalent revision or replacement.

5.4. Instrument/Equipment Maintenance

5.4.1. The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs.

5.4.2. The Operations Manager and/or department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

5.4.3. To minimize downtime and interruption of analytical work, preventative maintenance may routinely be performed on each analytical instrument. Up-to-date instructions on the use and maintenance of equipment are available to staff in the department where the equipment is used.

5.4.4. Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

5.4.5. All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation is, unless otherwise stated, identified as a system and not as individual pieces. The laboratory maintains equipment records that include the following:

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service
- Current location in the laboratory
- Condition when received (new, used, etc.)
- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

5.4.6. All instrument maintenance is documented in maintenance logbooks that are assigned to each particular instrument or system.

5.4.7. The maintenance log entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

5.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily. In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab if possible.

6.0. CONTROL OF DATA

Analytical results processing, verification, and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user.

All analytical data undergo a documented multi-tier review process prior to being reported to the customer. This section describes procedures used for translating raw analytical data into accurate final sample reports as well as Pace data storage policies.

When analytical, field, or product testing data is generated, it is documented appropriately. These logbooks and other laboratory records are kept in accordance with each facility's SOP for documentation storage and archival In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues

6.1. Primary Data Review

6.1.1. The primary analyst is responsible for initial data reduction and data review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets, LIMS fields, and data review checklists.

6.1.2. The primary analyst compiles the initial data for secondary data review. This compilation must include sufficient documentation for secondary data review.

6.1.3. Additional information regarding data review procedures can be found in SOP S-PAE-Q-004 **Data Integrity, Review and Validation** or its equivalent revision or replacement, as well as in SOP S-PAE-Q-003 **Manual Integration** or its equivalent revision or replacement.

6.2. Secondary Data Review

6.2.1. Secondary data review is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated, and that any non-conformances are properly documented.

6.2.2. The completed data from the primary analyst is sent to a designated qualified secondary data reviewer (this cannot be the primary analyst). The secondary data reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer validates the data entered into the LIMS and documents approval of manual integrations.

6.2.2. Additional information regarding data review procedures can be found in SOP S-PAE-Q-004 **Data Integrity, Review and Validation** or its equivalent revision or replacement, as well as in SOP S-PAE-Q- 003 **Manual Integration** or its equivalent revision or replacement.

6.2.3. The Laboratory Manager attempts to review approximately 10% of all laboratory data. Either the Quality Systems Manager or his designee will review 10% of all DoD data packages. This review is part of the oversight program and does not have to be completed in "real time." Project Managers complete the data validation process by reviewing final reports for completeness prior to submission to the client.

6.3. Data Reporting

6.3.1. Data for each analytical fraction pertaining to a particular Pace project number are delivered to the Project Manager for assembly into the final report. All points mentioned during technical and QC reviews are included in data qualifiers on the final report or in a separate case narrative if there is potential for data to be impacted.

6.3.2. Final reports are prepared according to the level of reporting required by the customer and can be transmitted to the customer via hardcopy or electronic deliverable.

6.3.3. Any changes made to a final report shall be designated as "Revised" or equivalent wording. The laboratory must keep sufficient archived records of all laboratory reports and revisions. For higher levels of data deliverables, a copy of all supporting raw data is sent to the customer along with a final report of results. Pace will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

6.3.4. Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

6.3.5. The following positions are the only approved signatories for Pace final reports:

- Senior General Manager
- General Manager
- Assistant General Manager
- Senior Quality Manager
- Quality Manager
- Client Services Manager
- Project Manager
- Project Coordinator

6.4. Data Security

6.4.1. All data including electronic files, logbooks, extraction/digestion/distillation worksheets, calculations, project files and reports, and any other information used to produce the technical report are maintained secured and retrievable by the Pace facility.

6.5. Data Archiving

6.5.1. All records compiled by Pace are archived in a suitable, limited-access environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. TNI-related records will be made readily available to

accrediting authorities. Access to archived data is documented and controlled by the SQM/QM or a designated Data Archivist.

6.5.2. Records that are computer-generated have either a hard copy or electronic backup copy. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

6.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained per the purchase agreement. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

6.6. Data Disposal

6.6.1. Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. Data disposal includes any preliminary or final reports that are disposed.

7.0. QUALITY SYSTEM AUDITS AND REVIEWS

7.1. Internal Audits

7.1.1. Responsibilities

7.1.1.1. The SQM/QM is responsible for managing and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The SQM/QM evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the effectiveness of the Quality System as outlined in this manual but may also include other quality programs applicable to an individual laboratory.

7.1.1.2. Additional information can be found in SOP S-PAE-Q-007 **Internal Audits** or its equivalent revision or replacement.

7.1.2. Scope and Frequency of Internal Audits

7.1.2.1. The complete internal audit process consists of the following four sections: 1) Raw Data Reviews, 2) traditional Quality Systems internal audits (including SOP and method compliance), 3) Final Report Reviews, and 4) Corrective Action Effectiveness Follow-up.

7.1.2.2. Internal systems audits are conducted yearly at a minimum. The scope of these audits includes evaluation of specific analytical departments or a specific quality related system as applied throughout the laboratory.

7.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible.

7.1.2.4. Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

7.1.2.5. The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the laboratory, and which final reports had to be re-issued. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed.

7.1.2.6. Internal Audits are scheduled and conducted by the Quality Systems Department or their designees.

7.1.3. Internal Audit Reports and Corrective Action Plans

7.1.3.1. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations

noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the SQM/QM writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

7.1.3.2. Corrective Action based upon the root cause analysis is indicated on a Corrective Action Report (see Figure 2-3). A Corrective Action Report is prepared for each deficiency listed on the Internal Audit Report. The Internal Audit Report and Corrective Action Report are forwarded to the appropriate Department Head for corrective action. A follow-up audit is conducted and documented.

7.1.3.3. If the audit was of a technical nature, the Audit Report will be forwarded to the Laboratory Manager. The Laboratory Manager will meet with the specific Department Manager the first business day once the audit findings are received. The audit will be discussed along with the recommendations for corrective action. Corrective action is expected to take place as soon as possible following the audit. A follow-up audit of any deficient area(s) will be conducted within 60-120 days of audit completion, or as soon as corrective action is completed in order to monitor the effectiveness of corrective action. This timeframe is just a guide.

7.1.3.4. When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's environmental test results, the laboratory will take timely corrective action as specified in PAES' Quality Policy Statement. If the subsequent investigation shows that laboratory results have been affected, the affected client shall be notified in writing by the Client Service Office.

7.1.3.5. Additional information can be found in SOP S-PAE-Q-007 **Internal Audits** or its equivalent revision or replacement.

7.2. External Audits

7.2.1. Pace laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

7.2.2. External audit teams review the laboratory to assess the effectiveness of quality systems. The SQM/QM host the external audit team and assist in facilitation of the audit process. After the audit, the external auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM, who provides a written response to the external audit team. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

7.3. Annual Managerial Review

7.3.1. A managerial review of Management and Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements. Additional information can be found in SOP S-ALL-Q-015 **Review of Laboratory Management System** or its equivalent revision or replacement.

7.3.2. The managerial review must include the following topics of discussion:

- Suitability of quality management policies and procedures

- Manager/Supervisor reports
- Internal audit results
- Corrective and preventive actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources, and staffing.

7.3.3. This managerial review must be documented for future reference by the SQM/QM and copies of the report are distributed to laboratory staff. Results must feed into the laboratory planning system and must include goals, objectives, and action plans for the coming year. The laboratory shall ensure that any actions identified during the review are carried out within an appropriate and agreed upon timescale.

8.0. CORRECTIVE ACTION

Additional information can be found in SOP SOP-PAE-Q-005 **Corrective Action Reports** or its equivalent revision or replacement.

During the process of sample handling, preparation, and analysis, or during review of quality control records, or during reviews of non-technical portions of the lab, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of Pace provides systematic procedures for the documentation, monitoring, completion of corrective actions, and follow-up verification of the effectiveness of these corrective actions. This can be done using procedures found in SOP-PAE-Q-005 that lists at a minimum, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

8.1. Corrective and Preventive Action Documentation

8.1.1. The person who discovers the deficiency or non-conformance initiates the corrective action documentation within the lab's corrective action system. The documentation must include (as applicable): the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

8.1.2. **Root Cause Analysis**: Laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if the condition is significant, and investigating the root cause. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within the lab's corrective action system.

8.1.3. Based on the root cause(s) determined, the lab implements applicable corrective actions and verifies their effectiveness. In the event that analytical testing or results do not conform to documented laboratory policies or procedures Project Management will notify the customer of the situation and will advise of any ramifications to data quality if impacted (with the possibility of work being recalled).

8.1.4. **Preventive Actions**: The preferred course of laboratory quality and improvement is to identify opportunities for improvement rather than react to the occurrence of problems or complaints. PAES is continually seeking ways to improve its performance and product. When these areas are identified, a plan is developed by the department managers. Preventative actions are implemented according to the time table specified in the plan. Preventive action procedures include follow-up actions and applications of controls in order to ensure effectiveness. The laboratory seeks both negative and positive feedback from its customers. Feedback provides acknowledgement, corrective actions when needed, and opportunities for improvement. A statement printed on the front page of all final reports gives an avenue for customers to provide comments to us on our performance. Random surveys may also be used as a means to gather feedback from customers. This information is forwarded to the Quality Systems Department.

8.1.5. **Management Arrangements for Permitting Departures from Documented Procedures or Standard Specifications**: It is PAES management's intent to ensure that documented procedures are followed. Rarely, a situation may occur that requires a departure from documented quality procedures. When this type of situation occurs, the AGM/QM and department managers whose department may be affected, will discuss and unanimously agree upon the action to be taken. The departure will be documented in memo form and kept on file in the Quality office. Corrective action will be taken as soon as possible to prevent the necessity of the departure from reoccurring.

8.2. Corrective Action Completion

8.2.1. Internal Laboratory Non-Conformance Trends

8.2.1.1. There are several types of non-conformance trends that may occur in the laboratory that would require the initiation of a corrective action report. Laboratories may choose to initiate a corrective action for all instances of one or more of these categories if they so choose, however the intent is that each of these would be handled according to its severity; one time instances could be handled with a footnote or qualifier whereas a systemic problem with any of these categories may require an official corrective action process. These categories, as defined in the Corrective Action SOP are as follows:

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting error

8.2.2. PE/PT Sample Results

8.2.2.1. Any PT result assessed as "not acceptable" requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

8.2.3. Internal and External Audits

8.2.3.1. The SQM/QM is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for responding to the auditing body, the root cause of the finding, and the corrective actions needed for resolution. The SQM/QM is also responsible for providing any back-up documentation used to demonstrate that a corrective action has been completed.

8.2.4. Data Review

8.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data reviews (e.g., by the SQM/QM), errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

8.2.5. Client Complaints

8.2.5.1. Project Managers are responsible for issuing corrective action forms, when warranted, for client complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor for investigation. After potential corrective actions have been determined, the Project Manager reviews the corrective action form to ensure all client needs or concerns are being adequately addressed.

8.2.6. Holding Time Violations

8.2.6.1. The Project Manager and the SQM/QM must be made aware of all holding time violations.

8.2.6.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the hold time excursion and the ultimate resolution is then documented and included in the customer project file.

9.0. GLOSSARY

The source of some of the definitions is indicated previous to the actual definition (e.g., TNI, DoD).

Terms and Definitions	
3P Program	The Pace continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all Pace labs.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies*. The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.

American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.
Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by Pace as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch	TNI- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100

Confirmation	TNI- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)

Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence $(1 - \alpha)$ that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.

Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.

Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).

Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.
In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.

Internal Standard	TNI and DoD- A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
LabTrack	Database used by Pace to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.

Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.

Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, 1 – β, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
MintMiner	Program used by Pace to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).

Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.

Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.

Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a chain-of-custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.

Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.

Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where $k > 1$).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

10.0. REFERENCES

10.1. "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, most current version.

10.2. "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.

10.3. "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.

10.4. U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis.

10.5. U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis.

10.6. "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.

10.7. "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.

10.8. "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.

10.9. "NIOSH Manual of Analytical Methods", U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health, most current version.

10.10. "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (Sep 1986).

10.11. Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.

10.12. Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.

10.13. Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.

10.14. Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.

10.15. Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.

10.16. Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, most current version.

10.17. National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.

10.18. ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories- most current version.

10.19. Department of Defense Quality Systems Manual (QSM), most current version.

10.20. TNI (The NELAC Institute) Standard- most current version applicable to each lab.

10.21. UCMR Laboratory Approval Requirements and Information Document, most current version.

11.0. REVISIONS

All current revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual	Initial change of format	July 31, 2017
Quality Assurance Manual Rev.00	Updated Attachments II, III, IV, V and VII with current information Corrected grammatical errors throughout the document	July 24, 2018

ATTACHMENT I- QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:
TrueValue = Amount spiked (can also be the $\overline{CF}$ or $\overline{RF}$ of the ICAL Standards)
Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentration} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:
R1 = Result Sample 1
R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^{N} W_i * (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} W_i * (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} W_i * (Y_i - \overline{Y})^2\right)}}$$

With:	N	Number of standard samples involved in the calibration
	i	Index for standard samples
	Wi	Weight factor of the standard sample no. i
	Xi	X-value of the standard sample no. i
	X(bar)	Average value of all x-values
	Yi	Y-value of the standard sample no. i
	Y(bar)	Average value of all y-values

ATTACHMENT I- QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{(n-1)}}$$

where:
n = number of data points
X_i = individual data point
$\overline{X}$ = average of all data points

AVERAGE ($\overline{X}$)

$$\overline{X} = \frac{\sum_{n=1}^{i} X_i}{n}$$

where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

$$RSD = \frac{S}{\overline{X}} * 100$$

where:
S = Standard Deviation of the data points
$\overline{X}$ = average of all data points

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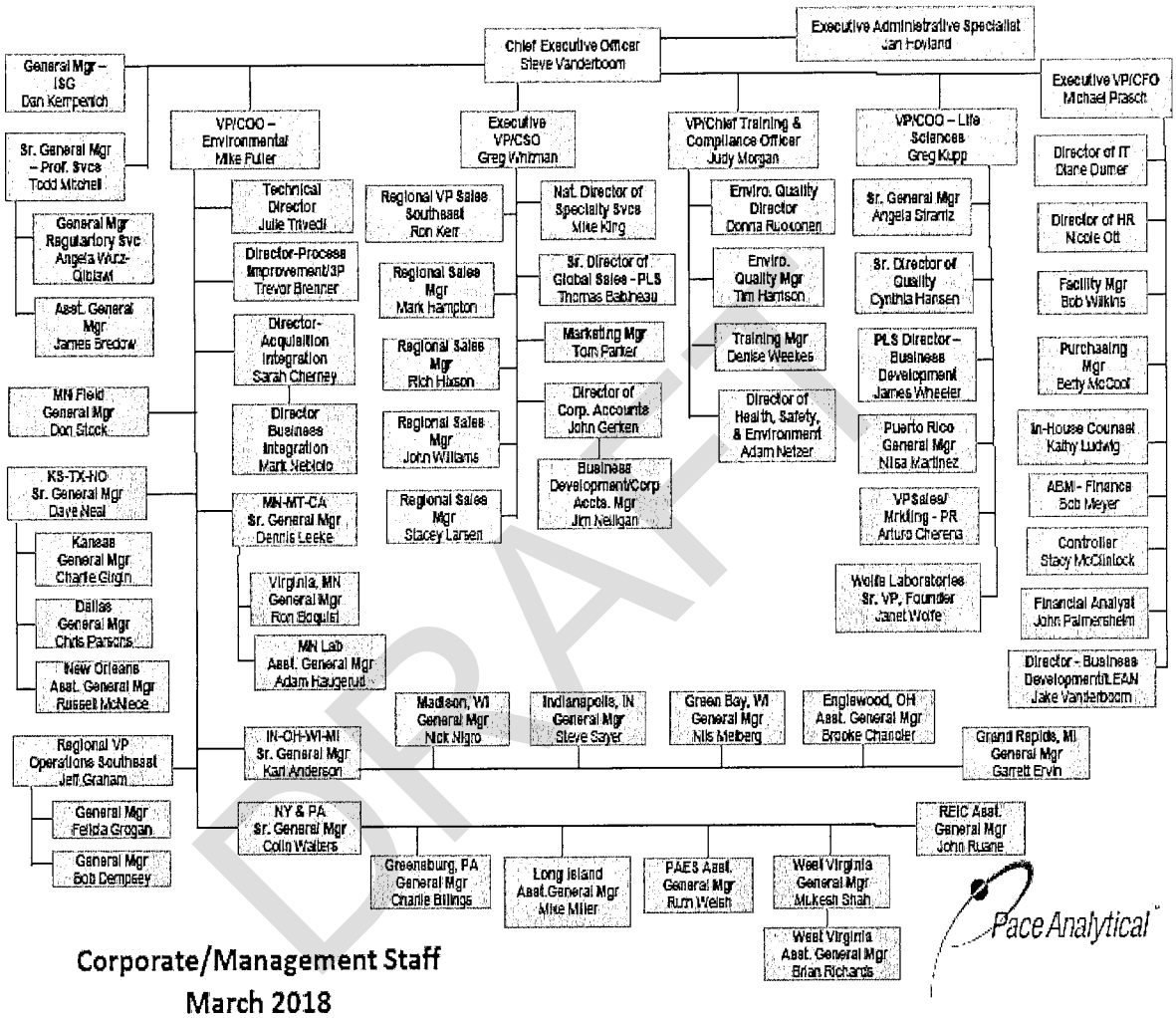
	Document No.: Quality Assurance Manual rev.01	Issuing Authorities: Pace Analytical Energy Services, LLC. Quality Office

ATTACHMENT III- CORPORATE ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



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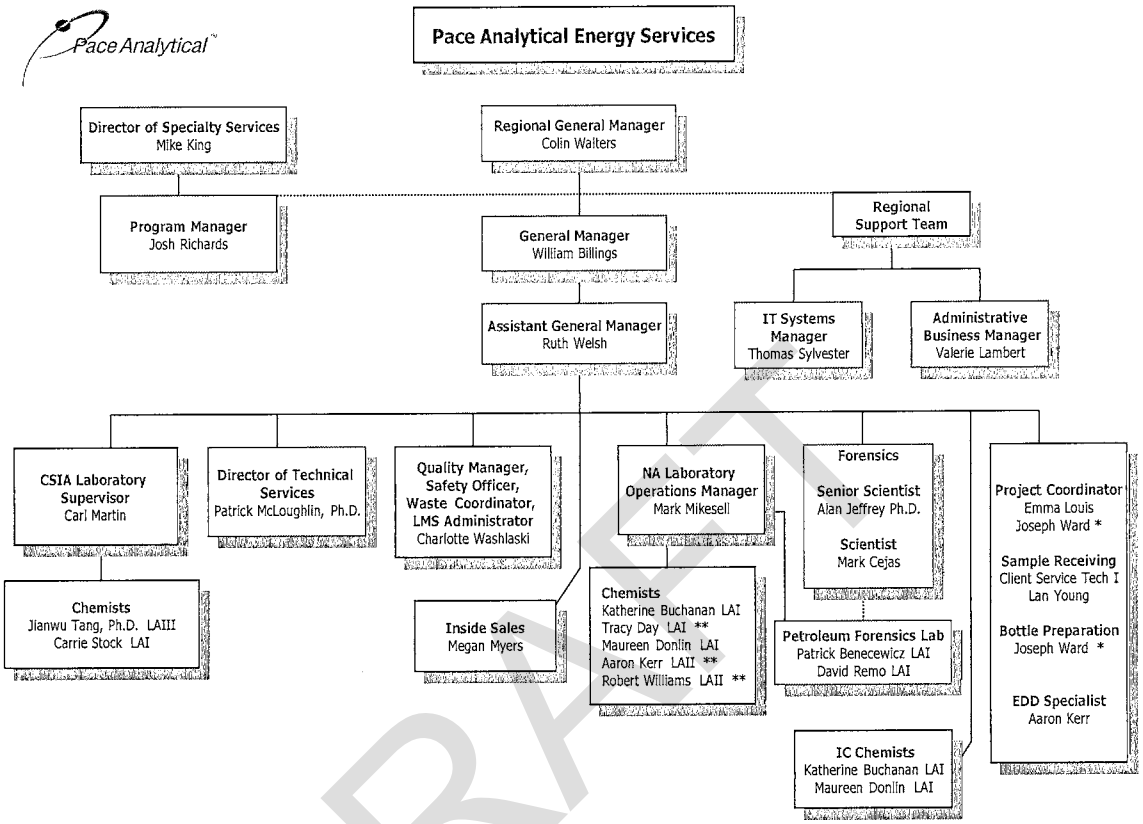
	Document No.: Quality Assurance Manual rev.01	Issuing Authorities: Pace Analytical Energy Services, LLC. Quality Office

ATTACHMENT II- LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



Last Revised – March 1, 2018
Last Reviewed – May 1, 2018

* holds safety responsibilities as well
** analyst in Petroleum Forensics lab as well

ATTACHMENT IV- EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

PAES Instrument and Equipment List

Natural Attenuation

EDONORS

Dionex Ion Chromatograph Model ISC 2000 with Degasser (Serial 08120332); Gradient Eluent Generator; AS-AP Autosampler (Serial 14092562), Columns.

Dionex Ion Chromatograph Model ISC 2100 with Degasser (Serial 14092120); Gradient Eluent Generator; AS-AP Autosampler (Serial 14092562), Columns.

Varian 3400 Gas Chromatograph (Serial 10272) with Varian 8100 Autosampler (Serial 1371)

Thermo-Fisher Scientific Ultra Trace GC (Serial 620120045) with TriPlus RSH Liquid Autosampler (Serial 241284)

Risk Analysis

Hewlett Packard 5890 Series A Gas Chromatograph (Serial 2536A05842) with Tekmar 7000 Autosampler (Serial 91099014/91135007)

Hewlett Packard 5890 Series II (Serial 3336A51836) with Tekmar 7000/7050 Autosampler (Serial 91346008/91346016)

Thermo-Fisher Scientific Ultra Trace GC (Serial 620120028) with TriPlus RSH Headspace Autosampler (Serial 237682)

Three Proprietary GCs

GOW MAC Series 580 Gas Chromatograph (Serial 580-200)

Ohaus Discovery Analytical Balance Model # DV215CD (Serial 1128122704)

Wet Chemistry/EACCEPTORS

Dionex ISC 3000 Ion Chromatograph with dual Autosamplers, columns, and ovens with conductivity and UV-VIS detectors

OI Analytical Aurora 1030 TOC Analyzer (Serial J025730751) with Autosampler (Serial E019788198)

Denver Instruments Model SI-4002 Top Loading Balance

Spectronic 20G Colorimeter

Spectronic 20D Colorimeter

Orion 601A pH Meter

Sartorius Model 1612 Analytical Balance

CSIA

Tekmar Aqua Tek 70 Autosampler (Serial US 06151001)

Tekmar Velocity XPT Purge and Trap (Serial US 06191003)

Entech 7100A Pre-concentrator (Serial 1304)

Thermo Trace GC Ultra Gas Chromatograph (Serial 200510408)

Thermo GC-Combustion III Interface (Serial 111201-175)

Thermo GC /TC Reactor OD (Serial 108520-349)

Thermo Delta V Plus Isotope Ratio Mass Spectrometer (Serial 8018)

Thermo-Electron GC (Serial 10603008) with DSQ II Mass Spectrometer (Serial 100442); Varian Archon Autosampler (Serial 14655) and Tekmar Velocity Concentrator (Serial US6047001)

Thermo Delta V Plus isotope ration mass spectrometer

Thermo Conflo IV interface

Thermo GC Isolink interface

Agilent 7890A GC System

Tekmar Aquatek 100 autosampler

Tekmar Stratum Purge and Trap concentrator

Entech 5400 Thermal Transfer System

Entech SL2 Perconcentrator

Agilent 6890N GC (Serial US10226064)

Agilent 5973N MSD (Serial US63810430)

Teledyne Tekmar Aquatek100 Autosampler (Serial US11348004) and Stratum Concentrator (Serial US11327002)

GC/MS Chemstation Datasystem (SN 2UA71516GF)

Agilent 6890N GC (serial # US10232118)

Agilent 7890A GC (serial # CN12121090)

Agilent 5975C MSD (serial # US12157802)

Agilent G4513A autosampler (serial # CN12090144)

Agilent G1888 Headspace Autosampler (serial IT40220036)

UPS (Model # TX90-10K)

UPS (Model # T90-EBP920)

Pacific Air Jun-Air Compressor Model 6 (serial # 1010200822)

Supelco 29541-U High Capacity Gas Purifier (serial # 1312955/1A-22)

Fisher Scientific Ultrasonic cleaner (serial # RUA030263007)

Eppendorf Centrifuge 5810R (serial # 581101849)

New Brunswick Scientific Innova 2000 Platform shaker (serial # 300544191)

Pelton & Crane Sterilizer (serial # AF - 005387)

Zymark TurboVap LV evaporator (serial # 04384)

Petroleum Forensics

HP Agilent 7890A GC/5975 MS System (Serial # CN12091092)

HP Agilent 6890 GC/5973 MS System (Serial # US00008852)

HP Agilent 6890 GC/5973 MS System (Serial # US00006875)

HP Agilent 6890N GC System (Serial # US10347026)

HP Agilent 6890 GC System (Serial # US00001417)

HP Agilent 5890 GC System

HP Agilent 5890 GC/5971 MS System

Tekmar LCS 2000 Purge and Trap

ESI Autosampler

Polyscience Refrigerated Recirculator

Zymark TurboVap 500 Concentrator

Sargent-Welch SWT-603D Scale (Serial # T0121781)

ATTACHMENT V- LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

Description	Document Number
Prep of SOPs	S-PAE-Q-001
Document Control and Management	S-PAE-Q-002
Manual Integration	S-PAE-Q-003
Data Validation	S-PAE-Q-004
Corrective Action Reports	S-PAE-Q-005
Equipment Maintenance	S-PAE-Q-006
Internal Audits	S-PAE-Q-007
Monitoring Controlled Temperature Units	S-PAE-Q-008
Support Equipment	S-PAE-Q-009
Determination of Detection Limits and Reporting Limits	S-PAE-Q-010
Reference Materials and Reagents	S-PAE-Q-011
Logbooks	S-PAE-Q-012
Evaluation and Qualification of Vendors	S-PAE-Q-013
Purchasing of Lab Supplies	S-PAE-Q-014
Training	S-PAE-Q-015
Bubble Strip Sampling	S-PAE-RISK-001
PM01C	S-PAE-RISK-002
RSK175M	S-PAE-RISK-003
AM20GAx	S-PAE-RISK-004
AM4.02	S-PAE-RISK-005
Document Numbering	S-ALL-Q-003-rev.09
Laboratory Documentation	S-ALL-Q-009-rev 06
Quarterly Quality Reports	S-ALL-Q-014-rev.07
Management Review	S-ALL-Q-015-rev.03
AM21G In house only	S-PAE-VFA-001 In house only
AM21G Scrubbed	S-PAE-VFA-001 (S)
AM23G In house only	S-PAE-LLVFA-001 In House only
AM23G Scrubbed	S-PAE-LLVFA-001 (S)
Waste Management Training	S-PAE-W-001
Waste Handling and Management	S-PAE-W-002
Fume Hood Monitoring	S-PAE-S-001
Regulated Soil Handling	S-PAE-S-002
Subcontracting	S-PAE-C-001
Bottle Prep	S-PAE-C-002
Sample Receiving	S-PAE-C-003
Chain of Custody	S-PAE-C-004

pH	S-PAE-I-001
TOC	S-PAE-I-002
Anions by IC	S-PAE-I-003
Cations by IC	S-PAE-I-004
HORIZON LIMS	S-PAE-IT-001
SPME In house only	S-PAE-CSIA-001
Purge & Trap for CSIA In house only	S-PAE-CSIA-002
VI	S-PAE-CSIA-003
Carbon & Hydrogen Isotopes (1,4 dioxane) In house only	S-PAE-CSIA-004
Chlorine In house only	S-PAE-CSIA-005
1,4 Dioxane prep In house only	S-PAE-CSIA-006
Carbon Soil Prep CSIA In house only	S-PAE-CSIA-007
Full Scan	S-PAE-PF-001
Whole Oil	S-PAE-PF-002
Oxygenates	S-PAE-PF-003
EDB & Organic Lead	S-PAE-PF-004
SIM DIS	S-PAE-PF-005

ATTACHMENT VI- LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE) SCOPE AND APPLICATION CERTIFICATES ARE MAINTAINED AND FILED IN THE LOCAL QUALITY DEPARTMENT

PAES holds the following certifications:

- National Environmental Laboratory Accreditation Program (NELAP): Pennsylvania
- Connecticut
- Virginia
- South Carolina
- Texas
- New York
- New Jersey
- New Hampshire
- West Virginia

Specific parameter lists for the various certifications are available from the Client Service Department upon request.

NELAC Accredited Parameters/Methods

Primary NELAC: Pennsylvania
Secondary NELAC: NY, NJ, NH, VA, CT, SC, TX, WV
(Not all states accredit all parameters.)

Parameter	Method
Chloride	SW846-9056
Nitrate	SW846-9056
Nitrite	SW846-9056
Sulfate	SW846-9056
TOC/DOC	SW846-9060, SM 5310C
pH	SM 4500H+
Light Hydrocarbons	RSK175M
Volatile Fatty Acids	PAES SOP-AM23G

Call Client Service Department for state-specific analyte list.

ATTACHMENT VII- METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE (CURRENT AS OF ISSUE DATE)

Note that all analyses listed are not necessarily performed at all Pace laboratories and there may be additional laboratory analyses performed that are not included in these tables.

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acid Base Accounting	Sobek	Solid	Plastic/Glass	None	N/A
Acidity	SM2310B	Water	Plastic/Glass	≤ 6°C	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	≤ 6°C	14 Days
Actinides	HASL-300	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid	Plastic/Glass	None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass (NY requires separate bottle filled to the exclusion of air)	≤ 6°C	14 Days
Alkylated PAHs		Water	1L Amber Glass	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid	8oz Glass	≤ 10°C	1 Year/40 Days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	≤ 6°C; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F,	300.0	Solid	Plastic/Glass	≤ 6°C	All analytes 28

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)					days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	Cool to above freezing but < 6°C	48 hours
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Asbestos	EPA 600/R-93/116	Solid	Plastic/Glass; bulk- 2" square; popcorn ceiling- 2tbsp; soil- 4oz	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤ 6°C; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	≤ 6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Biomarkers		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6°C	48 hours
Boiling Range Distribution of Petroleum Fractions (SIM DIS)	ASTM D2887-98	Product	2 X 40ml VOA vials	Unpreserved	Unlimited Cannot analyze on waters or soils
BTEX/Total	TO-3	Air	Summa	None	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Hydrocarbons			Canister		
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	72 Hours
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloroacetic acid pH <3; ≤ 6°C	28 Days
Carbamates	8318	Water	Glass	Monochloroacetic acid pH 4-5; ≤ 6°C	7/40 Days
Carbamates	8318	Solid	Glass	≤ 6°C	7/40 Days
Carbon Specific Isoptope Analysis (CSIA)	AM24	Water	40mL clear VOA vial with TLS	≤ 6°C, trisodium phosphate or HCl	N/A
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	Unknown
Cations (Ferrous Iron, Ferric Iron, Divalent Manganese)	Dionex Tech ote 10	Water	40mL clear VOA vials with mylar septum	≤ 6°C; HCl	48 Hours
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
VOC's in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum; evacuated	None	Unspecified
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil	≤ 6°C	48 Hours to filtration
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Fecal	SM9221E	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9221E	Solid	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	24 Hours
Coliform, Total	SM9222B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total	SM9221B	Solid	100mL	≤ 10°C; $Na_2S_2O_3$	8 Hours

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
			Plastic		
Coliform, Total, Fecal and E. coli	Colilert/ Quanti-tray	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	30 Hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/ 9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present-applies to SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics- TPH DRO	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range	Nw-TPH-Dx	Water	1L Amber	pH <2 HCl; ≤ 6°C	14/40 Days; 7

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Organics- NwTPH-Dx			Glass		Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C; pH <2 HCl	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	7/40 Days
Diquat/Paraquat	549.2	Water	Amber Plastic	≤ 6°C; $Na_2S_2O_3$	7/21 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Endothall	548.1	Water	Amber Glass	≤ 6°C; $Na_2S_2O_3$	7/14 Days
Enterococci	EPA 1600	Water	100mL Plastic	≤ 10°C	8 Hours
Enterococci	Enterolert	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Water	1L Amber Glass	pH < 2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Solid	4oz Glass Jar	≤ 6°C	14/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	Glass	None	Immediate
Flashpoint/ Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	≤ 6°C; pH <2 H_2SO_4 or HCl	7/40 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics (C3-C10)	8260B modified	Water	40mL vials	≤ 6°C; HCl	14 Days
Gasoline Range Organics (C3-C10)	8260B modified	Solid	4oz Glass Jar	≤ 6°C	14 Days
Gasoline Range Organics- Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics- Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6°C	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	≤ 6°C; packed jars with no headspace	14 Days
Gasoline Range Organics- Wisconsin	WI MOD GRO	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
GRO					
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	≤ 6°C in MeOH	21 Days
Glyphosate	547	Water	Glass	≤ 6°C; $Na_2S_2O_3$	14 Days (18 Months frozen)
Grain Size	ASTM D422	Solid	Not specified	Ambient	N/A
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤ 6°C	14/7 Days if extracts stored ≤ 6°C or 14/14 Days if extracts stored at ≤ -10°C
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/ SM3500Cr-B, C, D	Water	Plastic/Glass	≤ 6°C	24 Hours (see note 4)
Hexavalent Chromium	218.6/SM3500Cr-B, C, D	Water	Plastic/Glass	Ammonium Buffer pH 9.3-9.7	28 Days (see note 4)
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH >8	14 Days (see note 4)
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6°C	30 Days from collection to extraction and 7 days from extraction to analysis
Hydrocarbons in	AM4.02	Vapor	20cc vapor	None	N/A

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Vapor			vial with stopper septum; evacuated		
Hydrogen by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum; atm pressure	None	14 Days
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Light Hydrocarbons by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum; atm pressure	None	14 Days
Light Hydrocarbons in Vapor	AM20GAx	Vapor	20cc vapor vial with stopper septum; evacuated	None	Unspecified
Lipids	Pace Lipids	Tissue	Plastic/Glass	$\leq$ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	$\leq$ 6°C	28 Days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Mercury	7471/245.6	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	≤ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene, propane, propene, iso-butane, n-butane, 14 Daysacetylene	PM01/AM20GAx	Water	40ml clear VOA vials with butyl septa	trisodium phosphate or benzalkonium chloride, cool to above freezing but ≤6°C	14 Days; 7 Days unpreserved
Methane, Ethane, Ethene, propane, propene, iso-butane, n-butane	RSK 175M	Water	40ml VOA vials	trisodium phosphate, cool to above freezing but ≤6°C	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	28 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
Methanol, Ethanol	8015 modified	Water	40mL vials	≤ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	≤ 6°C	14 Days
Methyl Mercury	1630	Water	Teflon/ fluoropolymer	Fresh water- 4mL/L HCl; Saline water- 2mL/L H_2SO_4 (must be preserved within 48 hours of collection)	6 months
Methyl Mercury	1630	Tissue	2-4oz glass jar	≤ 0°C	28 Days; ethylated distillate 48 hours
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Total Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Total	SM4500-	Water	Plastic/Glass	pH<2 H_2SO_4; ≤	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Kjeldahl (TKN)	Norg/351.2			6°C	
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	28 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	72 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
Oil Range Organics	8015	Solid	Glass	≤ 6°C	14/40 Days
Oil Range Organics	8015	Water	Glass	≤ 6°C	7/40 Days
Organic Matter	ASA 29-3.5.2	Solid	Plastic/Glass	None; samples air-dried and processed prior to analysis	N/A
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Oxygenates on Product (GCMS SIM)	1625 modified	Product	10mL glass vial	≤ 6°C	14 Days (7 Days from extraction)
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine	608	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	Pest: 7/40 Days; PCB: 1 Year/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
(OC)					
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3; pH<2 HCl; ≤ 6°C	14/30 Days
PCBs, total as Decachlorobiphenyl	508A	Water	1L Glass, TFE lined cap	≤ 6°C	14/30 Days
Perchlorate	331	Water	Plastic/Glass	≥0-6°C, field filtered with headspace	28 Days
Permanent Gases (oxygen, nitrogen, carbon dioxide, carbon monoxide)	PM01/AM20GAx	Water	40ml amber VOA vials with butyl septa	benzalkonium chloride and cool to above freezing but ≤ 6°C	14 Days
Permanent Gases by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum; atm pressure	None	14 Days
Permanent Gases in Vapor	AM20GAx	Vapor	20cc vapor vial with stopper septum; evacuated	None	Unspecified
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	≤ -10°C	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; ≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6°C	1 Year/1 Year
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/1 Year
PCB Congeners	1668A	Water	1L Amber	≤ 6°C but above	1 Year/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
			Glass	freezing	
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particle Size	ASA 15-5 modified	Solid	Plastic/Glass (100g sample)	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	28 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	7 Days
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Purgeable Organic Halides (POX)	9021	Water	Glass; no headspace	≤ 6°C	14 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid	Plastic/Glass		
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days
Saturated Hydrocarbons		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Saturated Hydrocarbons		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source	EPA 201A	Air	Filters	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PM10					
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤ 6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid	Plastic/Glass	None	180 days
Total Inorganic Carbon (TIC)	PM01/AM20GAx	Water	40ml clear VOA vial with butyl septum	Cool to above freezing but < 6°C	14 Days
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black/Lloyd Kahn	Solid	Glass	≤ 6°C	14 Days
Total Organic Halogen (TOX)	SM5320/9020	Water	Glass; no headspace	≤ 6°C	14 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Water	40mL vials	pH<2 HCl, no headspace, ≤ 6°C	7 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Solid	Glass	≤ 6°C	14 days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
UCMR Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days
UCMR Hexavalent Chromium	218.7	Water	HDPE or propylene	Na_2CO_3/$NaHCO_3$/$(NH_4)_2SO_4$; pH>8	14 Days
UCMR Chlorate	300.1	Water	Plastic or glass	EDA	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
UCMR Perfluorinated Compounds	537	Water	Polypropylene	Trizma	14 Days
UCMR 1, 4 Dioxane	522	Water	Glass	Na_2SO_3, $NaHSO_4$; pH<4	28 Days
UV254	SM5910B	Water	Glass	≤ 6°C	48 Hours
Vermiculite	EPA 600/R-93/116	Solid	Plastic/Glass	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Volatile Fatty Acids	AM21G	Water	40mL clear VOA vials; Teflon septa	Cool to above freezing but < 6°C	21 Days
Volatile Fatty Acids (low level)	AM23G	Water	40mL clear VOA vials; Teflon septa	Cool to above freezing but < 6°C, Benzalkonium chloride	14 Days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C; packed jars with no headspace	7/28 Days
Volatiles	TO-14	Air	Summa Canister	None	28 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	TO-15	Air	Summa Canister or Tedlar Bag	None	28 Days
Volatiles	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	≤ 6°C but above freezing	28 Days
Volatiles	TO-18/8260	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	8260	Solid	5035 vial kit	See note 1 (analyze for acrolein and acrylonitrile per	14 days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				local requirements)	
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present (or unpreserved if run within 7 days of collection) (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6°C; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days
Whole Oil C3-C36 Whole Oil	ASTM D3328	Product	2 x 40ml VOA vials	Unpreserved	Unlimited Cannot analyze on water or soil
Parent and Alkylated PAHs	8270 (SIM) Modified	P, S, W	P - 2 x 40ml VOA Vials S - 1 x 4oz jar W – 2 x 1L Glass	Unpreserved Ice, maintained at ≤6°C	Unlimited if product, 14 days if solid or water
C8-C40 Full Scan	ASTM D5739 (GC/MS)	P,S,W	P – 2 x 40ml VOA Vials S – 1 x 4oz jar W – 2 x 1L	P-None S or W-ice, maintained at ≤6°C	Unlimited if product, 14 days if solid or water

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
			Glass		
Oxygenated Blending Agents	EPA 1624 Modified	P~~, W~~	2x40mL VOA vials ~~W – 2x40mL VOA vials~~	P – NONE ~~W – HClL; ice, maintained at 6°C~~	Unlimited ~~If water – 14 days~~
Organic Lead and Lead Scavengers	GC-ECD	Product	2 X 40ml VOA vials	None	Unlimited Cannot analyze water or soil
C3-C12~~0~~ PIANO	GC/MS	P, W, S	2x40mL VOA vials W – 2x40mL VOA vials S – 1 x 4oz jar	P – NONE W – HCIL; ice, maintained at 6°C	Unlimited If solid or water – 14 days

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4°C, **and** one vial methanol preserved at ≤6°C **and** one container of unpreserved sample stored at ≤6°C.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA Method Update Rule. Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate buffer to extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR program.

ATTACHMENT A-4

QUALITY ASSURANCE PLAN MCL-7701, MATERIAL AND CHEMISTRY LABORATORY, LLC
OAK RIDGE, TENNESSEE

MATERIALS & CHEMISTRY LABORATORY, INC.

"Linking Technology to Solutions"

Quality Assurance Plan
MCL-7701

Materials and Chemistry Laboratory, Inc.
East Tennessee Technology Park
Building K-1006
2010 Highway 58, Suite 1000
Oak Ridge, Tennessee 37830

Issued Revision 13
Revision 13.1, December 6, 2013
Revision 13.2, April 7, 2014
Revision 13.3, November 24, 2014
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Revision 14, September 2015
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Revision 14.3, November 22, 2016
Revision 14.4, December 20, 2016
Revision 14.5, May 3, 2017
Revision 14.6, December 2017
Revision 14.8, December 2018
Revision 14.9, May 2019

Controlled Copy No. _____

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Appendices

1.0 ORGANIZATION

1.1 Introduction

Materials and Chemistry Laboratory, Inc. (MCLinc) provides technical support to a variety of customers and programs. Work done may be classified at levels up to U.S. Department of Energy (DOE) "Q" or "QX" (S-RD [level: secret – Category: Restricted Data]), and may involve radioactive, special nuclear materials (SNM), and/or hazardous materials. Scope of work includes, but is not limited to, characterization studies, research projects, development efforts, lab-to-bench-to-pilot scale processes, process optimization, and methodology development. Quality is inherent in all aspects of MCLinc work. This plan and the references herein, ensure that a management framework is defined for the establishment of quality MCLinc practices.

It is noted that this plan does not specifically address all aspects of the Industrial Hygiene Analysis Laboratory (IHL) within MCLinc. The IHL is an American Industrial Hygiene Association Program (AIHA) Laboratory Accreditation Program (LAP), LLC (AIHA) accredited laboratory. The latest AIHA assessment was done under the requirements of International Organization for Standardization/International Engineering Consortium (ISO/IEC) 17025 international standard. The IHL operates under a stand-alone quality plan, MCLinc, "Industrial Hygiene Laboratory Quality Assurance Manual," MCL-7719. The information contained within the MCLinc Quality Assurance Plan (QAP) will still apply to the overall operation of all IHL functions, but will not specifically address some of the AIHA-required details that are unique to the IHL. This document and other supporting Standard Operating Procedures (SOPs) will apply to all MCLinc AIHA accreditations.

1.2 Quality Assurance Policy

The MCLinc Quality Assurance (QA) Policy approved by the MCLinc President is to assure that the *QA* practices utilized by the MCLinc staff conform to requirements, standards, and responsibilities necessary for maintaining a quality organization in conjunction with DOE, and customer-based expectations. This policy incorporated into this QAP will help to minimize the risk and environmental impact of processes influenced and performed by MCLinc as well as maximizing the safety, reliability and performance of MCLinc methodologies and practices. The MCLinc QA Policy must be

rigid to assure quality objectives are met, but also dynamic by having procedures in place to allow continual improvement in the quality management process (annual assessment, SOP change procedure, and corrective action procedures are examples of means to allow improvement).

This QAP is designed to specifically meet the management and technical requirements for testing facilities of the internationally recognized standard ISO/IEC 17025 and American Society of Mechanical Engineers, Nuclear Quality Assurance, Level 1 (ASME NQA-1), National Environmental Laboratory Accreditation Conference related U.S. Environmental Protection Agency (EPA) and DOE documents noted in the Reference Section. See Appendix A for a cross-reference table, by section of this QAP, to the specific requirements of ISO/IEC 17025, Title 10, Code of Federal Regulations (CFR) Part 830.120, American National Standards Institute/American Society for Quality Control (ANSI/ASQC) E4-1994, and ASME NQA-1. Changes to this document may only be made with the approval of the MCLinc President and Quality Assurance Officer (QAO) per the SOP "Document Control", MCL-7703.

1.3 Organizational Responsibilities

The organizational structure of MCLinc is shown in Appendix B. MCLinc must be able to maintain flexible work responsibilities to ensure that a wide variety of customer, Site (East Tennessee Technology Park and URS/CH2M Oak Ridge, LLC and Landlord (DOE and Community Reuse Organization of East Tennessee [CROET]) requirements can be met.

1.4 Functional Responsibilities

The MCLinc President provides daily guidance and administrative support to the MCLinc staff and is committed to ensuring compliance to this QAP and ISO/IEC Standard 17025, AIHA, and other quality requirements of our customers. The MCLinc President is supported by the Technical Director (TD) and the Laboratory Manager (LM). These positions provide routine assistance to personnel and customers on the capabilities and application of MCLinc resources to solving problems.

The TD has responsibility to provide technical direction to the Project Manager (PM) and technical assistance to our clients. The LM has responsibilities for the day-to-day operation of the laboratories and to make sure resources are available to meet the needs of our clients and this plan. The Quality Assurance Specialist (QAS) performs quality duties as assigned by the QAO. The QAS is the designated alternate for the QAO in his absence.

All staff members of MCLinc are responsible for ensuring that customer objectives (i.e., quality, time frame, budget, applicability) are met in accordance with this plan and any other applicable documentation. The work performed by any and all staff members is necessary to meet our management quality objectives and those of our clients. A staff

member is empowered to stop work due to any safety issue or when the quality of the product is endangered and report such concerns to the QAO. Appendix C lists MCLinc personnel and their support functions which will help ensure the quality objectives of MCLinc and its customers are met. The QAO is the point of contact for the implementation and enforcement of quality-based procedures. Conflicts in operating methods and procedures will be resolved by the QAO with support from the appropriate management personnel. The QAO is committed to compliance to this QAP and ISO/IEC Standard 17025 and other quality requirements of our customers. The QAO, as necessary, develops and issues SOPs or QA Directives in memo format to further define or explain items covered by this QAP or other areas needing procedures defined.

The QAO has the authority to stop work at any time to assess a reported problem or investigate a quality system failure or trend.

The PM is either self-appointed or is selected by the LM or MCLinc President based upon the nature of the project. The PM is the person responsible for the control and coordination of all activities associated with the successful completion of a customer task.

1.5 Facility/Security Attributes

MCLinc is leasing the K-1006 facility from the DOE, through CROET as part of the DOE reindustrialization initiative. The brick facility is approximately 28,000 square feet (ft^2). The area available to MCLinc under its lease with the CROET is approximately 25,300 ft^2. The facility is designed to be a laboratory facility. The second floor contains office space (15 rooms) and one storage closet. The first floor accommodates approximately: 30 labs, 12 offices, 5 administrative/common areas, 2 maintenance areas, 5 hallways, and 2 utility chases. There is approximately 12,445 ft^2 of laboratory space and 12,885 ft^2 of non-laboratory space within the MCLinc facility. The facility is dedicated to handling virtually any type of environmental contaminant and is operated by a multidisciplinary staff qualified to address technical issues pertaining to radiological and hazardous materials.

Although MCLinc is a private commercial entity, it still has some operational restrictions based upon where and what type of business it does. As part of the basis for MCLinc to continue to be authorized to perform classified work, a graded approach to physical security had to be implemented. The physical security, and hence the main basis for the security infrastructure of the MCLinc facility, is based upon three to four layers of access control. The first layer is that access to the site is monitored by Site Security. All visitors *must be properly badged per DOE requirements*. The second layer is the controlled access requirements (Hirsch badge reader system or controlled access key) to gain access into building K-1006. The third layer is controlled key access into the laboratory. The fourth layer is the controlled access storage area within various laboratories. The Facility Security Officer (FSO) will assign keys. All assigned keys will either be physically controlled by the individual person or will be controlled by that individual using a unique

lock/combination controlled storage area. Lost keys will be reported to the FSO immediately. This graded approach to physical security provides the required control over facility access for security issues as well as for proper chain of custody (COC) of certain materials and samples.

1.6 Commitment to Quality

The managers, owners and employees of MCLinc, an employee-owned company, are committed to a policy whereby all personnel are free of any undue internal or external commercial, financial, or other pressures and influences which may adversely affect the quality of the work. Any staff member feeling such pressures shall report this concern immediately to the LM, TD, QAO or the MCLinc President for investigation and corrective action.

As noted under the Facilities Section, the facility is secure and the staff is knowledgeable in the handling of confidential information for the DOE. This same approach is extended to all clients in that client confidential information or proprietary rights are maintained in confidence and all such documents or electronic files are stored in locked files or in computers password-protected and accessible only to authorized staff. See Section 5.5, Classified Materials. The MCLinc reputation and success depends on the high integrity of the staff. MCLinc's policy is that technical and business competence, impartiality, judgment, and data quality and operational integrity must be maintained at all times. These elements are key to maintaining the quality of our efforts. The employees therefore must be aware of their contributions to maintaining the management quality system.

The MCLinc management staff has the responsibility and authority to provide the resources to complete the above and through staff and project meetings and other communication tools (i.e., e-mail) encourage the staff to communicate their assessment of the management system. The MCLinc management also has the responsibility for training, implementation, maintenance and improvement of the management system and to identify and correct variances from the system. The QAO, TD, LM, PM and QA assessments are key in identifying any variance from this policy which must be investigated and corrective action taken including disciplinary action.

2.0 MANAGEMENT SYSTEMS

2.1 Standards and Reference Materials

MCLinc has a need for a variety of standards and reference materials. Where possible, these standards and reference materials must be purchased from an ISO/IEC 17025; 2005 certified vendor. Traceability of these standards must also be demonstrated on the Certificate of Analyses of the standard. The standards and reference material must be handled, stored and used per the manufacturer's specification to avoid contamination and deterioration.

Many standard methods require use of second source standards to check primary standards (i.e., organics and metals). The "Quality Systems for Analytical Services" (QSAS) requires radiation calibration standards to be verified prior to use and annually as follows:

- At least three verification measurements of a standard are used to determine the mean value and standard deviation of verification results.
- Mean value is within 5 percent (%) of certified value.
- Two sigma deviations is less than 10% of the mean value.

If specifications are not met, corrective actions must be evaluated and implemented.

2.2 Calibration

Instrument and equipment performance evaluation, maintenance, and documentation are the responsibility of the instrument owner. Appendix D lists the responsible owner and authorized operator for the major instrumentation with the MCLinc facility. These instruments have specific QA documents that outline the minimum calibration requirements. For the general or common data acquisition laboratory equipment (e.g., balances, pH meters,) the "Calibration, Maintenance and Inspection Plan," MCL-7711, outlines the calibration and documentation requirements for those components which may influence the work being performed. When there is a need for outside calibration of laboratory equipment, the vendor/material used must be traceable to national standard setting bodies such as National Institute of Standards and Technology (NIST) or ISO approved.

2.3 Facility Maintenance

The housekeeping and maintenance of each MCLinc office or laboratory facility is the direct responsibility of all MCLinc personnel. MCLinc facilities should be kept clean and orderly and the temperature and humidity controlled to meet the needs of the testing instrument. If the MCLinc staff member responsible for an area is temporarily or permanently unable to comply with this standard, he or she should advise management of the problem. Specific health and safety requirements are outlined in the "Chemical Hygiene Plan," MCL-7702 and the "Health and Safety Plan," MCL-7717. Specific requirements for maintenance and facility documentation are provided in the "Calibration, Maintenance and Inspection Plan," MCL-7711.

2.4 Work Environment

MCLinc maintains a safe and clean working environment. All laboratory areas and materials are maintained in a clean and orderly fashion to ensure the work performed will not be compromised by the local environment of the laboratory facility. The MCLinc personnel performing work are responsible for ensuring that all cleanliness requirements are met prior to commencing work. The "Chemical Hygiene Plan," MCL-7702, provides additional detail.

2.5 Laboratory Supplies

These materials are stored and controlled based upon the hazardous nature of the material. Individual personnel are responsible for ensuring that the integrity of the laboratory supplies is adequate to meet MCLinc and the customer's expectations. The ordering, reporting and tracking of chemicals is addressed in the "Chemical Hygiene Plan," MCL-7702 and the "Procurement Control Plan," MCL-7727. The ordering, reporting, and tracking of radiological materials are addressed in the "Implementation SOP for the Radiation Protection Plan," MCL-7715.

2.6 Special Nuclear Materials (SNM)

The control and monitoring of SNM is detailed in "Nuclear Materials Control and Accountability Plan," MCL-7706. The Radiological Safety Officer (RSO) is responsible for oversight and control of SNM.

2.7 Material and Sample Receipt

Samples and materials are received at the MCLinc facility from various sources and require various levels of oversight and control. Sample login, tracking, documentation, archival, disposal, and/or return procedures are detailed in "Project Management Plan," MCL-7704 and Operator Aid Appendix O in MCL-7756, "Operator Aids." This procedure provides guidance on issues such as non-conformance reports (NCR), cross contamination, inspection log sheets, sample tracking and management, and laboratory records associated with sample management. The "Procurement Control Plan," MCL-

7727 and Section 3.1 provide details on the quality control (QC) checks and documentation required for receipt of materials.

2.8 Controlled Samples

Controlled samples have COC documentation. COC forms can either be supplied by the customer or by MCLinc. The COC ensures that the samples will be maintained within the MCLinc facility. The COC form will be documented to reflect when the samples either leave the MCLinc facility or a non-MCLinc employee is provided direct, unsupervised, access to the samples. No internal COC control is required for samples remaining within the MCLinc facility.

2.9 Non-COC Samples

Many projects that are performed by MCLinc are on samples that do not have an associated COC and are typically representative of a process or condition that does not require COC control. These samples are maintained within the secure MCLinc facility. The use and control of these samples is the responsibility of the PM. The PM may elect to document any special handling or storage protocols that should be used for a given sample or group of samples. The PM is responsible for ensuring that proper documentation and labeling is provided to ensure that any MCLinc employee that may need to utilize these materials understands the specific requirements associated with the samples.

2.10 Sampling and Sample Preparation

In MCLinc projects where actual sampling of the process is required, the details of the sampling process and procedures to follow must be defined in a project sampling plan or scope of work. All samplers must be trained on the procedures and understand the critical importance of the sample to the project. The resultant sample must represent the source being sampled and the PM must define steps to be taken to best approximate a homogenized sample. This is also a critical step in sub-sampling samples received at the laboratory for testing.

The staff member responsible for the analysis shall determine sample preparation techniques utilized. Sample preparation techniques shall be documented. Good laboratory notebook protocols will be used when documenting the laboratory, data, and/or project activities. Additional quality, safety, and environmental aspects of sample preparation are provided in, "Sample Preparation Plan," MCL-7710.

2.11 Instrumentation and Maintenance

MCLinc has a variety of laboratory instrumentation ranging from very complex (e.g., transmission electron microscope) to standard laboratory instrumentation (e.g., pH meter). The level of the documentation required for the standardized use of instrumentation is decided by the LM. The instrumentation listed in Appendix D requires

some level of QA and/or operational guidance. The use and control of the general laboratory equipment is provided in "Calibration, Maintenance and Inspection Plan," MCL-7711, technology specific SOPs (e.g., MCL-7708, "Electron Microscope Operations Plan"), project specific QA plans, vendor manuals, and other customer specific documentation. MCLinc staff members using an instrument are responsible for documenting non-routine maintenance and repairs, and maintaining an inventory of consumables and commonly needed parts.

2.12 Quality Control Samples and Assessment of Data

Since the vast majority of the projects performed by MCLinc are non-routine or the application of a routine procedure to a non-routine use, the measurement quality objectives vary significantly. The basic objective of all MCLinc measurements/analyses are to assure: (1) the procedure measures the parameter of interest, (2) the instrument/system is calibrated and operating properly, (3) the sample was properly prepared and handled in a way to minimize contamination, and (4) the data is calculated, reviewed and reported properly. Depending on the procedure or technique utilized MCLinc achieves the above by using QC samples. These QC samples include one or more of the following:

- Method Blank
- Instrument Blank
- Calibration Check Standard
- Laboratory Control Sample's (Duplicates)
- Matrix Spike and/or Matrix Spike Duplicate
- Duplicate Sample
- Certified Sample

In many cases the QC sample requirements, if not defined by the procedure, are defined by the client and MCLinc at time of project inception. Any anomalies or failures of QC samples must be evaluated and if persistent reported as a non-conformance requiring corrective action.

The laboratory control samples (LCSs) shall be used by the analyst to evaluate method performance. In cases where the method is run infrequently (less than 20 samples per month), the analyst shall evaluate LCS recoveries against criteria in the method or use a default of 100% ± 25% recovery. Corrective action will include rerun of the LCS and if it still fails evaluation by the QA*O* verses client project needs.

For analytical methods requiring LCSs and run frequently (more than 20 samples per month), LCS data shall be tabulated for review by the analyst to see trends with calculation of the standard deviation (sigma). The data points generated for each sample set should be evaluated as follows:

± 1 Sigma shall contain 2/3 of the points
± 2 Sigma shall contain 19/20 filter points

± 3 Sigma shall contain ALL of the points

If not, corrective action as noted above shall be taken.

Shewhart type control charts may also be used to display and assess the data. These are especially useful for single analyte analyses.

The QA*O* will maintain a list of those methods using control tables or charts.

If required by the project the results of the QC samples may be utilized to calculate and estimation of uncertainty for the reported data in "Estimation of Uncertainty of Measurement (EUM)", MCL-7735.

2.13 Data Review and Evaluation (Design Control)

The PM will define the level of data review required to meet the project quality objectives and customer's expectations. The MCLinc minimum standard for data review is a two level review of an initial (level one) review by the analyst/instrument operator assuring that the analyses was properly performed, calculations are checked and the acceptance criteria for the method were met. The QAO, another analyst or TD will perform a second review (level 2 review) of the parameters listed plus the final report.

During any step in the data review and quality analysis process, any quali8ty data outside of the method or project set criteria must be evaluated for appropriate corrective action. If the analyst sees a QC failure, they shall rerun the QC sample. If it fails again, the results are presented to QA and the following evaluation must occur:

a) *If the QC is biased high (CCV, LCS or MS), and the sample results are below the reporting limit, report the results with a qualifier noted in the report*

b) If the QC recoveries are biased low even after being reanalyzed; the samples after the last acceptable QC sample should be re-prepped and reanalyzed. IH samples or samples consumed in the initial prep should be reported with the anomaly described in the case narrative. For low MSs, perform .a post digestion spike to evaluate the cause. (i.e., was it a matrix effect).

c) If the blank is contaminated above ½ of the reporting limit, but not detected in the samples, report the data and note the anomaly in the case narrative. The possible cause of the blank contamination shall be investigated and corrective action taken. If the samples appear to be contaminated when analyzed without dilution, then the samples and QC shall be re-prepped and reanalyzed. IH samples or samples consumed in the initial prep should be reported with the anomaly described in the case narrative and reported with a B qualifier. If one or more of the samples have a high concentration of the analyte requiring dilution to analyze, the sample is most likely the cause of the blank cross contamination and the results should be reviewed with QA for appropriate action prior to reporting.

d) An exception to the above requirement for re-prep and re-analysis is the case where such action would cause missing the client's turnaround time (TAT). In this case the Laboratory Manager (LM) or Quality Assurance Manager (QAM) should contact the client to discuss the issue and take the action agreed upon. Also, if insufficient sample is available to re-prep, the data must be qualified.

Additional internal data review (level three) will be provided by appropriate senior technical staff as warranted per the subject matter of the data and/or the requirements of the project. In no instance will data be reported without review. Information reported prior to completion of the review/evaluation process must be clearly identified as "preliminary data".

Computer programs that are used to produce test data or calculate test data must be self-checking or verified per "Verification of Data Software," MCL-7728.

2.14 Standard Operating Procedures (SOPs)

MCLinc uses SOPs to define routine analytical methods, quality systems, chemical hygiene, health and safety, security, and radiological. For simpler routine procedures, QC, or project specific requirements, MCLinc uses operator aids that are incorporated as controlled documents in MCLinc SOP MCL-7756, "Operator Aids," or as an attachment to an SOP.

The key elements of SOPs and Operator Aids are presented in Table 2.14. Any deviations from these elements must be approved by QA.

Current routine procedures to be covered by this document, operator aids or SOPs include the following:

- Reagent water preparation
- Sample receiving
- Balance checks
- Preparation of standards
- Temperature monitoring of ovens and refrigerators
- Calibration of thermometers
- Preventative maintenance
- Calibration of mechanical pipettes
- Checking of hood velocities
- Detection limits studies
- Preventive maintenance
- Inspection of glove boxes
- Assessment of data

A complete list of SOPs and Operator Aids is found on the MCLinc Controlled Document Status List maintained by the Document Control Coordinator (DCC).

Table 2.14 Key Elements of SOPs and Operator Aids

Section	SOP*	Operator Aid**
Title	Cover Usage in a complete statement referencing a regulatory procedure as appropriate	Clear, simple statement
Purpose	Purpose may be detailed	Included in scope
Scope	Covers use and application	Simple description of purpose and scope
Responsibilities	Defines roles of analyst, supervisor, QA and management	Not applicable
Definitions	Provide any definitions unique to SOP	Define unique terms when used

Section	SOP*	Operator Aid**
Reagents	Define each with all the details (i.e., chemical name, formula, % purity, manufacture).	Define in procedure- reagents used and source only if unique
Standards	Define each including concentration or purity	Define in procedure
Equipment	Define equipment or instrumentation utilized	State in procedure equipment used but provide details only on specialized equipment
Reagents Preparation	Describe details of preparing reagents listed above	Include for non-routine reagents in the procedure
Standardization and Calibration	Define process (including initial preparation of standards), standard numbers, levels, acceptance criteria, etc.	Cover in the procedure in stepwise format
Procedure	Stepwise details of the process which in some cases may include the why or background for each step	Simple steps to follow assuming a trained analyst
Safety (including any waste issues)	May be separate section or defined as necessary in SOP	Define in procedure where appropriate
Calculations	Outline information required and formula to use	Define in procedure the formula and its elements
QC	Define the types of QC samples required and the appropriate criteria for evaluating data. Define corrective action	Define in procedure QC samples required, corrective action, and define on prep sheet QC criteria
Documentation	Define preparation sheet or where data should be documented (i.e., notebooks)	Includes preparation sheet or defines what to record in notebook
Pollution Prevention and Waste Management	In all SOPs dealing with chemicals	Not usually needed
References	List applicable documents	List Documents
Other sections or attachments may be required to meet the needs of the SOP usage	Comment – It is also appropriate to have the SOP summarized as an Operator Aid and enclosed as an attachment.	Rarely needed

* These are the requirements for a SOP covering an analytical procedure. Other administrative or policy SOPs may not need all sections listed.

** An Operator Aid for operating equipment may just include a stepwise procedure. This aid is for an analytical procedure.

3.0 PROCUREMENT, SUBCONTRACTING AND DOCUMENTATION

3.1 Procurement

Procurement planning begins with the PM evaluating the needs of the project including the specifications of the required items. These needs are then compared to the approved sources/vendors.

Procurement will then be done through a qualified and established vendor. When a new vendor must be used prior to qualification, the vendor must provide and/or meet any requested technical and operational specifications that may influence quality, safety, and/or environmental concerns. These specifications are reviewed with the QAO and will become part of the final project documentation. The Controller at the direction of the QAO, maintains a list of approved vendors (in the MCLinc purchasing software database).

Using a MCLinc Purchase Order, the PM documents the desired product that meets the project or use required specifications by catalog or identification number. In cases where it is necessary to assure the quality of the product, the specifications required are defined in the purchase order. The purchase order is used to confirm the material or service upon receipt.

Documentation for project related purchases are maintained in the project files and the PM is responsible to assure the specifications of products received meet project needs.

Upon receipt, the procured items are checked by the PM or his designee, against the ordered item for compliance prior to use. The desired quality will be checked during initial use for critical consumables, supplies and services that affect the quality of MCLinc services. Any identified quality issue must be immediately reported to the QAO for investigation of root cause and determination of corrective action (See Section 2.7, Material and Sample Receipt).

For the purposes of NQA-1 requirements, MCLinc does not purchase materials for direct Nuclear Facility-Related use. All day-to-day procurements are via purchase order and are commercial grade items with specifications clearly defined by the vendor. If a unique

item is required, the PM under the direction of the QAO/TD reviews the design and/or specifications and seeks and evaluates qualified vendors or sources. Any new vendor must be approved by the QAO.

Further details of the procurement process are defined in "Procurement Control," MCL-7727.

3.2 Subcontracting

When MCLinc uses a subcontractor for support services or testing services that it does not provide, or for workload overflow; the client is informed of this approach and a competent pre-approved subcontractor is used. The subcontractor must meet any certifications required by the project, (i.e., AIHA).

The need for subcontract services is identified in the project planning stage and if the services required are not available through a previous approved subcontractor a new subcontractor will be sought. This involves definition of the requirements for the services needed, along with any certifications required and solicitation of the supporting documentation from potential vendors. The PM will review the documentation and make a recommendation to the QAO/TD for final approval.

MCLinc President — Date: 9/1/15

Quality Assurance Officer — Date: 9/1/15

Section: Non-Conformances, Corrective & Preventative Actions

Section No: 4

Revision: 14

Date: 09/01/2015

4.0 NON-CONFORMANCES, CORRECTIVE AND PREVENTATIVE ACTIONS

During the course of normal business activities, problems may arise that potentially impact the quality of the work and/or MCLinc's ability to meet our client's requirements. These problems must be reported by the individual identifying the problem in a timely manner to responsible staff (QAO, TD, LM, or MCLinc President).

The problem will then be investigated and appropriate corrective action taken to resolve and eliminate future reoccurrence as required by 10CFR21, "Reporting of Defects and Non-compliance." The goal of each investigation is to determine where possible the root cause or real source of the error or variance. When found this "root cause" must be documented and become part of the lessons learned information passed on to management and staff. The QAO will randomly assess the documentation and implementation of corrective actions on quality related issues. Consideration will be given during the investigation to any preventive actions necessary to avoid future issues (e.g., change SOP or perform process spot check, etc)."

The MCLinc quality program encourages each staff member to be proactive and point out potential problem areas. Management will implement this preventive action with the same priority as any corrective action.

The non-conformance process and Problem/Action Report format is detailed in "Procedure for Reporting Problems, Non-Conformances and Associated Actions, MCL-7722."

5.0 PERSONNEL TRAINING AND QUALIFICATIONS

MCLinc personnel are qualified to perform their job duties based upon a combination of one or more of the following criteria:

- Formal education
- On-the-job training
- Formal training (vendor courses, site and customer-specific training, etc.)

Training is performance based and proof of successful completion and understanding of the material must be demonstrated and documented. The training and qualification needs of the individual MCLinc staff members are determined by either the TD, or LM. For any new procedure the LM and TD will establish training requirements and have the analyst perform a Demonstration of Capability (DOC). This DOC procedure is explained on a DOC form available from the QAO.

5.1 Training

Personnel shall be in compliance with required training. Formal training classes will be used for the majority of the baseline and/or job-specific required training. MCLinc staff/safety meetings will be used to supplement education in safety- and technical-related issues. Off-site training (vendor schools, short courses, seminars, and conferences) can be used for continuing technical and professional training.

Training for procedure or guideline based methods is performed using required reading assignments and topical review in follow-up staff meetings. On-the-job training can be used to supplement any job performance activity. Details of MCLinc's Training Program are located in "MCL Training Program," MCL-7778.

Training records will be maintained for active laboratory personnel. The current training records for each member of the MCLinc staff will be maintained by the DCC.

New employees receive training in the following areas:

- QA/QC
- Chemical Hygiene Plan

- Health and Safety
- Radiochemistry
- Security

The level of this training will be dependent on employee job assignments, but all training will be documented and placed in their training files.

On-going updated training will be provided to all employees as required. Employees are required to read all SOPs issued to them and ask the QAO any questions or clarifications. On-going SOP training will be provided when significant changes are made to the SOP. The MCLinc staff is encouraged to suggest any training needs they have to better perform their jobs. Also, each year during the annual quality assessment, the need for additional training will be reviewed and the effectiveness of current training evaluated.

5.2 Certification of Qualification

In addition to specific training requirements, there are several areas of MCLinc operations that require special/specific qualifications. These are outlined below. If required by the project, this qualification must be further documented and clearly identify the area of qualification and the basis including, as required, any supporting documentation. See example Certificate of Qualification in Appendix E.

5.3 Instrument Operator Qualifications

Operator status will be determined and confirmed by either the TD or the LM. Appendix D lists the instrumentation which requires approval and the MCLinc personnel that are authorized (as of the date shown) as operators. The QAO is responsible for maintaining and distributing updates for the authorized operator listing.

5.4 Radiological Materials

MCLinc staff members must meet the training and authorization requirements as defined by the RSO. The specific requirements for radiological use authorization are defined in the Tennessee Department of Environment and Conservation, Division of Radiological Health, Application for Radioactive Material License and/or the DOE Radiological Protection Program.

5.5 Classified Materials

MCLinc staff members must meet all of the requirements specified in the "Facility Security Plan," MCL-7706. The most important criteria is the need-to-know. This aspect of control over the unauthorized distribution of classified and controlled information will be fully enforced within all aspects of MCLinc business practice. The FSO will maintain all associated documentation and records that are required for compliance with the Facility Security Plan.

6.0 TECHNICAL PROGRAMS

Since the vast majority of MCLinc's technical programs are non-routine or first time research driven activities, the work is performed based on the work plan or scope of work agreed upon with the client. The guidance to execution of their work is found in the MCLinc SOPs including Project Management and Instrument Operational Guides. The MCLinc President and the QAO must approve MCLinc SOPs. All laboratory work is documented in laboratory notebooks to assure recreation of the process followed. The other various guidelines, procedures, and plans that form the basis for the operations and quality performance of MCLinc are listed in the Reference Section of MCLinc's Controlled Documents, Volumes I, II and III.

6.1 Pre-Project Activities

Consideration will be given to the quality, safety and environmental impacts on project performance during the project conception, bidding, procurement, and initiation phases. These areas will be addressed either informally or formally for all MCLinc work. These issues will be documented when dealing with a customer whose work scope is estimated to take more than 80 man-hours to complete. This consideration will help ensure that all customer and MCLinc data quality objectives can be established and met during the successful completion of the work scope.

6.2 Project Conception

Project ideas will be reviewed by MCLinc staff members to determine if the work being requested or proposed is within the capabilities of the MCLinc staff, facility, and resource allocation. Consideration as to resources, facility requirements, waste generation/disposal, and total project life cycle costs and requirements will be considered. Discrepancies or concerns will be presented to either the TD or LM to obtain resolution on the discrepancies or concerns.

6.3 Bidding

When providing a cost estimate or quote for a specific set of services to a customer, the PM will have the cost estimate reviewed by either the MCLinc President, TD, or LM. The internal cost breakdown analysis will demonstrate that consideration has been

provided to meet the customer's quality, documentation, reporting, sample management, procurement, and waste disposal requirements within the cost estimate being provided.

6.4 Project Acceptance

Before a project is accepted and begun, the PM will meet with the LM and TD to make sure that all required MCLinc resources will be available and can be allocated for the successful completion of the customer work package. This includes a review of any possible procurement of goods and services to complete the project.

6.5 Project Documentation and Communication

At the discretion of the PM and the customer, the amount of project specific QA documentation and procedures will be determined. These documents are used as guidance and can be informally approved and accepted between the PM and the customer. The documents will be part of the permanent project file and are the responsibility of the PM to ensure that all proper documentation is archived. The project specific documentation may include but is not limited to:

- QAP,
- Data package/reporting requirements,
- Specific technical procedures or operational methods,
- Enhanced Chain of Custody procedures,
- Calibration and/or certification requirements,
- Environment, health and safety (EH&S) guidance,
- Budget, schedule, and deadline information, and/or
- Correspondence.

A key element of the MCLinc Project is effective communication to the client not only of the project problems or issues, but progress and significant achievements. This communication also allows an opportunity for input to the project from the client on technical matters, opinions and interpretations of the results. In most cases this input is best received during the project than after the fact. The mode of this communication is best dictated by the client and may mean phone calls, meetings, e-mail or other written progress reports. Document all oral communication to assure your understanding of the discussion.

6.6 Reporting and Project Closure

Report structure, detail, organization, and media selection will be determined by the PM and customer. The PM will ensure that all data reviews, data tabulations, laboratory work, and customer requests have been fully completed and documented prior to the compilation of the final report. Any non-conformity with the customer's request will be communicated and documented as soon as possible with the customer. Documented resolution will be noted within the project notes and summary. The PM will ensure that a complete data set,

laboratory notebook reference list, data location listing, and final copy of the customer report are maintained in their files. A copy of the final customer report will be maintained within the MCLinc project files. If a report is to be amended, the report is either reissued or marked as a new revised version, or a clearly defined amendment to the report is issued. In both cases, the new document is sent to the same distribution as the original report.

Upon completion of the project, the PM must place or reference all applicable documents in the project file, make sure any non-routine or special wastes generated during the project are properly stored and/or disposed per "Waste Management Plan," MCL-7718, and that all samples and residues are properly stored for disposal or returned to the client per the contract.

6.7 Clients Complaints

MCLinc welcomes feedback from our clients, be it positive or negative. Despite the efforts to the contrary, the probability exists that the client may express concerns or disfavor with the project to MCLinc staff. Anyone aware of such a complaint must report it to the appropriate QAO, PM or TD for follow-up. It is critical that MCLinc understand completely both sides of the complaint, the root cause and take immediate corrective action. The complaint will be documented with a nonconformance or corrective action memo per MCL-7722, "Procedure for Reporting Preventive Actions, Problems, Non-conformances, and Associated Actions." The QAO will review this corrective action and discuss with client as necessary. Since the majority of our projects include direct contact with the client, discussions concerning their satisfaction or dissatisfaction with our work can be held one on one with any staff member.

MCLinc President	Date 9/1/15	Section: Document Control Procedures Section No: 7 Revision: 14 Date: 09/01/2015
Quality Assurance Officer	Date 9/1/15	

7. 0 DOCUMENT CONTROL PROCEDURES

7.1 Document Control

Documents will be managed to ensure that a consistent record of activities exists to allow for a detailed review of current practices to determine if any modifications would permit the improvement of any process, in part or in whole. Documents which are determined to be important to the operation and control of materials and information within MCLinc will be controlled. Controlled documents will be maintained with respect to "Document Control," MCL-7703. Examples of controlled documents are:

- QAPs
- Quality Documents prepared for clients
- Standard Operating Procedures
- Chemical Hygiene Plans
- Health and Safety Plans
- Waste Management Plans
- Operator Aids

During the annual internal audit by the QAO, all controlled documents will be reviewed and if revisions are necessary, they will be scheduled and implemented. Those not revised will be marked "Reviewed without Revision with the date" in the Controlled Document Status form.

7.2 Changes to Controlled Documents

Changes to controlled documents may be initiated by anyone using the document to clarify or correct an error or reflect a change in the procedure. Changes shall be reviewed and approved by the same functions that approved the original document. Information needed to evaluate the requested change, if necessary, should be provided along with the MCLinc Change Form (Example in SOP, "Document Control," MCL-7703). The changes shall be noted on the change form and if required for clarity attachment of the revised document pages. Once signature approval is complete the DCC will issue a controlled copy of the change form and any attachments to all recipients of the original controlled document.

7.3 Notebooks

A critical document in use within MCLinc to record day-to-day work efforts, analyses, and experimentation is the laboratory notebook. Laboratory notebooks are issued by the DCC to individual personnel. These notebooks are assigned with a unique identification number and are maintained by the individual MCLinc personnel. The notebook is the responsibility of the individual user. It is good practice to maintain an index in the front of the book to track the time frames associated with various customers and/or projects which have documentation in the notebook. If it is felt that a section or entry into the logbook should be witnessed, the logbook owner is responsible for providing another cognizant MCLinc staff member to read, verify, and sign the logbook pages that the material has been properly documented and dated. Notebooks, when completed or retired, are returned to the DCC for safe storage. Notebook(s) will be randomly reviewed for compliance to the SOP, "Good Notebook Keeping Practices," MCL-7724, during the year as part of each internal assessment by QA or the TD.

8.0 RECORDS

8.1 QA Records

Records that show or demonstrate evidence of quality or a quality system are deemed quality records. They are to be legible, identifiable and retrievable. QA records may be hard copy or electronic media files. Quality records are maintained by the DCC and the QAO and include the following:

- Current and historical controlled documents
- Laboratory notebooks
- Laboratory / instrument logbooks
- Training files/records
- Instrument output, results, notes, design documents and calculations
- Standards traceability documentation
- Radiochemical inventory documentation (maintained by RSO)
- Non-conformance reports
- Demonstration of Capability Form (DOC)

The PM maintains QA Records that are specific to a project such as standard runs, daily calibrations, calculations and results in the project files.

8.2 Project Records

All technical and business records associated with a project constitute Project Records and are maintained accessible to the project staff during the project and are considered client proprietary. The technical records are maintained by the PM in the designated project files and the business records by the DCC in the Administrative Office files. Examples of Project Records are:

- Work plans or scope of work documents
- Project QAPs
- Project correspondence including phone logs
- Interim and final reports
- Computer files of project information
- Proposals, contracts and change orders

Further details on records are outlined in "Quality Assurance Records,"MCL-7729.

8.3 Record Retention

Record retention is the key to assuring our clients that information if needed in the future is retrievable. Project records are maintained for five (5) years or as otherwise defined in the project contract. QA Records not associated with a project are considered lifetime or permanent records and will be maintained for the usable life of the item. All records are maintained within the secure MCLinc facility in clean, dry areas with access controlled by the The Sample/Report Management Staff (SRM). The SRM receives project documents from the MCLinc staff and places the records into appropriate filing cabinets or new storage boxes and logs the contents into a records storage log which is then used to track the documents for future retrieval. All documents in storage are accessible only through the SRM or QAO.

MCLinc President — Date: 9/1/15

Quality Assurance Officer — Date: 9/1/15

9.0 ASSESSMENTS

9.1 Management Assessment

Ultimate responsibility for QA/QC and ES&H compliance within MCLinc rests with the MCLinc President. Unresolved MCLinc issues will be resolved by MCLinc management. MCLinc evaluates its performance in January for the previous year with an Annual Management Quality Assessment in January of each year. During this Annual Management Quality Assessment, issues are raised, resolved and documented. The purpose of the Annual Management Quality Assessment is to provide a means to understand the effectiveness of the management system, make recommendations for improvement to top management and implement the improvements. Tools like the quality policy, client and laboratory QA objectives, performance test (PT) sample results and internal and external assessments are used to allow these improvements while maintaining the integrity of the system.

As part of the MCLinc Annual Management Quality Assessment, the MCLinc management review shall take account of:

- Quality objectives of management met
- The suitability of policies and procedures
- Reports from management/supervisory staff
- Results of internal or third party audits/assessments
- Corrective and preventive actions
- The results of round robin or any PT program
- Changes in volume and type of work
- Client feedback or complaints
- Manpower/equipment needs
- Staff recommendations for improvement
- Other relevant factors such as QC activities resources and staff training

Upon completion of the draft Annual Management Quality Assurance Review, the document is submitted to the MCLinc President/Chief Executive Officer (CEO) for review and determination of any findings. Any findings resulting from this management

review will be defined with a designated person responsible and an agreed upon time schedule.

The management assessment will be documented by the QAO.

9.2 Internal Assessments

The QAO on an annual basis will schedule and initiate at least one internal quality assessment of the internal quality systems of all laboratory operations using a checklist approach and documenting all findings and observations. The Internal Audits/Assessments will be performed by personnel that have no direct responsibility for the activities being performed. Formal internal assessments will be performed using a checklist either AIHA, DOECAP, ASME NQA-1 or one composed by the QAO. The QAO will approve the corrective actions and follow up as necessary to assure corrective actions have been implemented and resolved the issue. The current and future auditors will be trained prior to performing an audit. The training will include review of the purpose of the audit and appropriate checklist and evaluation of any previous findings or observations.

9.3 Independent Assessments

Clients or MCLinc may utilize other organizations, independent of the day-to-day operations of the MCLinc facility, to provide an assessment of quality, safety, and environmental activities within the MCLinc facility. MCLinc will provide a safety orientation to the members of the independent assessment team at the beginning of the assessment kick-off meeting.

All documentation generated by the independent assessment will be addressed in a closeout report that will be generated by the appropriate MCLinc staff no later than twenty-five (25) working days after the independent assessment results are presented to management. Corrective actions will be documented and their effect on the deficiency tracked and noted. If it is felt that the corrective action has had a significant impact on other areas of operation, the corrective action documentation will be used by the appropriate MCLinc staff to compile a positive lesson learned document to ensure that all portions of the MCLinc organization is aware of the potential positive influence of the corrective action. MCLinc may initiate a third party additional audit for specific areas of the laboratory or total laboratory operation.

9.4 Performance Evaluation (PE) and Performance Testing (PT)

MCLinc will participate in PE and PT programs as necessary to evaluate the quality performance of the laboratory. MCLinc currently participates in Mixed Analyte Performance Evaluation Program (MAPEP) (Inorganic and Rad - soil and water); AIHA for metals, air asbestos, beryllium oxide, and bulk asbestos; internal PEs for hexavalent chromium and mercury; and a third-party asbestos program. Others will be added as needed. Any non-passing score in these programs will be investigated and a written report submitted to the QAO within 21 calendar days. Supplemental PE samples are hex-chrome in water and PCBs in oil, both bi-annual. The QAO will approve and follow-up on the corrective actions as needed.

10.0 REFERENCES

ANSI/ASQC E4-1994, "Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs."

ASME NQA-1-2000, Edition, "Quality Assurance Program Requirements for Nuclear Facilities."

DOE, "Consolidated Audit Program Quality Systems for Analytical Services Revision 2.9."

Energy, Nuclear Safety Management, Quality Assurance Requirements, Scope, 10 CFR Part 830.120.

Energy Reporting Defects and Noncompliance, 10 CFR Part 21.

ISO/IEC Standard 17025 – "General Requirements for the Competence of Testing and Calibration Laboratories, 2005."

MCL-7702, "Chemical Hygiene Plan."

MCL-7703, "Document Control."

MCL-7704, "Project Management Guide."

MCL-7705, "Nuclear Materials Control and Accountability Plan."

MCL-7706, "Facility Security Plan."

MCL-7708, "Electron Microscopy Operation Guide."

MCL-7710, "Sample Preparation Guide."

MCL-7711, "Calibration, Inspection, and Maintenance Guide."

MCL-7715, "Radiation Protection Plan."

MCL-7717, "Health and Safety Plan."

MCL-7718, "Waste Management Plan."

MCL-7719, "Asbestos Laboratory Quality Assurance Manual."

MCL-7722, "Procedure for Reporting Problems, Non-Conformances, and Associated Actions."

MCL-7724, "Good Notebook Keeping Practices."

MCL-7727, "Procurement Control."

MCL-7728, "Verification of Data Software."

MCL-7729, "Quality Assurance Records."

MCL-7735, "Estimation of Uncertainty of Measurement (EUM)."

MCL-7756, "Operator Aids."

MCLinc's Controlled Documents, Volumes, I, II, and III.
"National Environmental Laboratory Accreditation Conference Standards," Latest Approved Edition

MCLinc President — Date: 9/1/15

Quality Assurance Officer — Date: 9/1/15

Section: Appendices
Section No: 11
Revision: 14
Date: 09/01/2015

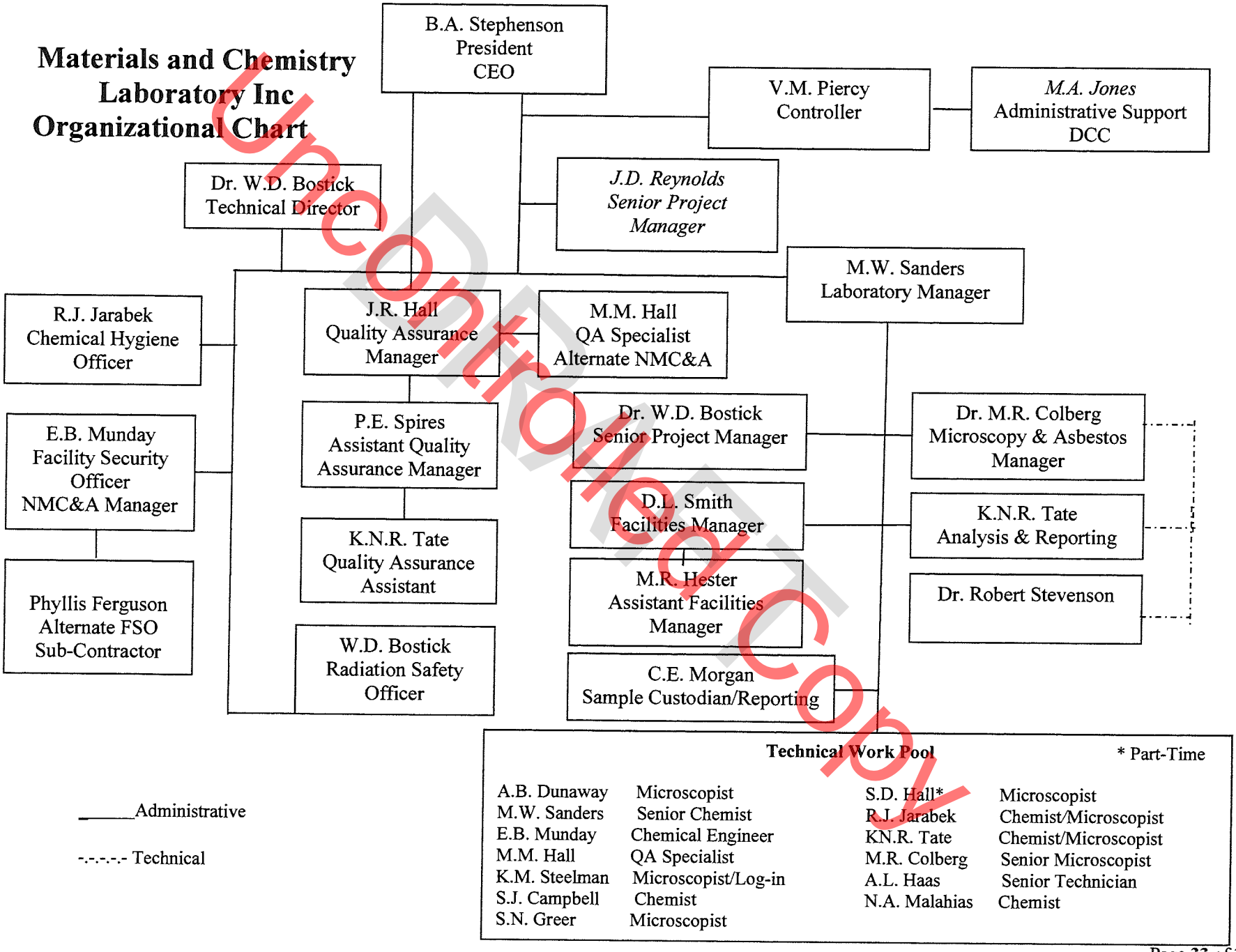
Appendix A - MCLinc QAP Cross-reference to National and International Quality Requirements

Basic Requirements of NQA-1	Requirements of ISO/IEC 17025	MCLinc QAP Section	10 CFR 830.120	ANSI/ASQC E4-1994
Organization	Organization and Management	1.0 Organization	Management	Management and Organization
Quality Assurance Program	Management System Personnel	2.0 Management Systems 5.0 Personnel Training and Qualifications	Quality Assurance Program; Personnel Training and Qualifications	Quality Systems and Description; Personnel Qualifications and Training
Design Control		2.13 Data Review and Evaluation	Design	Design
Procurement Document Control	Review of Requests, Tenders and Contracts	3.0 Procurement, Subcontracting and Documentation	Procurement	Procurement; Planning and Scoping
Instructions, Procedures, and Drawings	Technical Requirements	6.0 Technical Programs	Documents and Records	Documents and Records; Design of Systems
Document Control	Document Control	7.0 Document Control	Documents and Records	Documents and Records
Control of Purchased Items and Services	Purchasing Services and Supplies	3.1 Procurement	Procurement	Procurement
Identification and Control of Items	Measurement Traceability	2.1 Standards and Reference Materials	Procurement	Procurement
Control of Processes	Accommodation and Environmental Conditions	2.0 Quality Systems	Performance-Work Processes	Implement Work Processes; Operation of Systems
Inspection	Subcontracting of Tests and Calibrations	2.12 Data Review 3.2 Subcontracting 9.2 Internal Assessments	Inspection and Acceptance Testing	Computer Hardware/Software; Implementation of Planned Operations
Test Control	Assuring the Quality of Test and Calibration Results	2.2 Calibration	Quality Improvement	Quality Improvement
Control of Measuring and Test Equipment	Test and Calibrations Methods and Method Validation Equipment	2.2 Calibration 2.11 Instrumentation	Quality Assurance Criteria	Quality Systems
------	Service to the Client; Complaints	6.0 Technical Programs	Quality Assurance Criteria	Planning and Scoping
Handling, Storage, and Shipping	Sampling	2.10 Sampling and Sample Preparation	------	Design of Data Collection
Inspection, Test and Operating Status	Handling of Test and Calibration Items	2.0 Quality Systems	Inspection and Acceptance Testing	Design of Data Collection/Verification and Acceptance
Control of Non-Conforming Items	Control of Non-conforming Testing and/or Calibration Work	4.0 Non-conformances, Corrective and Preventative Actions	Quality Improvement	Quality Improvement
Corrective Action	Corrective Action Preventative Action	4.0 Non-conformances, Corrective and Preventative Actions	Quality Improvement	Quality Improvement
Quality Assurance Records	Reporting of Results Control of Records	6.6 Reporting 8.0 Records	Documents and Records	Documents and Records
Audits	Internal Audits Management Reviews	9.1 Management Assessment 9.2 Internal Assessments 9.3 Independent Assessments	Management Assessment; Independent Assessment	Assessment and Response

Appendix A Cross Reference (Continued)

DOE Quality Systems for Analytical Services Latest Revision		MCLinc Quality Assurance Plan Revision *14*	
Section	**Title**	**Section**	**Title**
1.0	Introduction, Scope, Applicability	1.1	Introduction
2.0	References	10.0	References
3.0	Terms and Definitions		Defined throughout QAP
4.0	Management Requirements	1.0 2.0	Organization Management Systems
4.1	Organization	1.0	Organization
4.2	Management	2.0	Management Systems
4.3	Document Control	7.1	Document Control
4.4	Review of Requests, Tenders and Contracts	6.3	Bidding
4.5	Subcontracting of Environmental Tests	3.2	Subcontracting
4.6	Purchasing Services and Supplies	3.1	Procurement
4.7	Service to the Client	6.5	Project Documentation and Communication
4.8	Complaints	6.7	Client Complaints
4.9	Control of Nonconforming Environmental Testing Work	4.0	Non-Conformances and Correction Preventative Action
4.10 4.11	Improvement Corrective Action	4.0	Non-Conformances and Correction Preventative Action
4.12	Preventive Action	4.0	Non-Conformances and Correction Preventative Action
4.13	Control of Records	8.0	Records
4.14	Internal Audits	9.2	Internal Assessments
4.16 4.17	Management Reviews Data Integrity Investigations	9.1	Management Assessments
5.0	Technical Requirements	2.0	Management Systems
5.1	General	2.0	Management Systems
5.2	Personnel	5.0	Personnel Training and Qualification
5.3	Accommodation and Environmental Conditions	2.4	Work Environment
5.4	Environmental Test Methods and Method Evaluation	2.14	Standard Operating Procedures
5.5	Calibration Requirements	2.11	Instrumentation and Maintenance
5.6	Measurement Traceability	2.1 2.5	Standards and Reference Materials Laboratory Supplies
5.7-5.8	Sampling and Handling of Samples	2.7 2.8 2.10	Material and Sample Receipt Controlled Samples Sampling and Sample Preparation
5.9	Quality of Environmental Test	2.12 2.13	Quality Control Samples and Assessments of Data Data Review and Evaluation
5.10	Reporting the Results	6.6	Reporting and Project Closure
6.0	Hazardous and Radioactive Materials Management and Health and Safety Practices		Various Locations and Two Separate SOPs (MCL-7718;& MCL-7717; and MCL-7715)
6.1	Radioactive Materials Management and Control	5.4	Radiological Materials and Radiation Protection Plan, MCL 7715 SOP
6.2	TSCA [Toxic Substance Control Act of 1976] Materials		Chemical Hygiene Plan MCL 7702 SOP
6.3	Laboratory Health and Safety		Health and Safety Plan MCL 7717 SOP
6.4	Waste Management and Disposal		Waste Management PIan MCL 7718 SOP

Materials and Chemistry Laboratory Inc Organizational Chart



Appendix C - MCL Support Function Assignments

Position	Personnel
Company Controller	V. Monique Piercy
Chemical Hygiene Officer	Robert J. Jarabek
Classified AIS Security Site Manager	Mark R. Colberg
Classified AIS System Security Officer	Earl B. Munday with Charlie Coffey
Classified Document Custodian	Earl B. Munday
Classified Document Custodian - Alternate	Robert J. Jarabek
Document Control Coordinator	Linda E. Maines
Facility Security Officer	Earl B. Munday
Facility Security Officer - Support	Atkins Nuclear Solutions US
MBA Custodian(s)	Robert J. Jarabek Earl B. Munday
NMC&A Manager	Earl B. Munday
NMC&A Alternate Manager	Mary M. Hall
OPSEC Manager	Phyllis Ferguson
OPSEC Alternate Manager	Earl B. Munday
MCLinc President	Barry A. Stephenson
Laboratory Manager	Michele W. Sanders
QA Officer	Jack R. Hall
QA Officer-Alternate	Mary M. Hall/*Preston E. Spires*
Radiological Safety Officer	*William D. Bostick*
Radiological Safety Officer - Alternate	Michele Sanders
Security Container #1 Custodian	Earl B. Munday
Security Container #1 Custodian - Alternate	Mark R. Colberg
Security Container #2 Custodian	Earl B. Munday
Security Container #2 Custodian - Alternate	Robert J. Jarabek
Security Container #3 Custodian	Mark R. Colberg
Security Container #3 Custodian - Alternate	Earl B. Munday
Technical Director	William D. Bostick

Appendix D – Instrumentation with Responsible Owner and Authorized Operator

Type	Model	Manufacturer	MCLinc Owner
FTIR	MB100	BOMEN	Munday
FTIR	GL3020/Nicolet (6400)	Mattson	Bostick/Tate
GC/*EC*	5890 (2), 7890A	Hewlett Packard	*Campbell*/Sanders /Tate
IC – Hex Cr	ICS1100	Dionex	*Lopez*/Sanders
IC - Anions	ICS-1100	Thermal Dionix	Sanders/*Malahias*
ICP-OES	2000	Perkin Elmer	Sanders/Jarabek
ICP-OES	AVIO	Perkin Elmer	*Campbell*
ICP/MS	Elan 9000	Perkin Elmer	Sanders
Mercury Analyzer (AA Cold Vapor)	410	Buck	*Campbell/Lopez*
Mercury Analyzer (Low Level)	Hydro C	Teledyne-Lehman	
Mercury Analyzer (Low Level)	Hydro II AF GOLD	Teledyne-Lehman	
Optical Microscope	Various	Various	Colberg/Jarabek/D. Hall/Tate Steelman
Rad Spectroscopy	Various	Various	Jarabek/Bostick
SEM	840	JEOL	Colberg/Dunaway
TEM	H600	Hitachi	Colberg/Dunaway
SEM	5800	Joel	Colberg/Dunaway
TEM	2010	JEOL	Colberg/Dunaway
UV-Vis Spectroscopy	PC1000	Ocean Optics	Bostick
XRD	MiniFlex II	Rigaku	Colberg/Dunaway/Haas*
XRD	MiniFlex 6A	Rigaku	Colberg/Dunaway/Haas*
TGA/DTA+MS+GC	6300 ThermoStar GSD301/8610C	SII/Pfeiffer/SRI	Sanders/*Tate*

AA-Atomic Absorption
DTA – Date Transfer Analyzer
EC – Electron Capture
FTIR – Fourier Transform Infrared Spectroscopy
IC – Ion Chromatography
MS – Mass Spectroscopy
TEM – Transmission Electron Microscope
UV-Vis – Ultraviolet – Visible

*In training

FID – Flame Ionization Detector
GC – Gas Chromatography
ICP – Inductively Coupled Plasma
SEM – Scanning Electron Microscope
TGA – Thermogravametric Analysis
XRD – X-Ray Defraction

Appendix E - CERTIFICATE OF QUALIFICATION AND AUTHORIZATION

MCLinc CERTIFICATE OF QUALIFICATION

Certification of: _____

Certified To Perform: _____

_____

_____

Certification based on:

- ❑ Education
- ❑ Indoctrination
- ❑ Experience
- ❑ Training
- ❑ Test Results (Attach)
- ❑ Capability Demonstration:

  (Observed by: _____)

Certification Level (I, II, III, per NQA-1): _____

Technical Director/QAO Approval: _____

Date of Certification: _____

Expiration Date: _____

Results of Periodic Evaluation: _____

_____

_____

Appendix F

MEMO

DATE: December 30, 2014

TO: Barry A. Stephenson, MCLinc Staff

FROM: Jack R. Hall

FAX#: (865) 576-8558 PHONE: (865) 574-9923

SUBJECT: McLinc QA Internal Assessment Schedule for 2015

MEMO:

As part of the MCLinc QA Plan there is a requirement for an annual assessment, which I am scheduling to occur over the year covering the various quality systems. The schedule is as follows:

January/February	Complete 2014 Management Assessment /Radiation Plan Review-Assessment
March/April	Project files, QA files, and Training files
May/June	Review QAP/ Corrective/Preventative Action Process
July/August	Instrument/Equipment Calibration/Reference Materials + Update QAP
September/October	Sample Log-in Process/ Random Notebook Review
November	Industrial Hygiene Laboratory Internal Assessment per AIHA
December	Procurement and Document Control and perform QA SOPs Review

As part of these quality systems assessments I will be asking for your help to take any needed corrective actions.

Materials and Chemistry Laboratory, Inc.
East Tennessee Technology Park, Building K-1006
2010 Highway 58, Suite 1000, Oak Ridge, Tennessee 37830-1702
Phone: (865) 576-4138 Fax: (865) 576-8558

ATTACHMENT A-5

KANSAS DEPARTMENT OF HEALTH & ENVIRONMENT ACCREDITATION
PACE ANALYTICAL SERVICES, LLC-INDIANAPOLIS

Division of Environment
Kansas Health and Environmental Laboratories
Environmental Laboratory Improvement Program
6810 SE Dwight Street
Topeka, KS 66620-0001

Phone: 785-296-3811
Fax: 785-559-5207
KDHE.ELIPO@KS.GOV
www.kdheks.gov/envlab

Lee A. Norman, M.D., Interim Secretary

Department of Health & Environment

Laura Kelly, Governor

The Kansas Department of Health and Environment encourages all clients and data users to verify the most current scope of accreditation for certification number E-10177

The analytes tested and the corresponding matrix and method which a laboratory is authorized to perform at any given time will be those indicated in the most recently issued scope of accreditation. The most recent scope of accreditation supersedes all previously issued scopes of accreditation. It is the certified laboratory's responsibility to review this document for any discrepancies. This scope of accreditation will be recalled in the event that your laboratory's certification is revoked.

Accreditation Start: 1/18/2019 Accreditation End: 4/30/2019

EPA Number: ***IN00043*** **Scope of Accreditation for Certification Number:** **E-10177**

Pace Analytical Services, LLC - Indianapolis IN

Primary AB

Program/Matrix: ***CWA (Non Potable Water)***

Method ASTM D516-07	
Sulfate	KS
Method ASTM D516-11	
Sulfate	KS
Method EPA 120.1	
Conductivity	KS
Method EPA 1631E	
Mercury	KS
Method EPA 1664A	
Oil & Grease	KS
Method EPA 180.1	
Turbidity	KS
Method EPA 200.7	
Aluminum	KS
Antimony	KS
Arsenic	KS
Barium	KS
Beryllium	KS
Boron	KS
Cadmium	KS
Calcium	KS
Chromium	KS
Cobalt	KS
Copper	KS
Iron	KS
Lead	KS

Pace Analytical Services, LLC - Indianapolis IN **Primary AB**

Program/Matrix: ***CWA (Non Potable Water)***

Magnesium	KS
Manganese	KS
Molybdenum	KS
Nickel	KS
Potassium	KS
Selenium	KS
Silver	KS
Sodium	KS
Strontium	KS
Thallium	KS
Tin	KS
Titanium	KS
Vanadium	KS
Zinc	KS

Method EPA 200.8

Aluminum	KS
Antimony	KS
Arsenic	KS
Barium	KS
Beryllium	KS
Boron	KS
Cadmium	KS
Chromium	KS
Cobalt	KS
Copper	KS
Lead	KS
Manganese	KS
Molybdenum	KS
Nickel	KS
Selenium	KS
Silver	KS
Thallium	KS
Tin	KS
Titanium	KS
Vanadium	KS
Zinc	KS

Method EPA 245.1

Mercury	KS

Method EPA 300.0

Bromide	KS
Chloride	KS
Fluoride	KS
Nitrate	KS
Nitrate-nitrite	KS
Nitrite	KS
Sulfate	KS

Pace Analytical Services, LLC - Indianapolis IN **Primary AB**

Program/Matrix: ***CWA (Non Potable Water)***

Method EPA 335.4	
Cyanide	KS
Method EPA 350.1	
Ammonia as N	KS
Method EPA 351.2	
Total Kjeldahl Nitrogen (TKN)	KS
Method EPA 351.2 minus EPA 350.1	
Organic nitrogen	KS
Method EPA 353.2	
Nitrate	KS
Nitrate-nitrite	KS
Nitrite	KS
Method EPA 365.1	
Phosphorus	KS
Method EPA 410.4	
Chemical oxygen demand	KS
Method EPA 420.4	
Total phenolics	KS
Method EPA 6010B	
Arsenic	KS
Cadmium	KS
Copper	KS
Lead	KS
Lithium	KS
Molybdenum	KS
Nickel	KS
Selenium	KS
Strontium	KS
Total chromium	KS
Zinc	KS
Method EPA 6020	
Arsenic	KS
Cadmium	KS
Copper	KS
Lead	KS
Molybdenum	KS
Nickel	KS
Selenium	KS
Total chromium	KS
Zinc	KS
Method EPA 608.3 GC-ECD	
4,4'-DDD	KS
4,4'-DDE	KS
4,4'-DDT	KS

Program/Matrix: ***CWA (Non Potable Water)***

Aldrin	KS
alpha-BHC (alpha-Hexachlorocyclohexane)	KS
Aroclor-1016 (PCB-1016)	KS
Aroclor-1221 (PCB-1221)	KS
Aroclor-1232 (PCB-1232)	KS
Aroclor-1242 (PCB-1242)	KS
Aroclor-1248 (PCB-1248)	KS
Aroclor-1254 (PCB-1254)	KS
Aroclor-1260 (PCB-1260)	KS
beta-BHC (beta-Hexachlorocyclohexane)	KS
Chlordane (tech.)(N.O.S.)	KS
delta-BHC	KS
Dieldrin	KS
Endosulfan I	KS
Endosulfan II	KS
Endosulfan sulfate	KS
Endrin	KS
Endrin aldehyde	KS
gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	KS
Heptachlor	KS
Heptachlor epoxide	KS
Methoxychlor	KS
Toxaphene (Chlorinated camphene)	KS

Method EPA 624.1

1,1,1-Trichloroethane	KS
1,1,2,2-Tetrachloroethane	KS
1,1,2-Trichloroethane	KS
1,1-Dichloroethane	KS
1,1-Dichloroethylene	KS
1,2-Dichlorobenzene (o-Dichlorobenzene)	KS
1,2-Dichloroethane (Ethylene dichloride)	KS
1,2-Dichloropropane	KS
1,3-Dichlorobenzene	KS
1,4-Dichlorobenzene	KS
2-Chloroethyl vinyl ether	KS
Acrolein (Propenal)	KS
Acrylonitrile	KS
Benzene	KS
Bromodichloromethane	KS
Bromoform	KS
Carbon tetrachloride	KS
Chlorobenzene	KS
Chlorodibromomethane	KS
Chloroethane (Ethyl chloride)	KS
Chloroform	KS
cis-1,3-Dichloropropene	KS

Program/Matrix: ***CWA (Non Potable Water)***

Ethylbenzene	KS
Methyl bromide (Bromomethane)	KS
Methyl chloride (Chloromethane)	KS
Methylene chloride (Dichloromethane)	KS
Naphthalene	KS
Tetrachloroethylene (Perchloroethylene)	KS
Toluene	KS
trans-1,2-Dichloroethylene	KS
trans-1,3-Dichloropropylene	KS
Trichloroethene (Trichloroethylene)	KS
Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	KS
Vinyl chloride	KS
Xylene (total)	KS

Method EPA 625.1

1,2,4-Trichlorobenzene	KS
1,2-Dichlorobenzene (o-Dichlorobenzene)	KS
1,3-Dichlorobenzene	KS
1,4-Dichlorobenzene	KS
2,2'-Oxybis(1-chloropropane), bis(2-Chloro-1-methylethyl)ether	KS
2,4,6-Trichlorophenol	KS
2,4-Dichlorophenol	KS
2,4-Dimethylphenol	KS
2,4-Dinitrophenol	KS
2,4-Dinitrotoluene (2,4-DNT)	KS
2,6-Dinitrotoluene (2,6-DNT)	KS
2-Chloronaphthalene	KS
2-Chlorophenol	KS
2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	KS
2-Nitrophenol	KS
3,3'-Dichlorobenzidine	KS
4-Bromophenyl phenyl ether	KS
4-Chloro-3-methylphenol	KS
4-Chlorophenyl phenylether	KS
4-Nitrophenol	KS
Acenaphthene	KS
Acenaphthylene	KS
Anthracene	KS
Benzidine	KS
Benzo(a)anthracene	KS
Benzo(a)pyrene	KS
Benzo(b)fluoranthene	KS
Benzo(g,h,i)perylene	KS
Benzo(k)fluoranthene	KS
bis(2-Chloroethoxy)methane	KS
bis(2-Chloroethyl) ether	KS
Butyl benzyl phthalate	KS

Program/Matrix: ***CWA (Non Potable Water)***

Chrysene	KS
Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	KS
Dibenz(a,h) anthracene	KS
Diethyl phthalate	KS
Dimethyl phthalate	KS
Di-n-butyl phthalate	KS
Di-n-octyl phthalate	KS
Fluoranthene	KS
Fluorene	KS
Hexachlorobenzene	KS
Hexachlorobutadiene	KS
Hexachloroethane	KS
Indeno(1,2,3-cd) pyrene	KS
Isophorone	KS
Naphthalene	KS
Nitrobenzene	KS
n-Nitrosodimethylamine	KS
n-Nitrosodi-n-propylamine	KS
n-Nitrosodiphenylamine	KS
Pentachlorophenol	KS
Phenanthrene	KS
Phenol	KS
Pyrene	KS
Method EPA 7470A	
Mercury	KS
Method EPA 7471A	
Mercury	KS
Method EPA 8015D	
Propylene glycol	KS
Method EPA 8260C	
1,1,2-Trichloro-1,2,2-trifluoroethane	KS
1,3,5-Trichlorobenzene	KS
Method EPA 8270C	
1-Methylnaphthalene	KS
Carbazole	KS
Method EPA RSK-175 (GC/FID)	
Ethane	KS
Ethene	KS
Methane	KS
Method SM 2310 B-2011	
Acidity, as CaCO3	KS
Method SM 2320 B-1997	
Alkalinity as CaCO3	KS
Method SM 2320 B-2011	
Alkalinity as CaCO3	KS

Pace Analytical Services, LLC - Indianapolis IN **Primary AB**

Program/Matrix: ***CWA (Non Potable Water)***

Method SM 2340 B-1997	
Hardness	KS
Method SM 2340 B-2011	
Hardness	KS
Method SM 2540 B-2011	
Residue-total	KS
Method SM 2540 C-2011	
Residue-filterable (TDS)	KS
Method SM 2540 D-2011	
Residue-nonfilterable (TSS)	KS
Method SM 2540 F-2011	
Residue-settleable	KS
Method SM 3500-Cr B-2011	
Chromium VI	KS
Method SM 4500-Cl G-2000	
Total residual chlorine	KS
Method SM 4500-Cl G-2011	
Total residual chlorine	KS
Method SM 4500-Cl^- E-1997	
Chloride	KS
Method SM 4500-Cl^- E-2011	
Chloride	KS
Method SM 4500-CN^- C-2011	
Cyanide	KS
Method SM 4500-CN^- E-2011	
Cyanide	KS
Method SM 4500-CN^- G-2011	
Amenable cyanide	KS
Method SM 4500-F^- C-1997	
Fluoride	KS
Method SM 4500-F^- C-2011	
Fluoride	KS
Method SM 4500-H+ B-2000	
pH	KS
Method SM 4500-H+ B-2011	
pH	KS
Method SM 4500-NH3 G-1997	
Ammonia as N	KS
Method SM 4500-NH3 G-2011	
Ammonia as N	KS
Method SM 4500-P E-1999	
Orthophosphate as P	KS

Pace Analytical Services, LLC - Indianapolis IN **Primary AB**

Program/Matrix: ***CWA (Non Potable Water)***

Phosphorus	KS
Method SM 4500-P E-2011	
Orthophosphate as P	KS
Phosphorus	KS
Method SM 4500-$S2^{-}$ D-2000	
Sulfide	KS
Method SM 5210 B-2011	
Biochemical oxygen demand	KS
Carbonaceous BOD, CBOD	KS
Method SM 5310 C-2011	
Total organic carbon	KS
Method SM 5540 C-2011	
Surfactants - MBAS	KS
Method TKN-NH3-CAL	
Organic nitrogen	KS

Pace Analytical Services, LLC - Indianapolis IN **Primary AB**

Program/Matrix: ***RCRA (Non Potable Water)***

Method EPA 1010A	
Ignitability	KS
Method EPA 1311	
Toxicity Characteristic Leaching Procedure (TCLP)	KS
Method EPA 1312	
Synthetic Precipitation Leaching Procedure (SCLP)	KS
Method EPA 6010B	
Aluminum	KS
Antimony	KS
Arsenic	KS
Barium	KS
Beryllium	KS
Boron	KS
Cadmium	KS
Calcium	KS
Chromium	KS
Cobalt	KS
Copper	KS
Iron	KS
Lead	KS
Magnesium	KS
Manganese	KS
Molybdenum	KS
Nickel	KS
Potassium	KS
Selenium	KS
Silver	KS
Sodium	KS
Strontium	KS
Thallium	KS
Tin	KS
Titanium	KS
Vanadium	KS
Zinc	KS
Method EPA 6020	
Aluminum	KS
Antimony	KS
Arsenic	KS
Barium	KS
Beryllium	KS
Cadmium	KS
Chromium	KS
Cobalt	KS
Copper	KS
Lead	KS
Manganese	KS

Program/Matrix: ***RCRA (Non Potable Water)***

Nickel	KS
Selenium	KS
Silver	KS
Thallium	KS
Vanadium	KS
Zinc	KS
Method EPA 7196A	
Chromium VI	KS
Method EPA 7470A	
Mercury	KS
Method EPA 7471A	
Mercury	KS
Method EPA 8011	
1,2-Dibromo-3-chloropropane (DBCP)	KS
1,2-Dibromoethane (EDB, Ethylene dibromide)	KS
Method EPA 8015D	
Diesel range organics (DRO)	KS
Ethanol	KS
Ethylene glycol	KS
Gasoline range organics (GRO)	KS
Isobutyl alcohol (2-Methyl-1-propanol)	KS
Isopropyl alcohol (2-Propanol, Isopropanol)	KS
Methanol	KS
n-Butyl alcohol (1-Butanol, n-Butanol)	KS
n-Propanol (1-Propanol)	KS
Propylene glycol	KS
Method EPA 8081B	
4,4'-DDD	KS
4,4'-DDE	KS
4,4'-DDT	KS
Aldrin	KS
alpha-BHC (alpha-Hexachlorocyclohexane)	KS
alpha-Chlordane, cis-Chlordane	KS
beta-BHC (beta-Hexachlorocyclohexane)	KS
Chlordane (tech.)(N.O.S.)	KS
delta-BHC	KS
Dieldrin	KS
Endosulfan I	KS
Endosulfan II	KS
Endosulfan sulfate	KS
Endrin	KS
Endrin aldehyde	KS
Endrin ketone	KS
gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	KS
gamma-Chlordane	KS

Program/Matrix: ***RCRA (Non Potable Water)***

Heptachlor	KS
Heptachlor epoxide	KS
Methoxychlor	KS
Toxaphene (Chlorinated camphene)	KS

Method EPA 8082A

Aroclor-1016 (PCB-1016)	KS
Aroclor-1221 (PCB-1221)	KS
Aroclor-1232 (PCB-1232)	KS
Aroclor-1242 (PCB-1242)	KS
Aroclor-1248 (PCB-1248)	KS
Aroclor-1254 (PCB-1254)	KS
Aroclor-1260 (PCB-1260)	KS

Method EPA 8141B

Atrazine	KS
Azinphos-methyl (Guthion)	KS
Chlorpyrifos	KS
Chlorpyrifos-methyl	KS
Demeton-o	KS
Demeton-s	KS
Diazinon	KS
Dichlorovos (DDVP, Dichlorvos)	KS
Dimethoate	KS
Disulfoton	KS
Famphur	KS
Malathion	KS
Merphos	KS
Methyl parathion (Parathion, methyl)	KS
Naled	KS
Parathion, ethyl	KS
Phorate	KS
Ronnel	KS
Simazine	KS
Terbufos	KS
Tetrachlorvinphos (Stirophos, Gardona) E-isomer	KS

Method EPA 8151A

2,4,5-T	KS
2,4-D	KS
2,4-DB	KS
3,5-Dichlorobenzoic acid	KS
Acifluorfen	KS
Bentazon	KS
Chloramben	KS
Dalapon	KS
DCPA di acid degradate	KS
Dicamba	KS
Dichloroprop (Dichlorprop)	KS

Program/Matrix: ***RCRA (Non Potable Water)***

Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	KS
MCPA	KS
MCPP	KS
Pentachlorophenol	KS
Picloram	KS
Silvex (2,4,5-TP)	KS

Method EPA 8260C

1,1,1,2-Tetrachloroethane	KS
1,1,1-Trichloroethane	KS
1,1,2,2-Tetrachloroethane	KS
1,1,2-Trichloro-1,2,2-trifluoroethane	KS
1,1,2-Trichloroethane	KS
1,1-Dichloroethane	KS
1,1-Dichloroethylene	KS
1,1-Dichloropropene	KS
1,2,3-Trichlorobenzene	KS
1,2,3-Trichloropropane	KS
1,2,4-Trichlorobenzene	KS
1,2,4-Trimethylbenzene	KS
1,2-Dibromo-3-chloropropane (DBCP)	KS
1,2-Dibromoethane (EDB, Ethylene dibromide)	KS
1,2-Dichlorobenzene (o-Dichlorobenzene)	KS
1,2-Dichloroethane (Ethylene dichloride)	KS
1,2-Dichloropropane	KS
1,3,5-Trichlorobenzene	KS
1,3,5-Trimethylbenzene	KS
1,3-Dichlorobenzene	KS
1,3-Dichloropropane	KS
1,4-Dichlorobenzene	KS
1,4-Dioxane (1,4- Diethyleneoxide)	KS
2,2-Dichloropropane	KS
2-Butanone (Methyl ethyl ketone, MEK)	KS
2-Chloroethyl vinyl ether	KS
2-Chlorotoluene	KS
2-Hexanone	KS
4-Chlorotoluene	KS
4-Isopropyltoluene (p-Cymene,p-Isopropyltoluene)	KS
4-Methyl-2-pentanone (MIBK)	KS
Acetone	KS
Acetonitrile	KS
Acrolein (Propenal)	KS
Acrylonitrile	KS
Allyl chloride (3-Chloropropene)	KS
Benzene	KS
Bromobenzene	KS
Bromochloromethane	KS

Program/Matrix: ***RCRA (Non Potable Water)***

Bromodichloromethane	KS
Bromoform	KS
Carbon disulfide	KS
Carbon tetrachloride	KS
Chlorobenzene	KS
Chlorodibromomethane	KS
Chloroethane (Ethyl chloride)	KS
Chloroform	KS
cis-1,2-Dichloroethylene	KS
cis-1,3-Dichloropropene	KS
Dibromomethane (Methylene bromide)	KS
Dichlorodifluoromethane (Freon-12)	KS
Diethyl ether	KS
Ethyl acetate	KS
Ethyl methacrylate	KS
Ethylbenzene	KS
Hexachlorobutadiene	KS
Iodomethane (Methyl iodide)	KS
Isopropylbenzene	KS
Methacrylonitrile	KS
Methyl bromide (Bromomethane)	KS
Methyl chloride (Chloromethane)	KS
Methyl methacrylate	KS
Methyl tert-butyl ether (MTBE)	KS
Methylene chloride (Dichloromethane)	KS
m-Xylene	KS
Naphthalene	KS
n-Butyl alcohol (1-Butanol, n-Butanol)	KS
n-Butylbenzene	KS
n-Propylbenzene	KS
o-Xylene	KS
Propionitrile (Ethyl cyanide)	KS
p-Xylene	KS
sec-Butylbenzene	KS
Styrene	KS
tert-Butyl alcohol	KS
tert-Butylbenzene	KS
Tetrachloroethylene (Perchloroethylene)	KS
Toluene	KS
trans-1,2-Dichloroethylene	KS
trans-1,3-Dichloropropylene	KS
trans-1,4-Dichloro-2-butene	KS
Trichloroethene (Trichloroethylene)	KS
Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	KS
Vinyl acetate	KS
Vinyl chloride	KS
Xylene (total)	KS

Program/Matrix: ***RCRA (Non Potable Water)***

Method **EPA 8270C**

1,2,4,5-Tetrachlorobenzene	KS
1,2,4-Trichlorobenzene	KS
1,2-Dichlorobenzene (o-Dichlorobenzene)	KS
1,2-Diphenylhydrazine	KS
1,3-Dichlorobenzene	KS
1,3-Dinitrobenzene (1,3-DNB)	KS
1,4-Dichlorobenzene	KS
1,4-Naphthoquinone	KS
1,4-Phenylenediamine	KS
1-Methylnaphthalene	KS
1-Naphthylamine	KS
2,2'-Oxybis(1-chloropropane), bis(2-Chloro-1-methylethyl)ether	KS
2,3,4,6-Tetrachlorophenol	KS
2,4,5-Trichlorophenol	KS
2,4,6-Trichlorophenol	KS
2,4-Dichlorophenol	KS
2,4-Dimethylphenol	KS
2,4-Dinitrophenol	KS
2,4-Dinitrotoluene (2,4-DNT)	KS
2,6-Dichlorophenol	KS
2,6-Dinitrotoluene (2,6-DNT)	KS
2-Acetylaminofluorene	KS
2-Chloronaphthalene	KS
2-Chlorophenol	KS
2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	KS
2-Methylaniline (o-Toluidine)	KS
2-Methylnaphthalene	KS
2-Methylphenol (o-Cresol)	KS
2-Naphthylamine	KS
2-Nitroaniline	KS
2-Nitrophenol	KS
2-Picoline (2-Methylpyridine)	KS
3,3'-Dichlorobenzidine	KS
3,3'-Dimethylbenzidine	KS
3-Methylcholanthrene	KS
3-Methylphenol (m-Cresol)	KS
3-Nitroaniline	KS
4-Aminobiphenyl	KS
4-Bromophenyl phenyl ether	KS
4-Chloro-3-methylphenol	KS
4-Chloroaniline	KS
4-Chlorophenyl phenylether	KS
4-Dimethyl aminoazobenzene	KS
4-Methylphenol (p-Cresol)	KS
4-Nitroaniline	KS

Program/Matrix: ***RCRA (Non Potable Water)***

4-Nitrophenol	KS
4-Nitroquinoline 1-oxide	KS
5-Nitro-o-toluidine	KS
7,12-Dimethylbenz(a) anthracene	KS
a-a-Dimethylphenethylamine	KS
Acenaphthene	KS
Acenaphthylene	KS
Acetophenone	KS
Aniline	KS
Anthracene	KS
Aramite	KS
Benzidine	KS
Benzo(a)anthracene	KS
Benzo(a)pyrene	KS
Benzo(b)fluoranthene	KS
Benzo(g,h,i)perylene	KS
Benzo(k)fluoranthene	KS
Benzoic acid	KS
Benzyl alcohol	KS
bis(2-Chloroethoxy)methane	KS
bis(2-Chloroethyl) ether	KS
Butyl benzyl phthalate	KS
Carbazole	KS
Chlorobenzilate	KS
Chrysene	KS
Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	KS
Diallate	KS
Dibenz(a,h) anthracene	KS
Diethyl phthalate	KS
Dimethoate	KS
Dimethyl phthalate	KS
Di-n-butyl phthalate	KS
Di-n-octyl phthalate	KS
Diphenylamine	KS
Disulfoton	KS
Ethyl methanesulfonate	KS
Famphur	KS
Fluoranthene	KS
Fluorene	KS
Hexachlorobenzene	KS
Hexachlorobutadiene	KS
Hexachlorocyclopentadiene	KS
Hexachloroethane	KS
Hexachlorophene	KS
Hexachloropropene	KS
Indeno(1,2,3-cd) pyrene	KS
Isodrin	KS

Program/Matrix: ***RCRA (Non Potable Water)***

Isophorone	KS
Isosafrole	KS
Kepone	KS
Methapyrilene	KS
Methyl methanesulfonate	KS
Methyl parathion (Parathion, methyl)	KS
Naphthalene	KS
Nitrobenzene	KS
n-Nitrosodiethylamine	KS
n-Nitrosodimethylamine	KS
n-Nitroso-di-n-butylamine	KS
n-Nitrosodi-n-propylamine	KS
n-Nitrosodiphenylamine	KS
n-Nitrosomethylethalamine	KS
n-Nitrosomorpholine	KS
n-Nitrosopiperidine	KS
n-Nitrosopyrrolidine	KS
o,o,o-Triethyl phosphorothioate	KS
Parathion, ethyl	KS
Pentachlorobenzene	KS
Pentachloronitrobenzene	KS
Pentachlorophenol	KS
Phenacetin	KS
Phenanthrene	KS
Phenol	KS
Phorate	KS
Pronamide (Kerb)	KS
Pyrene	KS
Pyridine	KS
Safrole	KS
Sulfotep (Tetraethyl dithiopyrophosphate)	KS
Thionazin (Zinophos)	KS

Method EPA 9012A

Amenable cyanide	KS
Cyanide	KS

Method EPA 9038

Sulfate	KS

Method EPA 9045C

pH	KS

Method EPA 9056A

Bromide	KS
Chloride	KS
Fluoride	KS
Nitrate	KS
Nitrite	KS
Sulfate	KS

Pace Analytical Services, LLC - Indianapolis IN **Primary AB**

Program/Matrix: ***RCRA (Non Potable Water)***

Method **EPA 9066**

Total phenolics KS

Method **EPA 9095B**

Paint Filter Test KS

Pace Analytical Services, LLC - Indianapolis IN **Primary AB**

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

Method EPA 1010A	
Ignitability	KS
Method EPA 1311	
Toxicity Characteristic Leaching Procedure (TCLP)	KS
Method EPA 1312	
Synthetic Precipitation Leaching Procedure (SCLP)	KS
Method EPA 6010B	
Aluminum	KS
Antimony	KS
Arsenic	KS
Barium	KS
Beryllium	KS
Boron	KS
Cadmium	KS
Calcium	KS
Chromium	KS
Cobalt	KS
Copper	KS
Iron	KS
Lead	KS
Magnesium	KS
Manganese	KS
Molybdenum	KS
Nickel	KS
Potassium	KS
Selenium	KS
Silver	KS
Sodium	KS
Strontium	KS
Thallium	KS
Tin	KS
Titanium	KS
Vanadium	KS
Zinc	KS
Method EPA 6020	
Aluminum	KS
Antimony	KS
Arsenic	KS
Barium	KS
Beryllium	KS
Cadmium	KS
Chromium	KS
Cobalt	KS
Copper	KS
Lead	KS
Manganese	KS

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

Nickel	KS
Selenium	KS
Silver	KS
Thallium	KS
Vanadium	KS
Zinc	KS
Method EPA 7196A	
Chromium VI	KS
Method EPA 7470A	
Mercury	KS
Method EPA 7471A	
Mercury	KS
Method EPA 8015D	
Diesel range organics (DRO)	KS
Ethanol	KS
Ethylene glycol	KS
Gasoline range organics (GRO)	KS
Isobutyl alcohol (2-Methyl-1-propanol)	KS
Isopropyl alcohol (2-Propanol, Isopropanol)	KS
Methanol	KS
n-Butyl alcohol (1-Butanol, n-Butanol)	KS
n-Propanol (1-Propanol)	KS
Propylene glycol	KS
Method EPA 8081B	
4,4'-DDD	KS
4,4'-DDE	KS
4,4'-DDT	KS
Aldrin	KS
alpha-BHC (alpha-Hexachlorocyclohexane)	KS
alpha-Chlordane, cis-Chlordane	KS
beta-BHC (beta-Hexachlorocyclohexane)	KS
Chlordane (tech.)(N.O.S.)	KS
delta-BHC	KS
Dieldrin	KS
Endosulfan I	KS
Endosulfan II	KS
Endosulfan sulfate	KS
Endrin	KS
Endrin aldehyde	KS
Endrin ketone	KS
gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)	KS
gamma-Chlordane	KS
Heptachlor	KS
Heptachlor epoxide	KS
Methoxychlor	KS
Toxaphene (Chlorinated camphene)	KS

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

Method EPA 8082A

Aroclor-1016 (PCB-1016)	KS
Aroclor-1221 (PCB-1221)	KS
Aroclor-1232 (PCB-1232)	KS
Aroclor-1242 (PCB-1242)	KS
Aroclor-1248 (PCB-1248)	KS
Aroclor-1254 (PCB-1254)	KS
Aroclor-1260 (PCB-1260)	KS

Method EPA 8141B

Atrazine	KS
Azinphos-methyl (Guthion)	KS
Chlorpyrifos	KS
Chlorpyrifos-methyl	KS
Demeton-o	KS
Demeton-s	KS
Diazinon	KS
Dichlorovos (DDVP, Dichlorvos)	KS
Dimethoate	KS
Disulfoton	KS
Famphur	KS
Malathion	KS
Merphos	KS
Methyl parathion (Parathion, methyl)	KS
Naled	KS
Parathion, ethyl	KS
Phorate	KS
Ronnel	KS
Simazine	KS
Terbufos	KS
Tetrachlorvinphos (Stirophos, Gardona) E-isomer	KS

Method EPA 8151A

2,4,5-T	KS
2,4-D	KS
2,4-DB	KS
3,5-Dichlorobenzoic acid	KS
Acifluorfen	KS
Bentazon	KS
Dalapon	KS
DCPA di acid degradate	KS
Dicamba	KS
Dichloroprop (Dichlorprop)	KS
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	KS
MCPA	KS
MCPP	KS
Pentachlorophenol	KS
Picloram	KS

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

Silvex (2,4,5-TP)	KS

Method EPA 8260C

1,1,1,2-Tetrachloroethane	KS
1,1,1-Trichloroethane	KS
1,1,2,2-Tetrachloroethane	KS
1,1,2-Trichloro-1,2,2-trifluoroethane	KS
1,1,2-Trichloroethane	KS
1,1-Dichloroethane	KS
1,1-Dichloroethylene	KS
1,1-Dichloropropene	KS
1,2,3-Trichlorobenzene	KS
1,2,3-Trichloropropane	KS
1,2,4-Trichlorobenzene	KS
1,2,4-Trimethylbenzene	KS
1,2-Dibromo-3-chloropropane (DBCP)	KS
1,2-Dibromoethane (EDB, Ethylene dibromide)	KS
1,2-Dichlorobenzene (o-Dichlorobenzene)	KS
1,2-Dichloroethane (Ethylene dichloride)	KS
1,2-Dichloropropane	KS
1,3,5-Trichlorobenzene	KS
1,3,5-Trimethylbenzene	KS
1,3-Dichlorobenzene	KS
1,3-Dichloropropane	KS
1,4-Dichlorobenzene	KS
1,4-Dioxane (1,4- Diethyleneoxide)	KS
2,2-Dichloropropane	KS
2-Butanone (Methyl ethyl ketone, MEK)	KS
2-Chloroethyl vinyl ether	KS
2-Chlorotoluene	KS
2-Hexanone	KS
4-Chlorotoluene	KS
4-Isopropyltoluene (p-Cymene,p-Isopropyltoluene)	KS
4-Methyl-2-pentanone (MIBK)	KS
Acetone	KS
Acetonitrile	KS
Acrolein (Propenal)	KS
Acrylonitrile	KS
Allyl chloride (3-Chloropropene)	KS
Benzene	KS
Bromobenzene	KS
Bromochloromethane	KS
Bromodichloromethane	KS
Bromoform	KS
Carbon disulfide	KS
Carbon tetrachloride	KS
Chlorobenzene	KS

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

Chlorodibromomethane	KS
Chloroethane (Ethyl chloride)	KS
Chloroform	KS
cis-1,2-Dichloroethylene	KS
cis-1,3-Dichloropropene	KS
Dibromomethane (Methylene bromide)	KS
Dichlorodifluoromethane (Freon-12)	KS
Diethyl ether	KS
Ethyl acetate	KS
Ethyl methacrylate	KS
Ethylbenzene	KS
Hexachlorobutadiene	KS
Iodomethane (Methyl iodide)	KS
Isopropylbenzene	KS
Methacrylonitrile	KS
Methyl bromide (Bromomethane)	KS
Methyl chloride (Chloromethane)	KS
Methyl methacrylate	KS
Methyl tert-butyl ether (MTBE)	KS
Methylene chloride (Dichloromethane)	KS
m-Xylene	KS
Naphthalene	KS
n-Butyl alcohol (1-Butanol, n-Butanol)	KS
n-Butylbenzene	KS
n-Propylbenzene	KS
o-Xylene	KS
Propionitrile (Ethyl cyanide)	KS
p-Xylene	KS
sec-Butylbenzene	KS
Styrene	KS
tert-Butyl alcohol	KS
tert-Butylbenzene	KS
Tetrachloroethylene (Perchloroethylene)	KS
Toluene	KS
trans-1,2-Dichloroethylene	KS
trans-1,3-Dichloropropylene	KS
trans-1,4-Dichloro-2-butene	KS
Trichloroethene (Trichloroethylene)	KS
Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	KS
Vinyl acetate	KS
Vinyl chloride	KS
Xylene (total)	KS

Method **EPA 8270C**

1,2,4,5-Tetrachlorobenzene	KS
1,2,4-Trichlorobenzene	KS
1,2-Dichlorobenzene (o-Dichlorobenzene)	KS

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

1,2-Diphenylhydrazine	KS
1,3-Dichlorobenzene	KS
1,3-Dinitrobenzene (1,3-DNB)	KS
1,4-Dichlorobenzene	KS
1,4-Naphthoquinone	KS
1,4-Phenylenediamine	KS
1-Methylnaphthalene	KS
1-Naphthylamine	KS
2,2'-Oxybis(1-chloropropane), bis(2-Chloro-1-methylethyl)ether	KS
2,3,4,6-Tetrachlorophenol	KS
2,4,5-Trichlorophenol	KS
2,4,6-Trichlorophenol	KS
2,4-Dichlorophenol	KS
2,4-Dimethylphenol	KS
2,4-Dinitrophenol	KS
2,4-Dinitrotoluene (2,4-DNT)	KS
2,6-Dichlorophenol	KS
2,6-Dinitrotoluene (2,6-DNT)	KS
2-Acetylaminofluorene	KS
2-Chloronaphthalene	KS
2-Chlorophenol	KS
2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	KS
2-Methylaniline (o-Toluidine)	KS
2-Methylnaphthalene	KS
2-Methylphenol (o-Cresol)	KS
2-Naphthylamine	KS
2-Nitroaniline	KS
2-Nitrophenol	KS
2-Picoline (2-Methylpyridine)	KS
3,3'-Dichlorobenzidine	KS
3,3'-Dimethylbenzidine	KS
3-Methylcholanthrene	KS
3-Methylphenol (m-Cresol)	KS
3-Nitroaniline	KS
4-Aminobiphenyl	KS
4-Bromophenyl phenyl ether	KS
4-Chloro-3-methylphenol	KS
4-Chloroaniline	KS
4-Chlorophenyl phenylether	KS
4-Dimethyl aminoazobenzene	KS
4-Methylphenol (p-Cresol)	KS
4-Nitroaniline	KS
4-Nitrophenol	KS
4-Nitroquinoline 1-oxide	KS
5-Nitro-o-toluidine	KS
7,12-Dimethylbenz(a) anthracene	KS
a-a-Dimethylphenethylamine	KS

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

Acenaphthene	KS
Acenaphthylene	KS
Acetophenone	KS
Aniline	KS
Anthracene	KS
Aramite	KS
Benzidine	KS
Benzo(a)anthracene	KS
Benzo(a)pyrene	KS
Benzo(b)fluoranthene	KS
Benzo(g,h,i)perylene	KS
Benzo(k)fluoranthene	KS
Benzoic acid	KS
Benzyl alcohol	KS
bis(2-Chloroethoxy)methane	KS
bis(2-Chloroethyl) ether	KS
Butyl benzyl phthalate	KS
Carbazole	KS
Chlorobenzilate	KS
Chrysene	KS
Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)	KS
Diallate	KS
Dibenz(a,h) anthracene	KS
Diethyl phthalate	KS
Dimethoate	KS
Dimethyl phthalate	KS
Di-n-butyl phthalate	KS
Di-n-octyl phthalate	KS
Diphenylamine	KS
Disulfoton	KS
Ethyl methanesulfonate	KS
Famphur	KS
Fluoranthene	KS
Fluorene	KS
Hexachlorobenzene	KS
Hexachlorobutadiene	KS
Hexachlorocyclopentadiene	KS
Hexachloroethane	KS
Hexachlorophene	KS
Hexachloropropene	KS
Indeno(1,2,3-cd) pyrene	KS
Isodrin	KS
Isophorone	KS
Isosafrole	KS
Kepone	KS
Methapyrilene	KS
Methyl methanesulfonate	KS

Program/Matrix: ***RCRA (Solid & Hazardous Material)***

Methyl parathion (Parathion, methyl)	KS
Naphthalene	KS
Nitrobenzene	KS
n-Nitrosodiethylamine	KS
n-Nitrosodimethylamine	KS
n-Nitroso-di-n-butylamine	KS
n-Nitrosodi-n-propylamine	KS
n-Nitrosodiphenylamine	KS
n-Nitrosomethylethalamine	KS
n-Nitrosomorpholine	KS
n-Nitrosopiperidine	KS
n-Nitrosopyrrolidine	KS
o,o,o-Triethyl phosphorothioate	KS
Parathion, ethyl	KS
Pentachlorobenzene	KS
Pentachloronitrobenzene	KS
Pentachlorophenol	KS
Phenacetin	KS
Phenanthrene	KS
Phenol	KS
Phorate	KS
Pronamide (Kerb)	KS
Pyrene	KS
Pyridine	KS
Safrole	KS
Sulfotep (Tetraethyl dithiopyrophosphate)	KS
Thionazin (Zinophos)	KS
Method EPA 9012A	
Amenable cyanide	KS
Cyanide	KS
Method EPA 9045C	
pH	KS
Method EPA 9066	
Total phenolics	KS
Method EPA 9095B	
Paint Filter Test	KS

End of Scope of Accreditation

ATTACHMENT A-6

COMMONWEALTH OF PENNSYLVANIA DEPARTMENT OF ENVIRONMENTAL PROTECTION
CERTIFICATION PROGRAM, PACE ANALYTICAL SERVICES, LLC-PITTSBURGH

COMMONWEALTH OF PENNSYLVANIA
DEPARTMENT OF ENVIRONMENTAL PROTECTION

BUREAU OF LABORATORIES

LABORATORY ACCREDITATION PROGRAM

pennsylvania
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certifies That

65-00282

Pace Analytical Services LLC - Pittsburgh PA

1638 Roseytown Suites 2, 3, & 4, Greensburg, PA, 15601

Having duly met the requirement of
The act of June 29, 2002 (P.L. 596, No. 90)
dealing with Environmental Laboratories Accreditation
(27 Pa. C.S. 4104-4113) and the
National Environmental Laboratory Accreditation Program Standard
is hereby approved as an

Accredited Laboratory

to conduct analysis within the fields of accreditations more fully described in the attached Scope of Accreditation

NELAP accreditation granted by the PA DEP to an environmental laboratory is conditioned upon continued compliance with the current edition of the NELAC Standard or TNI Standard and the following Subchapters and Sections of 25 Pa. Code Chapter 252: Subchapter A (relating to general provisions); Subchapter B (relating to application, fees and supporting documents); Subchapter E (relating to proficiency test study requirements); Subchapter F (relating to assessment requirements); Subchapter G (relating to miscellaneous provisions); Section 252.307; and Section 252.401.

Expiration Date: **03/31/2020**

Certificate Number: **019**

Aaren S. Alger, Chief
Laboratory Accreditation Program
Bureau of Laboratories

Continued accreditation status depends on successful ongoing participation in the program
Certificate not transferable Surrender upon revocation
To be conspicuously displayed at the Laboratory
Not valid unless accompanied by a valid Scope of Accreditation
Shall not be used to imply endorsement by the Commonwealth of Pennsylvania
Customers are urged to verify the laboratory's current accreditation status
PA DEP is a NELAP recognized accreditation body

pennsylvania
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Laboratory Scope of Accreditation

Attached to Certificate of Accreditation 019-001 expiration date 03/31/2020. This listing of accredited analytes should be used only when associated with a valid certificate of accreditation.

Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Drinking Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
ASTM D5174-97		Uranium, total	NELAP	PA	10/12/2010
ECLS-R-GA		Gross alpha (including radium & U, excluding radon)	NELAP	PA	08/01/2016
EPA 900.0		Gross alpha	NELAP	PA	05/27/2008
EPA 900.0		Gross beta	NELAP	PA	05/27/2008
EPA 901.1		Gamma emitters	NELAP	PA	05/27/2008
EPA 903.0		Total alpha radium	NELAP	PA	03/31/2017
EPA 903.1		Radium-226	NELAP	PA	07/15/2011
EPA 904.0		Radium-228	NELAP	PA	05/27/2008
EPA 905.0		Strontium-90	NELAP	PA	02/01/2011
EPA 906.0		Tritium	NELAP	PA	05/27/2008
HASL 300 U-02-RC		Uranium-234	NELAP	PA	01/16/2014
HASL 300 U-02-RC		Uranium-235	NELAP	PA	01/16/2014
HASL 300 U-02-RC		Uranium-238	NELAP	PA	01/16/2014
SM 7110 C		Gross alpha	NELAP	PA	09/25/2008
SM 7500-Rn B		Radon-222 in water	NELAP	PA	10/10/2008
SOP (00282) R-008		Americium-241	NELAP	PA	05/27/2008
SOP (00282) R-008		Plutonium-239	NELAP	PA	05/27/2008
SOP (00282) R-008		Thorium-230	NELAP	PA	05/27/2008
SOP (00282) R-008		Uranium-234	NELAP	PA	05/27/2008
SOP (00282) R-008		Uranium-238	NELAP	PA	05/27/2008

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
ASTM D516-02		Sulfate	NELAP	PA	05/06/2009
ASTM D516-11		Sulfate	NELAP	PA	04/02/2018
ASTM D516-90		Sulfate	NELAP	PA	05/06/2009
ASTM D5174-97		Uranium, total	NELAP	PA	08/12/2008
ASTM D7237-10		Free cyanide	NELAP	PA	03/31/2017
EPA 120.1		Conductivity	NELAP	PA	04/21/2014
EPA 1311		Toxicity characteristic leaching procedure (TCLP)	NELAP	PA	03/29/2005
EPA 1312		Synthetic precipitation leaching procedure (SPLP)	NELAP	PA	03/29/2005
EPA 160.4		Residue, volatile	NELAP	PA	07/28/2006
EPA 1664	A	Oil and grease	NELAP	PA	10/01/2014
EPA 1664	A	Total recoverable petroleum hydrocarbons (TRPH)	NELAP	PA	08/17/2017
EPA 180.1		Turbidity	NELAP	PA	07/28/2006
EPA 200.7	4.4	Aluminum	NELAP	PA	03/29/2005
EPA 200.7	4.4	Antimony	NELAP	PA	03/29/2005
EPA 200.7	4.4	Arsenic	NELAP	PA	03/29/2005
EPA 200.7	4.4	Barium	NELAP	PA	03/29/2005

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Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 200.7	4.4	Beryllium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Boron	NELAP	PA	03/29/2005
EPA 200.7	4.4	Cadmium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Calcium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Chromium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Cobalt	NELAP	PA	03/29/2005
EPA 200.7	4.4	Copper	NELAP	PA	03/29/2005
EPA 200.7	4.4	Iron	NELAP	PA	03/29/2005
EPA 200.7	4.4	Lead	NELAP	PA	03/29/2005
EPA 200.7	4.4	Lithium	NELAP	PA	06/22/2006
EPA 200.7	4.4	Magnesium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Manganese	NELAP	PA	03/29/2005
EPA 200.7	4.4	Molybdenum	NELAP	PA	03/29/2005
EPA 200.7	4.4	Nickel	NELAP	PA	03/29/2005
EPA 200.7	4.4	Phosphorus, total	NELAP	PA	01/04/2007
EPA 200.7	4.4	Potassium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Selenium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Silica, as SiO2	NELAP	PA	06/22/2006
EPA 200.7	4.4	Silicon	NELAP	PA	06/22/2006
EPA 200.7	4.4	Silver	NELAP	PA	03/29/2005
EPA 200.7	4.4	Sodium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Strontium	NELAP	PA	06/22/2006
EPA 200.7	4.4	Sulfur	NELAP	PA	01/09/2012
EPA 200.7	4.4	Thallium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Tin	NELAP	PA	01/04/2007
EPA 200.7	4.4	Titanium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Vanadium	NELAP	PA	03/29/2005
EPA 200.7	4.4	Zinc	NELAP	PA	03/29/2005
EPA 200.7	4.4	Zirconium	NELAP	PA	06/22/2006
EPA 245.1	3.0	Mercury	NELAP	PA	03/29/2005
EPA 300.0	2.1	Bromide	NELAP	PA	05/18/2009
EPA 300.0	2.1	Chloride	NELAP	PA	09/29/2010
EPA 300.0	2.1	Fluoride	NELAP	PA	05/06/2009
EPA 300.0	2.1	Sulfate	NELAP	PA	09/29/2010
EPA 3005	A	Preconcentration under acid	NELAP	PA	03/29/2005
EPA 335.4		Total cyanide	NELAP	PA	05/06/2009
EPA 350.1		Ammonia as N	NELAP	PA	05/06/2009
EPA 351.2		Kjeldahl nitrogen, total (TKN)	NELAP	PA	05/06/2009
EPA 3510	C	Separatory funnel liquid-liquid extraction	NELAP	PA	03/29/2005
EPA 3535	A	Solid-phase extraction (SPE)	NELAP	PA	09/18/2013
EPA 3535		Solid-phase extraction (SPE)	NELAP	PA	03/29/2005
EPA 3660	B	Sulfur cleanup	NELAP	PA	03/29/2005

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(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 3665	A	Sulfuric acid/permanganate clean-up	NELAP	PA	03/29/2005
EPA 410.4		Chemical oxygen demand (COD)	NELAP	PA	05/06/2009
EPA 420.1		Total phenolics	NELAP	PA	05/06/2009
EPA 5030	B	Aqueous-phase purge-and-trap	NELAP	PA	03/29/2005
EPA 5030	C	Aqueous-phase purge-and-trap	NELAP	PA	09/18/2013
EPA 6010	B	Metals by ICP/AES	NELAP	PA	02/25/2010
EPA 6010	C	Metals by ICP/AES	NELAP	PA	09/18/2013
EPA 6010		Aluminum	NELAP	PA	02/25/2010
EPA 6010		Antimony	NELAP	PA	02/25/2010
EPA 6010		Arsenic	NELAP	PA	02/25/2010
EPA 6010		Barium	NELAP	PA	02/25/2010
EPA 6010		Beryllium	NELAP	PA	02/25/2010
EPA 6010		Boron	NELAP	PA	02/25/2010
EPA 6010		Cadmium	NELAP	PA	02/25/2010
EPA 6010		Calcium	NELAP	PA	02/25/2010
EPA 6010		Chromium	NELAP	PA	02/25/2010
EPA 6010		Cobalt	NELAP	PA	02/25/2010
EPA 6010		Copper	NELAP	PA	02/25/2010
EPA 6010		Iron	NELAP	PA	02/25/2010
EPA 6010		Lead	NELAP	PA	02/25/2010
EPA 6010		Lithium	NELAP	PA	02/25/2010
EPA 6010		Magnesium	NELAP	PA	02/25/2010
EPA 6010		Manganese	NELAP	PA	02/25/2010
EPA 6010		Molybdenum	NELAP	PA	02/25/2010
EPA 6010		Nickel	NELAP	PA	02/25/2010
EPA 6010		Phosphorus, total	NELAP	PA	02/25/2010
EPA 6010		Potassium	NELAP	PA	02/25/2010
EPA 6010		Selenium	NELAP	PA	02/25/2010
EPA 6010		Silica, as SiO2	NELAP	PA	02/25/2010
EPA 6010		Silicon	NELAP	PA	02/25/2010
EPA 6010		Silver	NELAP	PA	02/25/2010
EPA 6010		Sodium	NELAP	PA	02/25/2010
EPA 6010		Strontium	NELAP	PA	02/25/2010
EPA 6010		Sulfur	NELAP	PA	01/09/2012
EPA 6010		Thallium	NELAP	PA	02/25/2010
EPA 6010		Tin	NELAP	PA	02/25/2010
EPA 6010		Titanium	NELAP	PA	02/25/2010
EPA 6010		Vanadium	NELAP	PA	02/25/2010
EPA 6010		Zinc	NELAP	PA	02/25/2010
EPA 6010		Zirconium	NELAP	PA	02/25/2010
EPA 608		4,4'-DDD	NELAP	PA	03/29/2005
EPA 608		4,4'-DDE	NELAP	PA	03/29/2005

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(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 608		4,4'-DDT	NELAP	PA	03/29/2005
EPA 608		Aldrin (HHDN)	NELAP	PA	03/29/2005
EPA 608		Aroclor-1016 (PCB-1016)	NELAP	PA	03/29/2005
EPA 608		Aroclor-1221 (PCB-1221)	NELAP	PA	03/29/2005
EPA 608		Aroclor-1232 (PCB-1232)	NELAP	PA	03/29/2005
EPA 608		Aroclor-1242 (PCB-1242)	NELAP	PA	03/29/2005
EPA 608		Aroclor-1248 (PCB-1248)	NELAP	PA	03/29/2005
EPA 608		Aroclor-1254 (PCB-1254)	NELAP	PA	03/29/2005
EPA 608		Aroclor-1260 (PCB-1260)	NELAP	PA	03/29/2005
EPA 608		Aroclor-1262 (PCB-1262)	NELAP	PA	02/09/2007
EPA 608		Aroclor-1268 (PCB-1268)	NELAP	PA	02/09/2007
EPA 608		Chlordane (tech.)	NELAP	PA	03/29/2005
EPA 608		Dieldrin	NELAP	PA	03/29/2005
EPA 608		Endosulfan I	NELAP	PA	03/29/2005
EPA 608		Endosulfan II	NELAP	PA	03/29/2005
EPA 608		Endosulfan sulfate	NELAP	PA	03/29/2005
EPA 608		Endrin	NELAP	PA	03/29/2005
EPA 608		Endrin aldehyde	NELAP	PA	03/29/2005
EPA 608		Endrin ketone	NELAP	PA	02/05/2007
EPA 608		Heptachlor	NELAP	PA	03/29/2005
EPA 608		Heptachlor epoxide	NELAP	PA	03/29/2005
EPA 608		Toxaphene (Chlorinated camphene)	NELAP	PA	03/29/2005
EPA 608		alpha-BHC (alpha-Hexachlorocyclohexane)	NELAP	PA	03/29/2005
EPA 608		alpha-Chlordane	NELAP	PA	02/22/2013
EPA 608		beta-BHC (beta-Hexachlorocyclohexane)	NELAP	PA	03/29/2005
EPA 608		delta-BHC (delta-Hexachlorocyclohexane)	NELAP	PA	03/29/2005
EPA 608		gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NELAP	PA	03/29/2005
EPA 608		gamma-Chlordane	NELAP	PA	02/22/2013
EPA 608.3		4,4'-DDD	NELAP	PA	03/21/2019
EPA 608.3		4,4'-DDE	NELAP	PA	03/21/2019
EPA 608.3		4,4'-DDT	NELAP	PA	03/21/2019
EPA 608.3		Aldrin (HHDN)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1016 (PCB-1016)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1221 (PCB-1221)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1232 (PCB-1232)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1242 (PCB-1242)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1248 (PCB-1248)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1254 (PCB-1254)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1260 (PCB-1260)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1262 (PCB-1262)	NELAP	PA	03/21/2019
EPA 608.3		Aroclor-1268 (PCB-1268)	NELAP	PA	03/21/2019

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Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 608.3		Chlordane (tech.)	NELAP	PA	03/21/2019
EPA 608.3		Dieldrin	NELAP	PA	03/21/2019
EPA 608.3		Endosulfan I	NELAP	PA	03/21/2019
EPA 608.3		Endosulfan II	NELAP	PA	03/21/2019
EPA 608.3		Endosulfan sulfate	NELAP	PA	03/21/2019
EPA 608.3		Endrin	NELAP	PA	03/21/2019
EPA 608.3		Endrin aldehyde	NELAP	PA	03/21/2019
EPA 608.3		Heptachlor	NELAP	PA	03/21/2019
EPA 608.3		Heptachlor epoxide	NELAP	PA	03/21/2019
EPA 608.3		Toxaphene (Chlorinated camphene)	NELAP	PA	03/21/2019
EPA 608.3		alpha-BHC (alpha-Hexachlorocyclohexane)	NELAP	PA	03/21/2019
EPA 608.3		alpha-Chlordane	NELAP	PA	03/21/2019
EPA 608.3		beta-BHC (beta-Hexachlorocyclohexane)	NELAP	PA	03/21/2019
EPA 608.3		delta-BHC (delta-Hexachlorocyclohexane)	NELAP	PA	03/21/2019
EPA 608.3		gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NELAP	PA	03/21/2019
EPA 608.3		gamma-Chlordane	NELAP	PA	03/21/2019
EPA 624		1,1,1,2-Tetrachloroethane	NELAP	PA	06/22/2006
EPA 624		1,1,1-Trichloroethane	NELAP	PA	03/29/2005
EPA 624		1,1,2,2-Tetrachloroethane	NELAP	PA	03/29/2005
EPA 624		1,1,2-Trichloroethane	NELAP	PA	03/29/2005
EPA 624		1,1-Dichloroethane	NELAP	PA	03/29/2005
EPA 624		1,1-Dichloroethene (1,1-Dichloroethylene)	NELAP	PA	03/29/2005
EPA 624		1,1-Dichloropropene	NELAP	PA	06/22/2006
EPA 624		1,2,3-Trichlorobenzene	NELAP	PA	06/22/2006
EPA 624		1,2,3-Trichloropropane (1,2,3-TCP)	NELAP	PA	06/22/2006
EPA 624		1,2,4-Trichlorobenzene	NELAP	PA	06/22/2006
EPA 624		1,2,4-Trimethylbenzene	NELAP	PA	06/22/2006
EPA 624		1,2-Dibromo-3-chloropropane (DBCP, Dibromochloropropane)	NELAP	PA	06/22/2006
EPA 624		1,2-Dibromoethane (EDB, Ethylene dibromide)	NELAP	PA	06/22/2006
EPA 624		1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	03/29/2005
EPA 624		1,2-Dichloroethane	NELAP	PA	03/29/2005
EPA 624		1,2-Dichloropropane	NELAP	PA	03/29/2005
EPA 624		1,3,5-Trimethylbenzene	NELAP	PA	10/01/2014
EPA 624		1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	04/29/2016
EPA 624		1,3-Dichloropropane	NELAP	PA	05/30/2013
EPA 624		1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	03/29/2005
EPA 624		1,4-Dioxane (1,4-Diethyleneoxide)	NELAP	PA	05/30/2013
EPA 624		2,2-Dichloropropane	NELAP	PA	06/22/2006
EPA 624		2-Butanone (Methyl ethyl ketone, MEK)	NELAP	PA	06/22/2006
EPA 624		2-Chloroethyl vinyl ether	NELAP	PA	06/22/2006

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Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 624		2-Chlorotoluene	NELAP	PA	06/22/2006
EPA 624		2-Hexanone	NELAP	PA	06/22/2006
EPA 624		2-Methylnaphthalene	NELAP	PA	05/30/2013
EPA 624		2-Nitropropane	NELAP	PA	05/30/2013
EPA 624		4-Chlorotoluene	NELAP	PA	05/30/2013
EPA 624		4-Methyl-2-pentanone (MIBK)	NELAP	PA	06/22/2006
EPA 624		Acetone	NELAP	PA	06/22/2006
EPA 624		Acetonitrile	NELAP	PA	05/30/2013
EPA 624		Acrolein (Propenal)	NELAP	PA	06/22/2006
EPA 624		Acrylonitrile	NELAP	PA	06/22/2006
EPA 624		Allyl chloride (3-Chloropropene)	NELAP	PA	05/30/2013
EPA 624		Benzene	NELAP	PA	03/29/2005
EPA 624		Bromobenzene	NELAP	PA	06/22/2006
EPA 624		Bromochloromethane	NELAP	PA	05/30/2013
EPA 624		Bromodichloromethane	NELAP	PA	03/29/2005
EPA 624		Bromoform	NELAP	PA	03/29/2005
EPA 624		Carbon disulfide	NELAP	PA	06/22/2006
EPA 624		Carbon tetrachloride	NELAP	PA	03/29/2005
EPA 624		Chlorobenzene	NELAP	PA	03/29/2005
EPA 624		Chloroethane	NELAP	PA	03/29/2005
EPA 624		Chloroform	NELAP	PA	03/29/2005
EPA 624		Chloroprene (2-Chloro-1,3-butadiene)	NELAP	PA	05/30/2013
EPA 624		Cyclohexane	NELAP	PA	05/30/2013
EPA 624		Cyclohexanone	NELAP	PA	05/30/2013
EPA 624		Dibromochloromethane	NELAP	PA	03/29/2005
EPA 624		Dibromomethane	NELAP	PA	06/22/2006
EPA 624		Dichlorodifluoromethane (Freon 12)	NELAP	PA	06/22/2006
EPA 624		Diethyl ether (Ethyl ether)	NELAP	PA	05/30/2013
EPA 624		Diisopropyl ether (DIPE)	NELAP	PA	05/30/2013
EPA 624		Ethanol	NELAP	PA	05/30/2013
EPA 624		Ethyl acetate	NELAP	PA	05/30/2013
EPA 624		Ethyl methacrylate	NELAP	PA	05/30/2013
EPA 624		Ethylbenzene	NELAP	PA	03/29/2005
EPA 624		Hexachlorobutadiene (1,3-Hexachlorobutadiene)	NELAP	PA	06/22/2006
EPA 624		Iodomethane (Methyl iodide)	NELAP	PA	05/30/2013
EPA 624		Isobutyl alcohol (2-Methyl-1-propanol)	NELAP	PA	05/30/2013
EPA 624		Isopropylbenzene (Cumene)	NELAP	PA	06/22/2006
EPA 624		Methacrylonitrile	NELAP	PA	05/30/2013
EPA 624		Methyl acetate	NELAP	PA	05/30/2013
EPA 624		Methyl bromide (Bromomethane)	NELAP	PA	03/29/2005
EPA 624		Methyl chloride (Chloromethane)	NELAP	PA	03/29/2005
EPA 624		Methyl tert-butyl ether (MTBE)	NELAP	PA	06/22/2006

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DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 624		Methylcyclohexane	NELAP	PA	05/30/2013
EPA 624		Methylene chloride (Dichloromethane)	NELAP	PA	03/29/2005
EPA 624		Methylmethacrylate	NELAP	PA	08/12/2015
EPA 624		Naphthalene	NELAP	PA	06/22/2006
EPA 624		Propionitrile (Ethyl cyanide)	NELAP	PA	05/30/2013
EPA 624		Styrene	NELAP	PA	06/22/2006
EPA 624		Tetrachloroethene (PCE, Perchloroethylene)	NELAP	PA	03/29/2005
EPA 624		Tetrahydrofuran (THF)	NELAP	PA	05/30/2013
EPA 624		Toluene	NELAP	PA	03/29/2005
EPA 624		Trichloroethene (TCE, Trichloroethylene)	NELAP	PA	03/29/2005
EPA 624		Trichlorofluoromethane (Freon 11)	NELAP	PA	01/04/2007
EPA 624		Vinyl acetate	NELAP	PA	05/30/2013
EPA 624		Vinyl chloride (Chloroethene)	NELAP	PA	03/29/2005
EPA 624		Xylenes, total	NELAP	PA	03/29/2005
EPA 624		cis-1,2-Dichloroethene	NELAP	PA	06/22/2006
EPA 624		cis-1,3-Dichloropropene	NELAP	PA	03/29/2005
EPA 624		m+p-Xylene	NELAP	PA	06/22/2006
EPA 624		n-Butylbenzene	NELAP	PA	06/22/2006
EPA 624		n-Hexane	NELAP	PA	05/30/2013
EPA 624		n-Propylbenzene	NELAP	PA	06/22/2006
EPA 624		o-Xylene	NELAP	PA	06/22/2006
EPA 624		p-Isopropyltoluene (4-Isopropyltoluene)	NELAP	PA	06/22/2006
EPA 624		sec-Butylbenzene	NELAP	PA	06/22/2006
EPA 624		tert-Butyl alcohol (2-Methyl-2-propanol)	NELAP	PA	06/22/2006
EPA 624		tert-Butyl ethyl ether	NELAP	PA	05/30/2013
EPA 624		trans-1,2-Dichloroethene	NELAP	PA	03/29/2005
EPA 624		trans-1,3-Dichloropropene	NELAP	PA	03/29/2005
EPA 624		trans-1,4-Dichloro-2-butene	NELAP	PA	05/30/2013
EPA 625		1,1'-Biphenyl (Biphenyl, Lemonene)	NELAP	PA	02/22/2013
EPA 625		1,2,4-Trichlorobenzene	NELAP	PA	03/29/2005
EPA 625		1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	03/29/2005
EPA 625		1,2-Diphenylhydrazine	NELAP	PA	06/22/2006
EPA 625		1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	03/29/2005
EPA 625		1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	03/29/2005
EPA 625		1-Methylnaphthalene	NELAP	PA	02/22/2013
EPA 625		2,2'-oxybis(1-Chloropropane)	NELAP	PA	03/29/2005
EPA 625		2,4,5-Trichlorophenol	NELAP	PA	06/22/2006
EPA 625		2,4,6-Trichlorophenol	NELAP	PA	03/29/2005
EPA 625		2,4-Dichlorophenol	NELAP	PA	03/29/2005
EPA 625		2,4-Dimethylphenol	NELAP	PA	03/29/2005
EPA 625		2,4-Dinitrophenol	NELAP	PA	03/29/2005
EPA 625		2,4-Dinitrotoluene (2,4-DNT)	NELAP	PA	03/29/2005

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(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 625		2,6-Dinitrotoluene (2,6-DNT)	NELAP	PA	03/29/2005
EPA 625		2-Chloronaphthalene	NELAP	PA	03/29/2005
EPA 625		2-Chlorophenol	NELAP	PA	03/29/2005
EPA 625		2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NELAP	PA	03/29/2005
EPA 625		2-Methylnaphthalene	NELAP	PA	02/22/2013
EPA 625		2-Methylphenol (o-Cresol)	NELAP	PA	02/22/2013
EPA 625		2-Nitroaniline	NELAP	PA	02/22/2013
EPA 625		2-Nitrophenol	NELAP	PA	03/29/2005
EPA 625		3+4-Methylphenol (m+p-Cresol)	NELAP	PA	06/14/2011
EPA 625		3,3'-Dichlorobenzidine	NELAP	PA	03/29/2005
EPA 625		4-Bromophenyl phenyl ether	NELAP	PA	03/29/2005
EPA 625		4-Chloro-3-methylphenol	NELAP	PA	03/29/2005
EPA 625		4-Chloroaniline	NELAP	PA	02/22/2013
EPA 625		4-Chlorophenyl phenyl ether	NELAP	PA	03/29/2005
EPA 625		4-Nitroaniline	NELAP	PA	02/22/2013
EPA 625		4-Nitrophenol	NELAP	PA	03/29/2005
EPA 625		Acenaphthene	NELAP	PA	03/29/2005
EPA 625		Acenaphthylene	NELAP	PA	03/29/2005
EPA 625		Acetophenone	NELAP	PA	02/22/2013
EPA 625		Aniline	NELAP	PA	02/22/2013
EPA 625		Anthracene	NELAP	PA	03/29/2005
EPA 625		Atrazine	NELAP	PA	02/22/2013
EPA 625		Benzaldehyde	NELAP	PA	02/22/2013
EPA 625		Benzidine	NELAP	PA	03/29/2005
EPA 625		Benzo[a]anthracene	NELAP	PA	03/29/2005
EPA 625		Benzo[a]pyrene	NELAP	PA	03/29/2005
EPA 625		Benzo[b]fluoranthene	NELAP	PA	03/29/2005
EPA 625		Benzo[ghi]perylene	NELAP	PA	03/29/2005
EPA 625		Benzo[k]fluoranthene	NELAP	PA	03/29/2005
EPA 625		Benzoic acid	NELAP	PA	02/22/2013
EPA 625		Benzyl alcohol	NELAP	PA	02/22/2013
EPA 625		Butyl benzyl phthalate (Benzyl butyl phthalate)	NELAP	PA	03/29/2005
EPA 625		Caprolactam	NELAP	PA	02/22/2013
EPA 625		Carbazole	NELAP	PA	02/22/2013
EPA 625		Chrysene (Benzo[a]phenanthrene)	NELAP	PA	03/29/2005
EPA 625		Di-n-butyl phthalate	NELAP	PA	03/29/2005
EPA 625		Di-n-octyl phthalate	NELAP	PA	03/29/2005
EPA 625		Dibenzo[a,h]anthracene	NELAP	PA	03/29/2005
EPA 625		Dibenzofuran	NELAP	PA	02/22/2013
EPA 625		Diethyl phthalate	NELAP	PA	03/29/2005
EPA 625		Dimethyl phthalate	NELAP	PA	03/29/2005

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Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 625		Fluoranthene	NELAP	PA	03/29/2005
EPA 625		Fluorene	NELAP	PA	03/29/2005
EPA 625		Hexachlorobenzene	NELAP	PA	03/29/2005
EPA 625		Hexachlorobutadiene (1,3-Hexachlorobutadiene)	NELAP	PA	03/29/2005
EPA 625		Hexachlorocyclopentadiene	NELAP	PA	03/29/2005
EPA 625		Hexachloroethane	NELAP	PA	03/29/2005
EPA 625		Indeno(1,2,3-cd)pyrene	NELAP	PA	03/29/2005
EPA 625		Isophorone	NELAP	PA	03/29/2005
EPA 625		N-Nitrosodi-n-propylamine	NELAP	PA	03/29/2005
EPA 625		N-Nitrosodimethylamine	NELAP	PA	03/29/2005
EPA 625		N-Nitrosodiphenylamine	NELAP	PA	03/29/2005
EPA 625		Naphthalene	NELAP	PA	03/29/2005
EPA 625		Nitrobenzene	NELAP	PA	03/29/2005
EPA 625		Pentachlorophenol (PCP)	NELAP	PA	03/29/2005
EPA 625		Phenanthrene	NELAP	PA	03/29/2005
EPA 625		Phenol	NELAP	PA	03/29/2005
EPA 625		Pyrene	NELAP	PA	03/29/2005
EPA 625		bis(2-Chloroethoxy)methane	NELAP	PA	03/29/2005
EPA 625		bis(2-Chloroethyl) ether	NELAP	PA	03/29/2005
EPA 625		bis(2-Ethylhexyl) phthalate (DEHP)	NELAP	PA	03/29/2005
EPA 7.3.3.2		Reactive cyanide	NELAP	PA	03/29/2005
EPA 7.3.4.2		Reactive sulfide	NELAP	PA	03/29/2005
EPA 7196	A	Chromium VI	NELAP	PA	05/06/2009
EPA 7470	A	Mercury	NELAP	PA	03/29/2005
EPA 8011		1,2-Dibromo-3-chloropropane (DBCP, Dibromochloropropane)	NELAP	PA	03/04/2015
EPA 8011		1,2-Dibromoethane (EDB, Ethylene dibromide)	NELAP	PA	03/04/2015
EPA 8015	B	Nonhalogenated organics by GC/FID	NELAP	PA	02/25/2010
EPA 8015	D	Nonhalogenated organics by GC/FID	NELAP	PA	09/18/2013
EPA 8015		Diesel-range organics (DRO)	NELAP	PA	02/25/2010
EPA 8015		Gasoline-range organics (GRO)	NELAP	PA	02/25/2010
EPA 8081	B	Organochlorine pesticides by GC/ECD	NELAP	PA	09/18/2013
EPA 8081		4,4'-DDD	NELAP	PA	02/25/2010
EPA 8081		4,4'-DDE	NELAP	PA	02/25/2010
EPA 8081		4,4'-DDT	NELAP	PA	02/25/2010
EPA 8081		Aldrin (HHDN)	NELAP	PA	02/25/2010
EPA 8081		Chlordane (tech.)	NELAP	PA	02/25/2010
EPA 8081		Dieldrin	NELAP	PA	02/25/2010
EPA 8081		Endosulfan I	NELAP	PA	02/25/2010
EPA 8081		Endosulfan II	NELAP	PA	02/25/2010
EPA 8081		Endosulfan sulfate	NELAP	PA	02/25/2010
EPA 8081		Endrin	NELAP	PA	02/25/2010

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PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8081		Endrin aldehyde	NELAP	PA	02/25/2010
EPA 8081		Endrin ketone	NELAP	PA	02/25/2010
EPA 8081		Heptachlor	NELAP	PA	02/25/2010
EPA 8081		Heptachlor epoxide	NELAP	PA	02/25/2010
EPA 8081		Methoxychlor	NELAP	PA	02/25/2010
EPA 8081		Toxaphene (Chlorinated camphene)	NELAP	PA	02/25/2010
EPA 8081		alpha-BHC (alpha-Hexachlorocyclohexane)	NELAP	PA	02/25/2010
EPA 8081		alpha-Chlordane	NELAP	PA	02/25/2010
EPA 8081		beta-BHC (beta-Hexachlorocyclohexane)	NELAP	PA	02/25/2010
EPA 8081		delta-BHC (delta-Hexachlorocyclohexane)	NELAP	PA	02/25/2010
EPA 8081		gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NELAP	PA	02/25/2010
EPA 8081		gamma-Chlordane	NELAP	PA	02/25/2010
EPA 8082	A	PCBs by GC/ECD	NELAP	PA	09/18/2013
EPA 8082		Aroclor-1016 (PCB-1016)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1221 (PCB-1221)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1232 (PCB-1232)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1242 (PCB-1242)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1248 (PCB-1248)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1254 (PCB-1254)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1260 (PCB-1260)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1262 (PCB-1262)	NELAP	PA	02/09/2007
EPA 8082		Aroclor-1268 (PCB-1268)	NELAP	PA	02/09/2007
EPA 8260	B	VOCs by GC/MS	NELAP	PA	02/25/2010
EPA 8260	C	VOCs by GC/MS	NELAP	PA	09/18/2013
EPA 8260		1,1,1,2-Tetrachloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1,1-Trichloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1,2,2-Tetrachloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	NELAP	PA	02/25/2010
EPA 8260		1,1,2-Trichloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1-Dichloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1-Dichloroethene (1,1-Dichloroethylene)	NELAP	PA	02/25/2010
EPA 8260		1,1-Dichloropropene	NELAP	PA	02/25/2010
EPA 8260		1,2,3-Trichlorobenzene	NELAP	PA	02/25/2010
EPA 8260		1,2,3-Trichloropropane (1,2,3-TCP)	NELAP	PA	02/25/2010
EPA 8260		1,2,4-Trichlorobenzene	NELAP	PA	02/25/2010
EPA 8260		1,2,4-Trimethylbenzene	NELAP	PA	02/25/2010
EPA 8260		1,2-Dibromo-3-chloropropane (DBCP, Dibromochloropropane)	NELAP	PA	02/25/2010
EPA 8260		1,2-Dibromoethane (EDB, Ethylene dibromide)	NELAP	PA	02/25/2010
EPA 8260		1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8260		1,2-Dichloroethane	NELAP	PA	02/25/2010

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Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8260		1,2-Dichloropropane	NELAP	PA	02/25/2010
EPA 8260		1,3,5-Trimethylbenzene	NELAP	PA	10/01/2014
EPA 8260		1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	04/29/2016
EPA 8260		1,3-Dichloropropane	NELAP	PA	02/25/2010
EPA 8260		1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8260		1,4-Dioxane (1,4-Diethyleneoxide)	NELAP	PA	02/25/2010
EPA 8260		2,2-Dichloropropane	NELAP	PA	02/25/2010
EPA 8260		2-Butanone (Methyl ethyl ketone, MEK)	NELAP	PA	02/25/2010
EPA 8260		2-Chloroethyl vinyl ether	NELAP	PA	02/25/2010
EPA 8260		2-Chlorotoluene	NELAP	PA	02/25/2010
EPA 8260		2-Hexanone	NELAP	PA	02/25/2010
EPA 8260		2-Methylnaphthalene	NELAP	PA	05/30/2013
EPA 8260		2-Nitropropane	NELAP	PA	02/25/2010
EPA 8260		4-Chlorotoluene	NELAP	PA	02/25/2010
EPA 8260		4-Methyl-2-pentanone (MIBK)	NELAP	PA	02/25/2010
EPA 8260		Acetone	NELAP	PA	02/25/2010
EPA 8260		Acetonitrile	NELAP	PA	02/25/2010
EPA 8260		Acrolein (Propenal)	NELAP	PA	02/25/2010
EPA 8260		Acrylonitrile	NELAP	PA	02/25/2010
EPA 8260		Allyl chloride (3-Chloropropene)	NELAP	PA	02/25/2010
EPA 8260		Benzene	NELAP	PA	02/25/2010
EPA 8260		Bromobenzene	NELAP	PA	02/25/2010
EPA 8260		Bromochloromethane	NELAP	PA	02/25/2010
EPA 8260		Bromodichloromethane	NELAP	PA	02/25/2010
EPA 8260		Bromoform	NELAP	PA	02/25/2010
EPA 8260		Bromomethane (Methyl bromide)	NELAP	PA	02/25/2010
EPA 8260		Carbon disulfide	NELAP	PA	02/25/2010
EPA 8260		Carbon tetrachloride	NELAP	PA	02/25/2010
EPA 8260		Chlorobenzene	NELAP	PA	02/25/2010
EPA 8260		Chloroethane	NELAP	PA	02/25/2010
EPA 8260		Chloroform	NELAP	PA	02/25/2010
EPA 8260		Chloromethane (Methyl chloride)	NELAP	PA	02/25/2010
EPA 8260		Chloroprene (2-Chloro-1,3-butadiene)	NELAP	PA	02/25/2010
EPA 8260		Cyclohexane	NELAP	PA	02/25/2010
EPA 8260		Cyclohexanone	NELAP	PA	02/25/2010
EPA 8260		Dibromochloromethane	NELAP	PA	02/25/2010
EPA 8260		Dibromomethane	NELAP	PA	02/25/2010
EPA 8260		Dichlorodifluoromethane (Freon 12)	NELAP	PA	02/25/2010
EPA 8260		Diethyl ether (Ethyl ether)	NELAP	PA	02/25/2010
EPA 8260		Diisopropyl ether (DIPE)	NELAP	PA	02/25/2010
EPA 8260		Ethanol	NELAP	PA	06/14/2011
EPA 8260		Ethyl acetate	NELAP	PA	02/25/2010

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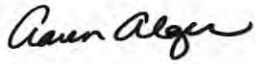
Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8260		Ethyl methacrylate	NELAP	PA	02/25/2010
EPA 8260		Ethyl tert-butyl ether (ETBE)	NELAP	PA	02/25/2010
EPA 8260		Ethylbenzene	NELAP	PA	02/25/2010
EPA 8260		Hexachlorobutadiene (1,3-Hexachlorobutadiene)	NELAP	PA	02/25/2010
EPA 8260		Iodomethane (Methyl iodide)	NELAP	PA	02/25/2010
EPA 8260		Isobutyl alcohol (2-Methyl-1-propanol)	NELAP	PA	02/25/2010
EPA 8260		Isopropylbenzene (Cumene)	NELAP	PA	02/25/2010
EPA 8260		Methacrylonitrile	NELAP	PA	02/25/2010
EPA 8260		Methyl acetate	NELAP	PA	02/25/2010
EPA 8260		Methyl tert-butyl ether (MTBE)	NELAP	PA	02/25/2010
EPA 8260		Methylacrylate	NELAP	PA	02/25/2010
EPA 8260		Methylcyclohexane	NELAP	PA	02/25/2010
EPA 8260		Methylene chloride (Dichloromethane)	NELAP	PA	02/25/2010
EPA 8260		Methylmethacrylate	NELAP	PA	08/12/2015
EPA 8260		Naphthalene	NELAP	PA	02/25/2010
EPA 8260		Propionitrile (Ethyl cyanide)	NELAP	PA	02/25/2010
EPA 8260		Styrene	NELAP	PA	02/25/2010
EPA 8260		Tetrachloroethene (PCE, Perchloroethylene)	NELAP	PA	02/25/2010
EPA 8260		Tetrahydrofuran (THF)	NELAP	PA	02/25/2010
EPA 8260		Toluene	NELAP	PA	02/25/2010
EPA 8260		Trichloroethene (TCE, Trichloroethylene)	NELAP	PA	02/25/2010
EPA 8260		Trichlorofluoromethane (Freon 11)	NELAP	PA	02/25/2010
EPA 8260		Vinyl acetate	NELAP	PA	02/25/2010
EPA 8260		Vinyl chloride (Chloroethene)	NELAP	PA	02/25/2010
EPA 8260		Xylenes, total	NELAP	PA	02/25/2010
EPA 8260		cis-1,2-Dichloroethene	NELAP	PA	02/25/2010
EPA 8260		cis-1,3-Dichloropropene	NELAP	PA	02/25/2010
EPA 8260		m+p-Xylene	NELAP	PA	02/25/2010
EPA 8260		n-Butylbenzene	NELAP	PA	02/25/2010
EPA 8260		n-Hexane	NELAP	PA	02/25/2010
EPA 8260		n-Propylbenzene	NELAP	PA	02/25/2010
EPA 8260		o-Xylene	NELAP	PA	02/25/2010
EPA 8260		p-Isopropyltoluene (4-Isopropyltoluene)	NELAP	PA	02/25/2010
EPA 8260		sec-Butylbenzene	NELAP	PA	02/25/2010
EPA 8260		tert-Amyl ethyl ether (TAEE)	NELAP	PA	02/25/2010
EPA 8260		tert-Amyl methyl ether (TAME)	NELAP	PA	08/11/2011
EPA 8260		tert-Butyl alcohol (2-Methyl-2-propanol)	NELAP	PA	02/25/2010
EPA 8260		tert-Butylbenzene	NELAP	PA	02/25/2010
EPA 8260		trans-1,2-Dichloroethene	NELAP	PA	02/25/2010
EPA 8260		trans-1,3-Dichloropropene	NELAP	PA	02/25/2010
EPA 8260		trans-1,4-Dichloro-2-butene	NELAP	PA	02/25/2010
EPA 8270	D	SOCs by GC/MS	NELAP	PA	09/18/2013



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Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8270		1,1'-Biphenyl (Biphenyl, Lemonene)	NELAP	PA	02/25/2010
EPA 8270		1,2,4,5-Tetrachlorobenzene	NELAP	PA	10/02/2012
EPA 8270		1,2,4-Trichlorobenzene	NELAP	PA	02/25/2010
EPA 8270		1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8270		1,2-Diphenylhydrazine	NELAP	PA	02/25/2010
EPA 8270		1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8270		1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8270		1,4-Dioxane (1,4-Diethyleneoxide)	NELAP	PA	02/25/2010
EPA 8270		1-Methylnaphthalene	NELAP	PA	09/18/2013
EPA 8270		2,2'-oxybis(1-Chloropropane)	NELAP	PA	02/25/2010
EPA 8270		2,3,4,6-Tetrachlorophenol	NELAP	PA	10/02/2012
EPA 8270		2,4,5-Trichlorophenol	NELAP	PA	02/25/2010
EPA 8270		2,4,6-Trichlorophenol	NELAP	PA	02/25/2010
EPA 8270		2,4-Dichlorophenol	NELAP	PA	02/25/2010
EPA 8270		2,4-Dimethylphenol	NELAP	PA	02/25/2010
EPA 8270		2,4-Dinitrophenol	NELAP	PA	02/25/2010
EPA 8270		2,4-Dinitrotoluene (2,4-DNT)	NELAP	PA	02/25/2010
EPA 8270		2,6-Dinitrotoluene (2,6-DNT)	NELAP	PA	02/25/2010
EPA 8270		2-Chloronaphthalene	NELAP	PA	02/25/2010
EPA 8270		2-Chlorophenol	NELAP	PA	02/25/2010
EPA 8270		2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NELAP	PA	02/25/2010
EPA 8270		2-Methylnaphthalene	NELAP	PA	02/25/2010
EPA 8270		2-Methylphenol (o-Cresol)	NELAP	PA	02/25/2010
EPA 8270		2-Nitroaniline	NELAP	PA	02/25/2010
EPA 8270		2-Nitrophenol	NELAP	PA	02/25/2010
EPA 8270		3+4-Methylphenol (m+p-Cresol)	NELAP	PA	02/25/2010
EPA 8270		3,3'-Dichlorobenzidine	NELAP	PA	02/25/2010
EPA 8270		3-Nitroaniline	NELAP	PA	02/25/2010
EPA 8270		4-Bromophenyl phenyl ether	NELAP	PA	02/25/2010
EPA 8270		4-Chloro-3-methylphenol	NELAP	PA	02/25/2010
EPA 8270		4-Chloroaniline	NELAP	PA	02/25/2010
EPA 8270		4-Chlorophenyl phenyl ether	NELAP	PA	02/25/2010
EPA 8270		4-Nitroaniline	NELAP	PA	02/25/2010
EPA 8270		4-Nitrophenol	NELAP	PA	02/25/2010
EPA 8270		8-Hydroxyquinoline	NELAP	PA	02/25/2010
EPA 8270		Acenaphthene	NELAP	PA	02/25/2010
EPA 8270		Acenaphthylene	NELAP	PA	02/25/2010
EPA 8270		Acetophenone	NELAP	PA	02/25/2010
EPA 8270		Aniline	NELAP	PA	02/25/2010
EPA 8270		Anthracene	NELAP	PA	02/25/2010
EPA 8270		Atrazine	NELAP	PA	02/25/2010

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(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8270		Benzaldehyde	NELAP	PA	02/25/2010
EPA 8270		Benzidine	NELAP	PA	02/25/2010
EPA 8270		Benzo[a]anthracene	NELAP	PA	02/25/2010
EPA 8270		Benzo[a]pyrene	NELAP	PA	02/25/2010
EPA 8270		Benzo[b]fluoranthene	NELAP	PA	02/25/2010
EPA 8270		Benzo[ghi]perylene	NELAP	PA	02/25/2010
EPA 8270		Benzo[k]fluoranthene	NELAP	PA	02/25/2010
EPA 8270		Benzoic acid	NELAP	PA	02/25/2010
EPA 8270		Benzyl alcohol	NELAP	PA	02/25/2010
EPA 8270		Benzyl butyl phthalate (Butyl benzyl phthalate)	NELAP	PA	02/25/2010
EPA 8270		Caprolactam	NELAP	PA	02/25/2010
EPA 8270		Carbazole	NELAP	PA	02/25/2010
EPA 8270		Chrysene (Benzo[a]phenanthrene)	NELAP	PA	02/25/2010
EPA 8270		Di-n-butyl phthalate	NELAP	PA	02/25/2010
EPA 8270		Di-n-octyl phthalate	NELAP	PA	02/25/2010
EPA 8270		Dibenzo[a,h]anthracene	NELAP	PA	02/25/2010
EPA 8270		Dibenzofuran	NELAP	PA	02/25/2010
EPA 8270		Diethyl phthalate	NELAP	PA	02/25/2010
EPA 8270		Dimethyl phthalate	NELAP	PA	02/25/2010
EPA 8270		Fluoranthene	NELAP	PA	02/25/2010
EPA 8270		Fluorene	NELAP	PA	02/25/2010
EPA 8270		Hexachlorobenzene	NELAP	PA	02/25/2010
EPA 8270		Hexachlorobutadiene (1,3-Hexachlorobutadiene)	NELAP	PA	02/25/2010
EPA 8270		Hexachlorocyclopentadiene	NELAP	PA	02/25/2010
EPA 8270		Hexachloroethane	NELAP	PA	02/25/2010
EPA 8270		Indeno(1,2,3-cd)pyrene	NELAP	PA	02/25/2010
EPA 8270		Isophorone	NELAP	PA	02/25/2010
EPA 8270		N-Nitrosodi-n-propylamine	NELAP	PA	02/25/2010
EPA 8270		N-Nitrosodimethylamine	NELAP	PA	02/25/2010
EPA 8270		N-Nitrosodiphenylamine	NELAP	PA	02/25/2010
EPA 8270		Naphthalene	NELAP	PA	02/25/2010
EPA 8270		Nitrobenzene	NELAP	PA	02/25/2010
EPA 8270		Pentachlorophenol (PCP)	NELAP	PA	02/25/2010
EPA 8270		Phenanthrene	NELAP	PA	02/25/2010
EPA 8270		Phenol	NELAP	PA	02/25/2010
EPA 8270		Pyrene	NELAP	PA	02/25/2010
EPA 8270		Pyridine	NELAP	PA	02/25/2010
EPA 8270		Tributyl phosphate	NELAP	PA	02/25/2010
EPA 8270		bis(2-Chloroethoxy)methane	NELAP	PA	02/25/2010
EPA 8270		bis(2-Chloroethyl) ether	NELAP	PA	02/25/2010
EPA 8270		bis(2-Ethylhexyl) phthalate (DEHP)	NELAP	PA	02/25/2010
EPA 900.0		Gross alpha	NELAP	PA	05/27/2008

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Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 900.0		Gross beta	NELAP	PA	05/27/2008
EPA 901.1		Gamma emitters	NELAP	PA	08/12/2008
EPA 9010	C	Amenable cyanide	NELAP	PA	08/31/2006
EPA 9010	C	Total cyanide	NELAP	PA	08/31/2006
EPA 9012	A	Total cyanide	NELAP	PA	02/22/2013
EPA 9012	B	Total cyanide	NELAP	PA	08/31/2006
EPA 9014		Total cyanide	NELAP	PA	03/25/2014
EPA 903.0		Total alpha radium	NELAP	PA	03/31/2017
EPA 903.1		Radium-226	NELAP	PA	05/27/2008
EPA 9038		Sulfate	NELAP	PA	05/06/2009
EPA 904.0		Radium-228	NELAP	PA	08/12/2008
EPA 9040	B	pH	NELAP	PA	03/29/2005
EPA 9040	C	pH	NELAP	PA	02/22/2013
EPA 905.0		Strontium-90	NELAP	PA	08/12/2008
EPA 9050	A	Conductivity	NELAP	PA	04/21/2014
EPA 906.0		Tritium	NELAP	PA	08/12/2008
EPA 9060	A	Total organic carbon (TOC)	NELAP	PA	09/18/2013
EPA 9060		Total organic carbon (TOC)	NELAP	PA	02/03/2009
EPA 9065		Total phenolics	NELAP	PA	05/06/2009
EPA 9251		Chloride	NELAP	PA	05/06/2009
EPA 9310		Gross alpha	NELAP	PA	05/27/2008
EPA 9310		Gross beta	NELAP	PA	05/27/2008
EPA 9315		Radium-226	NELAP	PA	05/30/2013
EPA 9315		Total radium	NELAP	PA	05/27/2008
EPA 9320		Radium-228	NELAP	PA	05/27/2008
SM 2120 B		Color	NELAP	PA	04/10/2007
SM 2310 B		Acidity as CaCO3	NELAP	PA	04/21/2014
SM 2320 B		Alkalinity as CaCO3	NELAP	PA	01/04/2007
SM 2540 B		Residue, total	NELAP	PA	04/10/2007
SM 2540 C		Residue, filterable (TDS)	NELAP	PA	04/10/2007
SM 2540 D		Residue, nonfilterable (TSS)	NELAP	PA	04/10/2007
SM 2540 F		Residue, settleable	NELAP	PA	04/10/2007
SM 2550 B		Temperature, deg. C	NELAP	PA	04/10/2007
SM 3500-Cr B	20-22	Chromium VI	NELAP	PA	09/18/2013
SM 3500-Fe B	20/21	Ferrous iron	NELAP	PA	01/09/2012
SM 3500-Fe D	18/19	Ferrous iron	NELAP	PA	01/09/2012
SM 4500-CN- C/E		Total cyanide	NELAP	PA	04/10/2007
SM 4500-CN- G		Amenable cyanide	NELAP	PA	04/10/2007
SM 4500-CN- I		Weak acid dissociable cyanide	NELAP	PA	05/06/2009
SM 4500-CN- M		Thiocyanate	NELAP	PA	05/06/2009
SM 4500-Cl G		Total residual chlorine	NELAP	PA	04/10/2007
SM 4500-Cl- E		Chloride	NELAP	PA	05/06/2009

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Matrix: Non-Potable Water

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
SM 4500-F- C		Fluoride	NELAP	PA	05/06/2009
SM 4500-H+ B		pH	NELAP	PA	04/10/2007
SM 4500-NO2- B		Nitrite as N	NELAP	PA	06/30/2011
SM 4500-NO3- F		Nitrate as N	NELAP	PA	05/06/2009
SM 4500-NO3- F		Total nitrate-nitrite	NELAP	PA	04/13/2017
SM 4500-O G		Oxygen (dissolved)	NELAP	PA	04/10/2007
SM 4500-P B		Preliminary treatment of phosphate samples	NELAP	PA	05/06/2009
SM 4500-P E		Orthophosphate as P	NELAP	PA	05/06/2009
SM 4500-P E		Phosphorus, total	NELAP	PA	05/06/2009
SM 4500-S F		Sulfide	NELAP	PA	04/10/2007
SM 4500-SO3 B		Sulfite, SO3	NELAP	PA	04/10/2007
SM 5210 B		Biochemical oxygen demand (BOD)	NELAP	PA	05/06/2009
SM 5210 B		Carbonaceous BOD (CBOD)	NELAP	PA	05/06/2009
SM 5310 C		Total organic carbon (TOC)	NELAP	PA	04/25/2008
SM 5540 C		Surfactants as MBAS	NELAP	PA	05/06/2009
SM 7110 C-00		Gross alpha	NELAP	PA	05/27/2008
SOP (00282) PGH-I-065-0		Fluoroborate	NELAP	PA	10/01/2014

Matrix: Solid and Chemical Materials

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
ASTM D3987-85		Water leach	NELAP	PA	10/18/2013
ASTM D93-13		Flashpoint	NELAP	PA	04/21/2014
DOE 4.5.2.3		Gamma emitters	NELAP	PA	09/18/2013
EPA 1010	A	Ignitability	NELAP	PA	04/21/2014
EPA 1010		Ignitability	NELAP	PA	03/29/2005
EPA 1311		Toxicity characteristic leaching procedure (TCLP)	NELAP	PA	03/29/2005
EPA 1312		Synthetic precipitation leaching procedure (SPLP)	NELAP	PA	03/29/2005
EPA 160.4		Residue, volatile	NELAP	PA	05/30/2013
EPA 1664	A	Non-polar material	NELAP	PA	03/31/2017
EPA 3050	B	Acid digestion of solids	NELAP	PA	03/29/2005
EPA 3060	A	Alkaline digestion of Cr(VI)	NELAP	PA	02/22/2013
EPA 3060		Alkaline digestion of Cr(VI)	NELAP	PA	05/06/2009
EPA 350.1		Ammonia as N	NELAP	PA	08/01/2013
EPA 351.2		Kjeldahl nitrogen, total (TKN)	NELAP	PA	09/18/2013
EPA 3546		Microwave extraction	NELAP	PA	04/20/2009
EPA 3580	A	Waste dilution	NELAP	PA	03/29/2005
EPA 3660	B	Sulfur cleanup	NELAP	PA	03/29/2005
EPA 3665	A	Sulfuric acid/permanganate clean-up	NELAP	PA	03/29/2005
EPA 5035	A	Closed-system purge-and-trap (bisulfate option)	NELAP	PA	10/29/2009
EPA 5035	A	Closed-system purge-and-trap (methanol option)	NELAP	PA	10/29/2009

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Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Solid and Chemical Materials

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 5035	A	Closed-system purge-and-trap (unpreserved)	NELAP	PA	10/29/2009
EPA 6010	B	Metals by ICP/AES	NELAP	PA	02/25/2010
EPA 6010	C	Metals by ICP/AES	NELAP	PA	09/18/2013
EPA 6010		Aluminum	NELAP	PA	02/25/2010
EPA 6010		Antimony	NELAP	PA	02/25/2010
EPA 6010		Arsenic	NELAP	PA	02/25/2010
EPA 6010		Barium	NELAP	PA	02/25/2010
EPA 6010		Beryllium	NELAP	PA	02/25/2010
EPA 6010		Boron	NELAP	PA	02/25/2010
EPA 6010		Cadmium	NELAP	PA	02/25/2010
EPA 6010		Calcium	NELAP	PA	02/25/2010
EPA 6010		Chromium	NELAP	PA	02/25/2010
EPA 6010		Cobalt	NELAP	PA	02/25/2010
EPA 6010		Copper	NELAP	PA	02/25/2010
EPA 6010		Iron	NELAP	PA	02/25/2010
EPA 6010		Lead	NELAP	PA	02/25/2010
EPA 6010		Lithium	NELAP	PA	02/25/2010
EPA 6010		Magnesium	NELAP	PA	02/25/2010
EPA 6010		Manganese	NELAP	PA	02/25/2010
EPA 6010		Molybdenum	NELAP	PA	02/25/2010
EPA 6010		Nickel	NELAP	PA	02/25/2010
EPA 6010		Phosphorus, total	NELAP	PA	02/25/2010
EPA 6010		Potassium	NELAP	PA	02/25/2010
EPA 6010		Selenium	NELAP	PA	02/25/2010
EPA 6010		Silica, as SiO2	NELAP	PA	02/25/2010
EPA 6010		Silicon	NELAP	PA	03/25/2014
EPA 6010		Silver	NELAP	PA	02/25/2010
EPA 6010		Sodium	NELAP	PA	02/25/2010
EPA 6010		Strontium	NELAP	PA	02/25/2010
EPA 6010		Thallium	NELAP	PA	02/25/2010
EPA 6010		Tin	NELAP	PA	02/25/2010
EPA 6010		Titanium	NELAP	PA	02/25/2010
EPA 6010		Vanadium	NELAP	PA	02/25/2010
EPA 6010		Zinc	NELAP	PA	02/25/2010
EPA 6010		Zirconium	NELAP	PA	02/25/2010
EPA 7.3.3.2		Reactive cyanide	NELAP	PA	03/29/2005
EPA 7.3.4.2		Reactive sulfide	NELAP	PA	03/29/2005
EPA 7196	A	Chromium VI	NELAP	PA	05/06/2009
EPA 7470	A	Mercury	NELAP	PA	03/29/2005
EPA 7471	A	Mercury	NELAP	PA	03/29/2005
EPA 7471	B	Mercury	NELAP	PA	09/18/2013
EPA 8015	B	Nonhalogenated organics by GC/FID	NELAP	PA	02/25/2010

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EPA 8015	D	Nonhalogenated organics by GC/FID	NELAP	PA	09/18/2013
EPA 8015		Diesel-range organics (DRO)	NELAP	PA	02/25/2010
EPA 8015		Gasoline-range organics (GRO)	NELAP	PA	02/25/2010
EPA 8081	B	Organochlorine pesticides by GC/ECD	NELAP	PA	09/18/2013
EPA 8081		4,4'-DDD	NELAP	PA	02/25/2010
EPA 8081		4,4'-DDE	NELAP	PA	02/25/2010
EPA 8081		4,4'-DDT	NELAP	PA	02/25/2010
EPA 8081		Aldrin (HHDN)	NELAP	PA	02/25/2010
EPA 8081		Chlordane (tech.)	NELAP	PA	02/25/2010
EPA 8081		Dieldrin	NELAP	PA	02/25/2010
EPA 8081		Endosulfan I	NELAP	PA	02/25/2010
EPA 8081		Endosulfan II	NELAP	PA	02/25/2010
EPA 8081		Endosulfan sulfate	NELAP	PA	02/25/2010
EPA 8081		Endrin	NELAP	PA	02/25/2010
EPA 8081		Endrin aldehyde	NELAP	PA	02/25/2010
EPA 8081		Endrin ketone	NELAP	PA	02/25/2010
EPA 8081		Heptachlor	NELAP	PA	02/25/2010
EPA 8081		Heptachlor epoxide	NELAP	PA	02/25/2010
EPA 8081		Methoxychlor	NELAP	PA	02/25/2010
EPA 8081		Toxaphene (Chlorinated camphene)	NELAP	PA	02/25/2010
EPA 8081		alpha-BHC (alpha-Hexachlorocyclohexane)	NELAP	PA	02/25/2010
EPA 8081		alpha-Chlordane	NELAP	PA	02/25/2010
EPA 8081		beta-BHC (beta-Hexachlorocyclohexane)	NELAP	PA	02/25/2010
EPA 8081		delta-BHC (delta-Hexachlorocyclohexane)	NELAP	PA	02/25/2010
EPA 8081		gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	NELAP	PA	02/25/2010
EPA 8081		gamma-Chlordane	NELAP	PA	02/25/2010
EPA 8082	A	PCBs by GC/ECD	NELAP	PA	09/18/2013
EPA 8082		Aroclor-1016 (PCB-1016)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1221 (PCB-1221)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1232 (PCB-1232)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1242 (PCB-1242)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1248 (PCB-1248)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1254 (PCB-1254)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1260 (PCB-1260)	NELAP	PA	03/29/2005
EPA 8082		Aroclor-1262 (PCB-1262)	NELAP	PA	02/09/2007
EPA 8082		Aroclor-1268 (PCB-1268)	NELAP	PA	02/09/2007
EPA 8260	B	VOCs by GC/MS	NELAP	PA	02/25/2010
EPA 8260	C	VOCs by GC/MS	NELAP	PA	09/18/2013
EPA 8260		1,1,1,2-Tetrachloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1,1-Trichloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1,2,2-Tetrachloroethane	NELAP	PA	02/25/2010

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DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Solid and Chemical Materials

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8260		1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	NELAP	PA	02/25/2010
EPA 8260		1,1,2-Trichloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1-Dichloroethane	NELAP	PA	02/25/2010
EPA 8260		1,1-Dichloroethene (1,1-Dichloroethylene)	NELAP	PA	02/25/2010
EPA 8260		1,1-Dichloropropene	NELAP	PA	02/25/2010
EPA 8260		1,2,3-Trichlorobenzene	NELAP	PA	02/25/2010
EPA 8260		1,2,3-Trichloropropane (1,2,3-TCP)	NELAP	PA	02/25/2010
EPA 8260		1,2,4-Trichlorobenzene	NELAP	PA	02/25/2010
EPA 8260		1,2,4-Trimethylbenzene	NELAP	PA	02/25/2010
EPA 8260		1,2-Dibromo-3-chloropropane (DBCP, Dibromochloropropane)	NELAP	PA	02/25/2010
EPA 8260		1,2-Dibromoethane (EDB, Ethylene dibromide)	NELAP	PA	02/25/2010
EPA 8260		1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8260		1,2-Dichloroethane	NELAP	PA	02/25/2010
EPA 8260		1,2-Dichloropropane	NELAP	PA	02/25/2010
EPA 8260		1,3,5-Trimethylbenzene	NELAP	PA	02/25/2010
EPA 8260		1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8260		1,3-Dichloropropane	NELAP	PA	05/30/2013
EPA 8260		1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8260		1,4-Dioxane (1,4-Diethyleneoxide)	NELAP	PA	02/25/2010
EPA 8260		2,2-Dichloropropane	NELAP	PA	02/25/2010
EPA 8260		2-Butanone (Methyl ethyl ketone, MEK)	NELAP	PA	02/25/2010
EPA 8260		2-Chloroethyl vinyl ether	NELAP	PA	02/25/2010
EPA 8260		2-Chlorotoluene	NELAP	PA	02/25/2010
EPA 8260		2-Hexanone	NELAP	PA	02/25/2010
EPA 8260		2-Methylnaphthalene	NELAP	PA	05/30/2013
EPA 8260		2-Nitropropane	NELAP	PA	05/30/2013
EPA 8260		4-Chlorotoluene	NELAP	PA	02/25/2010
EPA 8260		4-Methyl-2-pentanone (MIBK)	NELAP	PA	02/25/2010
EPA 8260		Acetone	NELAP	PA	02/25/2010
EPA 8260		Acetonitrile	NELAP	PA	05/30/2013
EPA 8260		Acrolein (Propenal)	NELAP	PA	02/25/2010
EPA 8260		Acrylonitrile	NELAP	PA	02/25/2010
EPA 8260		Allyl chloride (3-Chloropropene)	NELAP	PA	05/30/2013
EPA 8260		Benzene	NELAP	PA	02/25/2010
EPA 8260		Bromobenzene	NELAP	PA	02/25/2010
EPA 8260		Bromochloromethane	NELAP	PA	02/25/2010
EPA 8260		Bromodichloromethane	NELAP	PA	02/25/2010
EPA 8260		Bromoform	NELAP	PA	02/25/2010
EPA 8260		Bromomethane (Methyl bromide)	NELAP	PA	02/25/2010
EPA 8260		Carbon disulfide	NELAP	PA	02/25/2010
EPA 8260		Carbon tetrachloride	NELAP	PA	02/25/2010

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Matrix: Solid and Chemical Materials

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8260		Chlorobenzene	NELAP	PA	02/25/2010
EPA 8260		Chloroethane	NELAP	PA	02/25/2010
EPA 8260		Chloroform	NELAP	PA	02/25/2010
EPA 8260		Chloromethane (Methyl chloride)	NELAP	PA	02/25/2010
EPA 8260		Chloroprene (2-Chloro-1,3-butadiene)	NELAP	PA	05/30/2013
EPA 8260		Cyclohexane	NELAP	PA	02/25/2010
EPA 8260		Cyclohexanone	NELAP	PA	05/30/2013
EPA 8260		Dibromochloromethane	NELAP	PA	02/25/2010
EPA 8260		Dibromomethane	NELAP	PA	02/25/2010
EPA 8260		Dichlorodifluoromethane (Freon 12)	NELAP	PA	02/25/2010
EPA 8260		Diethyl ether (Ethyl ether)	NELAP	PA	05/30/2013
EPA 8260		Diisopropyl ether (DIPE)	NELAP	PA	02/25/2010
EPA 8260		Ethanol	NELAP	PA	05/30/2013
EPA 8260		Ethyl acetate	NELAP	PA	05/30/2013
EPA 8260		Ethyl methacrylate	NELAP	PA	05/30/2013
EPA 8260		Ethyl tert-butyl ether (ETBE)	NELAP	PA	02/25/2010
EPA 8260		Ethylbenzene	NELAP	PA	02/25/2010
EPA 8260		Hexachlorobutadiene (1,3-Hexachlorobutadiene)	NELAP	PA	02/25/2010
EPA 8260		Iodomethane (Methyl iodide)	NELAP	PA	05/30/2013
EPA 8260		Isobutyl alcohol (2-Methyl-1-propanol)	NELAP	PA	05/30/2013
EPA 8260		Isopropylbenzene (Cumene)	NELAP	PA	02/25/2010
EPA 8260		Methacrylonitrile	NELAP	PA	05/30/2013
EPA 8260		Methyl acetate	NELAP	PA	02/25/2010
EPA 8260		Methyl tert-butyl ether (MTBE)	NELAP	PA	02/25/2010
EPA 8260		Methylcyclohexane	NELAP	PA	02/25/2010
EPA 8260		Methylene chloride (Dichloromethane)	NELAP	PA	02/25/2010
EPA 8260		Methylmethacrylate	NELAP	PA	08/12/2015
EPA 8260		Naphthalene	NELAP	PA	02/25/2010
EPA 8260		Propionitrile (Ethyl cyanide)	NELAP	PA	05/30/2013
EPA 8260		Styrene	NELAP	PA	02/25/2010
EPA 8260		Tetrachloroethene (PCE, Perchloroethylene)	NELAP	PA	02/25/2010
EPA 8260		Tetrahydrofuran (THF)	NELAP	PA	05/30/2013
EPA 8260		Toluene	NELAP	PA	02/25/2010
EPA 8260		Trichloroethene (TCE, Trichloroethylene)	NELAP	PA	02/25/2010
EPA 8260		Trichlorofluoromethane (Freon 11)	NELAP	PA	02/25/2010
EPA 8260		Vinyl acetate	NELAP	PA	02/25/2010
EPA 8260		Vinyl chloride (Chloroethene)	NELAP	PA	02/25/2010
EPA 8260		Xylenes, total	NELAP	PA	02/25/2010
EPA 8260		cis-1,2-Dichloroethene	NELAP	PA	02/25/2010
EPA 8260		cis-1,3-Dichloropropene	NELAP	PA	02/25/2010
EPA 8260		m+p-Xylene	NELAP	PA	02/25/2010
EPA 8260		n-Butylbenzene	NELAP	PA	02/25/2010

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DEP Laboratory ID: 65-00282
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PADWIS ID: 65282

Matrix: Solid and Chemical Materials

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8260		n-Hexane	NELAP	PA	02/25/2010
EPA 8260		n-Propylbenzene	NELAP	PA	02/25/2010
EPA 8260		o-Xylene	NELAP	PA	02/25/2010
EPA 8260		p-Isopropyltoluene (4-Isopropyltoluene)	NELAP	PA	02/25/2010
EPA 8260		sec-Butylbenzene	NELAP	PA	02/25/2010
EPA 8260		tert-Amyl methyl ether (TAME)	NELAP	PA	02/25/2010
EPA 8260		tert-Butyl alcohol (2-Methyl-2-propanol)	NELAP	PA	02/25/2010
EPA 8260		tert-Butylbenzene	NELAP	PA	02/25/2010
EPA 8260		trans-1,2-Dichloroethene	NELAP	PA	02/25/2010
EPA 8260		trans-1,3-Dichloropropene	NELAP	PA	02/25/2010
EPA 8260		trans-1,4-Dichloro-2-butene	NELAP	PA	05/30/2013
EPA 8270	D	SOCs by GC/MS	NELAP	PA	09/18/2013
EPA 8270		1,1'-Biphenyl (Biphenyl, Lemonene)	NELAP	PA	03/30/2015
EPA 8270		1,2,4,5-Tetrachlorobenzene	NELAP	PA	10/02/2012
EPA 8270		1,2,4-Trichlorobenzene	NELAP	PA	02/25/2010
EPA 8270		1,2-Dichlorobenzene (o-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8270		1,2-Diphenylhydrazine	NELAP	PA	02/25/2010
EPA 8270		1,3-Dichlorobenzene (m-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8270		1,4-Dichlorobenzene (p-Dichlorobenzene)	NELAP	PA	02/25/2010
EPA 8270		1,4-Dioxane (1,4-Diethyleneoxide)	NELAP	PA	02/25/2010
EPA 8270		1-Methylnaphthalene	NELAP	PA	09/18/2013
EPA 8270		2,2'-oxybis(1-Chloropropane)	NELAP	PA	02/25/2010
EPA 8270		2,3,4,6-Tetrachlorophenol	NELAP	PA	10/02/2012
EPA 8270		2,4,5-Trichlorophenol	NELAP	PA	02/25/2010
EPA 8270		2,4,6-Trichlorophenol	NELAP	PA	02/25/2010
EPA 8270		2,4-Dichlorophenol	NELAP	PA	02/25/2010
EPA 8270		2,4-Dimethylphenol	NELAP	PA	02/25/2010
EPA 8270		2,4-Dinitrophenol	NELAP	PA	02/25/2010
EPA 8270		2,4-Dinitrotoluene (2,4-DNT)	NELAP	PA	02/25/2010
EPA 8270		2,6-Dinitrotoluene (2,6-DNT)	NELAP	PA	02/25/2010
EPA 8270		2-Chloronaphthalene	NELAP	PA	02/25/2010
EPA 8270		2-Chlorophenol	NELAP	PA	02/25/2010
EPA 8270		2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	NELAP	PA	02/25/2010
EPA 8270		2-Methylnaphthalene	NELAP	PA	02/25/2010
EPA 8270		2-Methylphenol (o-Cresol)	NELAP	PA	02/25/2010
EPA 8270		2-Nitroaniline	NELAP	PA	02/25/2010
EPA 8270		2-Nitrophenol	NELAP	PA	02/25/2010
EPA 8270		3+4-Methylphenol (m+p-Cresol)	NELAP	PA	02/25/2010
EPA 8270		3,3'-Dichlorobenzidine	NELAP	PA	02/25/2010
EPA 8270		3-Nitroaniline	NELAP	PA	02/25/2010
EPA 8270		4-Bromophenyl phenyl ether	NELAP	PA	02/25/2010

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PADWIS ID: 65282

Matrix: Solid and Chemical Materials

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8270		4-Chloro-3-methylphenol	NELAP	PA	02/25/2010
EPA 8270		4-Chloroaniline	NELAP	PA	02/25/2010
EPA 8270		4-Chlorophenyl phenyl ether	NELAP	PA	02/25/2010
EPA 8270		4-Nitroaniline	NELAP	PA	02/25/2010
EPA 8270		4-Nitrophenol	NELAP	PA	02/25/2010
EPA 8270		Acenaphthene	NELAP	PA	02/25/2010
EPA 8270		Acenaphthylene	NELAP	PA	02/25/2010
EPA 8270		Acetophenone	NELAP	PA	02/25/2010
EPA 8270		Aniline	NELAP	PA	02/25/2010
EPA 8270		Anthracene	NELAP	PA	02/25/2010
EPA 8270		Atrazine	NELAP	PA	03/30/2015
EPA 8270		Benzaldehyde	NELAP	PA	03/30/2015
EPA 8270		Benzidine	NELAP	PA	02/25/2010
EPA 8270		Benzo[a]anthracene	NELAP	PA	02/25/2010
EPA 8270		Benzo[a]pyrene	NELAP	PA	02/25/2010
EPA 8270		Benzo[b]fluoranthene	NELAP	PA	02/25/2010
EPA 8270		Benzo[ghi]perylene	NELAP	PA	02/25/2010
EPA 8270		Benzo[k]fluoranthene	NELAP	PA	02/25/2010
EPA 8270		Benzoic acid	NELAP	PA	02/25/2010
EPA 8270		Benzyl alcohol	NELAP	PA	02/25/2010
EPA 8270		Benzyl butyl phthalate (Butyl benzyl phthalate)	NELAP	PA	02/25/2010
EPA 8270		Caprolactam	NELAP	PA	03/30/2015
EPA 8270		Carbazole	NELAP	PA	02/25/2010
EPA 8270		Chrysene (Benzo[a]phenanthrene)	NELAP	PA	02/25/2010
EPA 8270		Di-n-butyl phthalate	NELAP	PA	02/25/2010
EPA 8270		Di-n-octyl phthalate	NELAP	PA	02/25/2010
EPA 8270		Dibenzo[a,h]anthracene	NELAP	PA	02/25/2010
EPA 8270		Dibenzofuran	NELAP	PA	02/25/2010
EPA 8270		Diethyl phthalate	NELAP	PA	02/25/2010
EPA 8270		Dimethyl phthalate	NELAP	PA	02/25/2010
EPA 8270		Fluoranthene	NELAP	PA	02/25/2010
EPA 8270		Fluorene	NELAP	PA	02/25/2010
EPA 8270		Hexachlorobenzene	NELAP	PA	02/25/2010
EPA 8270		Hexachlorobutadiene (1,3-Hexachlorobutadiene)	NELAP	PA	02/25/2010
EPA 8270		Hexachlorocyclopentadiene	NELAP	PA	02/25/2010
EPA 8270		Hexachloroethane	NELAP	PA	02/25/2010
EPA 8270		Indeno(1,2,3-cd)pyrene	NELAP	PA	02/25/2010
EPA 8270		Isophorone	NELAP	PA	02/25/2010
EPA 8270		N-Nitrosodi-n-propylamine	NELAP	PA	02/25/2010
EPA 8270		N-Nitrosodimethylamine	NELAP	PA	02/25/2010
EPA 8270		N-Nitrosodiphenylamine	NELAP	PA	02/25/2010
EPA 8270		Naphthalene	NELAP	PA	02/25/2010

Attached to Certificate of Accreditation 019-001 expiration date 03/31/2020. This listing of accredited analytes should be used only when associated with a valid certificate of accreditation.

Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Solid and Chemical Materials

Method	Revision	Analyte	Accreditation Type	Primary State	Effective Date
EPA 8270		Nitrobenzene	NELAP	PA	02/25/2010
EPA 8270		Pentachlorophenol (PCP)	NELAP	PA	02/25/2010
EPA 8270		Phenanthrene	NELAP	PA	02/25/2010
EPA 8270		Phenol	NELAP	PA	02/25/2010
EPA 8270		Pyrene	NELAP	PA	02/25/2010
EPA 8270		Pyridine	NELAP	PA	02/25/2010
EPA 8270		bis(2-Chloroethoxy)methane	NELAP	PA	02/25/2010
EPA 8270		bis(2-Chloroethyl) ether	NELAP	PA	02/25/2010
EPA 8270		bis(2-Ethylhexyl) phthalate (DEHP)	NELAP	PA	02/25/2010
EPA 901.1		Gamma emitters	NELAP	PA	08/12/2008
EPA 9010	C	Total cyanide	NELAP	PA	02/22/2013
EPA 9012	A	Total cyanide	NELAP	PA	02/05/2007
EPA 9012	B	Total cyanide	NELAP	PA	02/22/2013
EPA 9013		Cyanide extraction for solids and oils	NELAP	PA	04/22/2008
EPA 9014		Total cyanide	NELAP	PA	04/22/2008
EPA 9038		Sulfate	NELAP	PA	04/15/2009
EPA 9040	B	pH	NELAP	PA	06/22/2006
EPA 9040	C	pH	NELAP	PA	02/22/2013
EPA 9045	C	pH	NELAP	PA	03/29/2005
EPA 9045	D	pH	NELAP	PA	02/22/2013
EPA 905.0 (Modified)		Strontium-90	NELAP	PA	08/12/2008
EPA 906.0 (Modified)		Tritium	NELAP	PA	08/12/2008
EPA 9065		Total phenolics	NELAP	PA	05/06/2009
EPA 9071	B	Oil and grease	NELAP	PA	05/07/2010
EPA 9071	B	Total petroleum hydrocarbons (TPH)	NELAP	PA	05/07/2010
EPA 9095	A	Paint filter liquids test	NELAP	PA	03/29/2005
EPA 9095	B	Paint filter liquids test	NELAP	PA	02/22/2013
EPA 9251		Chloride	NELAP	PA	10/30/2015
EPA 9310		Gross alpha	NELAP	PA	05/27/2008
EPA 9310		Gross beta	NELAP	PA	05/27/2008
EPA 9315		Total radium	NELAP	PA	05/27/2008
EPA 9320		Radium-228	NELAP	PA	05/27/2008
SM 4500-P B		Preliminary treatment of phosphate samples	NELAP	PA	09/11/2009
SM 4500-P E		Phosphorus, total	NELAP	PA	09/11/2009

Aaron Alger

Laboratory Status Summary

Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601
(724) 850-5600

DEP Laboratory ID: 65-00282
EPA Lab Code: PA01457
TNI Code: TNI02141
PADWIS ID: 65282

Matrix: Non-Potable Water

Method	Revision	Analyte	Status	Effective Date
EPA 6010	D	Metals by ICP/AES	Applied	02/14/2018
EPA 608.3		Endrin ketone	Applied	02/14/2018
EPA 624.1		1,1,1,2-Tetrachloroethane	Applied	02/14/2018
EPA 624.1		1,1,1-Trichloroethane	Applied	02/14/2018
EPA 624.1		1,1,2,2-Tetrachloroethane	Applied	02/14/2018
EPA 624.1		1,1,2-Trichloroethane	Applied	02/14/2018
EPA 624.1		1,1-Dichloroethane	Applied	02/14/2018
EPA 624.1		1,1-Dichloroethene (1,1-Dichloroethylene)	Applied	02/14/2018
EPA 624.1		1,1-Dichloropropene	Applied	02/14/2018
EPA 624.1		1,2,3-Trichlorobenzene	Applied	02/14/2018
EPA 624.1		1,2,3-Trichloropropane (1,2,3-TCP)	Applied	02/14/2018
EPA 624.1		1,2,4-Trichlorobenzene	Applied	02/14/2018
EPA 624.1		1,2,4-Trimethylbenzene	Applied	02/14/2018
EPA 624.1		1,2-Dibromo-3-chloropropane (DBCP, Dibromochloropropane)	Applied	02/14/2018
EPA 624.1		1,2-Dibromoethane (EDB, Ethylene dibromide)	Applied	02/14/2018
EPA 624.1		1,2-Dichlorobenzene (o-Dichlorobenzene)	Applied	02/14/2018
EPA 624.1		1,2-Dichloroethane	Applied	02/14/2018
EPA 624.1		1,2-Dichloropropane	Applied	02/14/2018
EPA 624.1		1,3,5-Trimethylbenzene	Applied	02/14/2018
EPA 624.1		1,3-Dichlorobenzene (m-Dichlorobenzene)	Applied	02/14/2018
EPA 624.1		1,3-Dichloropropane	Applied	02/14/2018
EPA 624.1		1,4-Dichlorobenzene (p-Dichlorobenzene)	Applied	02/14/2018
EPA 624.1		1,4-Dioxane (1,4-Diethyleneoxide)	Applied	02/14/2018
EPA 624.1		2,2-Dichloropropane	Applied	02/14/2018
EPA 624.1		2-Butanone (Methyl ethyl ketone, MEK)	Applied	02/14/2018
EPA 624.1		2-Chloroethyl vinyl ether	Applied	02/14/2018
EPA 624.1		2-Chlorotoluene	Applied	02/14/2018
EPA 624.1		2-Hexanone	Applied	02/14/2018
EPA 624.1		2-Methylnaphthalene	Applied	02/14/2018
EPA 624.1		2-Nitropropane	Applied	02/14/2018
EPA 624.1		4-Chlorotoluene	Applied	02/14/2018
EPA 624.1		4-Methyl-2-pentanone (MIBK)	Applied	02/14/2018
EPA 624.1		Acetone	Applied	02/14/2018
EPA 624.1		Acetonitrile	Applied	02/14/2018
EPA 624.1		Acrolein (Propenal)	Applied	02/14/2018
EPA 624.1		Acrylonitrile	Applied	02/14/2018
EPA 624.1		Allyl chloride (3-Chloropropene)	Applied	02/14/2018
EPA 624.1		Benzene	Applied	02/14/2018
EPA 624.1		Bromobenzene	Applied	02/14/2018
EPA 624.1		Bromochloromethane	Applied	02/14/2018
EPA 624.1		Bromodichloromethane	Applied	02/14/2018
EPA 624.1		Bromoform	Applied	02/14/2018
EPA 624.1		Bromomethane (Methyl bromide)	Applied	02/14/2018
EPA 624.1		Carbon disulfide	Applied	02/14/2018
EPA 624.1		Carbon tetrachloride	Applied	02/14/2018
EPA 624.1		Chlorobenzene	Applied	02/14/2018
EPA 624.1		Chloroethane	Applied	02/14/2018
EPA 624.1		Chloroform	Applied	02/14/2018
EPA 624.1		Chloromethane (Methyl chloride)	Applied	02/14/2018
EPA 624.1		Chloroprene (2-Chloro-1,3-butadiene)	Applied	02/14/2018
EPA 624.1		Cyclohexane	Applied	02/14/2018
EPA 624.1		Cyclohexanone	Applied	02/14/2018
EPA 624.1		Dibromochloromethane	Applied	02/14/2018

EPA 624.1	Dibromomethane	Applied	02/14/2018
EPA 624.1	Dichlorodifluoromethane (Freon 12)	Applied	02/14/2018
EPA 624.1	Diethyl ether (Ethyl ether)	Applied	02/14/2018
EPA 624.1	Diisopropyl ether (DIPE)	Applied	02/14/2018
EPA 624.1	Ethanol	Applied	02/14/2018
EPA 624.1	Ethyl acetate	Applied	02/14/2018
EPA 624.1	Ethyl methacrylate	Applied	02/14/2018
EPA 624.1	Ethylbenzene	Applied	02/14/2018
EPA 624.1	Hexachlorobutadiene (1,3-Hexachlorobutadiene)	Applied	02/14/2018
EPA 624.1	Iodomethane (Methyl iodide)	Applied	02/14/2018
EPA 624.1	Isobutyl alcohol (2-Methyl-1-propanol)	Applied	02/14/2018
EPA 624.1	Isopropylbenzene (Cumene)	Applied	02/14/2018
EPA 624.1	Methacrylonitrile	Applied	02/14/2018
EPA 624.1	Methyl acetate	Applied	02/14/2018
EPA 624.1	Methyl tert-butyl ether (MTBE)	Applied	02/14/2018
EPA 624.1	Methylcyclohexane	Applied	02/14/2018
EPA 624.1	Methylene chloride (Dichloromethane)	Applied	02/14/2018
EPA 624.1	Methylmethacrylate	Applied	02/14/2018
EPA 624.1	Naphthalene	Applied	02/14/2018
EPA 624.1	Propionitrile (Ethyl cyanide)	Applied	02/14/2018
EPA 624.1	Styrene	Applied	02/14/2018
EPA 624.1	Tetrachloroethene (PCE, Perchloroethylene)	Applied	02/14/2018
EPA 624.1	Tetrahydrofuran (THF)	Applied	02/14/2018
EPA 624.1	Toluene	Applied	02/14/2018
EPA 624.1	Trichloroethene (TCE, Trichloroethylene)	Applied	02/14/2018
EPA 624.1	Trichlorofluoromethane (Freon 11)	Applied	02/14/2018
EPA 624.1	Vinyl acetate	Applied	02/14/2018
EPA 624.1	Vinyl chloride (Chloroethene)	Applied	02/14/2018
EPA 624.1	Xylenes, total	Applied	02/14/2018
EPA 624.1	cis-1,2-Dichloroethene	Applied	02/14/2018
EPA 624.1	cis-1,3-Dichloropropene	Applied	02/14/2018
EPA 624.1	m+p-Xylene	Applied	02/14/2018
EPA 624.1	n-Butylbenzene	Applied	02/14/2018
EPA 624.1	n-Hexane	Applied	02/14/2018
EPA 624.1	n-Propylbenzene	Applied	02/14/2018
EPA 624.1	o-Xylene	Applied	02/14/2018
EPA 624.1	p-Isopropyltoluene (4-Isopropyltoluene)	Applied	02/14/2018
EPA 624.1	sec-Butylbenzene	Applied	02/14/2018
EPA 624.1	tert-Butyl alcohol (2-Methyl-2-propanol)	Applied	02/14/2018
EPA 624.1	tert-Butyl ethyl ether	Applied	02/14/2018
EPA 624.1	trans-1,2-Dichloroethene	Applied	02/14/2018
EPA 624.1	trans-1,3-Dichloropropene	Applied	02/14/2018
EPA 624.1	trans-1,4-Dichloro-2-butene	Applied	02/14/2018
EPA 625.1	1,1'-Biphenyl (Biphenyl, Lemonene)	Applied	02/14/2018
EPA 625.1	1,2,4-Trichlorobenzene	Applied	02/14/2018
EPA 625.1	1,2-Dichlorobenzene (o-Dichlorobenzene)	Applied	02/14/2018
EPA 625.1	1,2-Diphenylhydrazine	Applied	02/14/2018
EPA 625.1	1,3-Dichlorobenzene (m-Dichlorobenzene)	Applied	02/14/2018
EPA 625.1	1,4-Dichlorobenzene (p-Dichlorobenzene)	Applied	02/14/2018
EPA 625.1	1-Methylnaphthalene	Applied	02/14/2018
EPA 625.1	2,2'-oxybis(1-Chloropropane)	Applied	02/14/2018
EPA 625.1	2,4,5-Trichlorophenol	Applied	02/14/2018
EPA 625.1	2,4,6-Trichlorophenol	Applied	02/14/2018
EPA 625.1	2,4-Dichlorophenol	Applied	02/14/2018
EPA 625.1	2,4-Dimethylphenol	Applied	02/14/2018
EPA 625.1	2,4-Dinitrophenol	Applied	02/14/2018
EPA 625.1	2,4-Dinitrotoluene (2,4-DNT)	Applied	02/14/2018
EPA 625.1	2,6-Dinitrotoluene (2,6-DNT)	Applied	02/14/2018
EPA 625.1	2-Chloronaphthalene	Applied	02/14/2018
EPA 625.1	2-Chlorophenol	Applied	02/14/2018
EPA 625.1	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)	Applied	02/14/2018

EPA 625.1		2-Methylnaphthalene	Applied	02/14/2018
EPA 625.1		2-Methylphenol (o-Cresol)	Applied	02/14/2018
EPA 625.1		2-Nitroaniline	Applied	02/14/2018
EPA 625.1		2-Nitrophenol	Applied	02/14/2018
EPA 625.1		3+4-Methylphenol (m+p-Cresol)	Applied	02/14/2018
EPA 625.1		3,3'-Dichlorobenzidine	Applied	02/14/2018
EPA 625.1		4-Bromophenyl phenyl ether	Applied	02/14/2018
EPA 625.1		4-Chloro-3-methylphenol	Applied	02/14/2018
EPA 625.1		4-Chloroaniline	Applied	02/14/2018
EPA 625.1		4-Nitroaniline	Applied	02/14/2018
EPA 625.1		4-Nitrophenol	Applied	02/14/2018
EPA 625.1		Acenaphthene	Applied	02/14/2018
EPA 625.1		Acenaphthylene	Applied	02/14/2018
EPA 625.1		Acetophenone	Applied	02/14/2018
EPA 625.1		Aniline	Applied	02/14/2018
EPA 625.1		Anthracene	Applied	02/14/2018
EPA 625.1		Atrazine	Applied	02/14/2018
EPA 625.1		Benzaldehyde	Applied	02/14/2018
EPA 625.1		Benzidine	Applied	02/14/2018
EPA 625.1		Benzo[a]anthracene	Applied	02/14/2018
EPA 625.1		Benzo[a]pyrene	Applied	02/14/2018
EPA 625.1		Benzo[b]fluoranthene	Applied	02/14/2018
EPA 625.1		Benzo[ghi]perylene	Applied	02/14/2018
EPA 625.1		Benzo[k]fluoranthene	Applied	02/14/2018
EPA 625.1		Benzoic acid	Applied	02/14/2018
EPA 625.1		Benzyl alcohol	Applied	02/14/2018
EPA 625.1		Butyl benzyl phthalate (Benzyl butyl phthalate)	Applied	02/14/2018
EPA 625.1		Caprolactam	Applied	02/14/2018
EPA 625.1		Carbazole	Applied	02/14/2018
EPA 625.1		Chrysene (Benzo[a]phenanthrene)	Applied	02/14/2018
EPA 625.1		Di-n-butyl phthalate	Applied	02/14/2018
EPA 625.1		Di-n-octyl phthalate	Applied	02/14/2018
EPA 625.1		Dibenzo[a,h]anthracene	Applied	02/14/2018
EPA 625.1		Dibenzofuran	Applied	02/14/2018
EPA 625.1		Diethyl phthalate	Applied	02/14/2018
EPA 625.1		Dimethyl phthalate	Applied	02/14/2018
EPA 625.1		Fluoranthene	Applied	02/14/2018
EPA 625.1		Fluorene	Applied	02/14/2018
EPA 625.1		Hexachlorobenzene	Applied	02/14/2018
EPA 625.1		Hexachlorobutadiene (1,3-Hexachlorobutadiene)	Applied	02/14/2018
EPA 625.1		Hexachlorocyclopentadiene	Applied	02/14/2018
EPA 625.1		Hexachloroethane	Applied	02/14/2018
EPA 625.1		Indeno(1,2,3-cd)pyrene	Applied	02/14/2018
EPA 625.1		Isophorone	Applied	02/14/2018
EPA 625.1		N-Nitrosodi-n-propylamine	Applied	02/14/2018
EPA 625.1		N-Nitrosodimethylamine	Applied	02/14/2018
EPA 625.1		N-Nitrosodiphenylamine	Applied	02/14/2018
EPA 625.1		Naphthalene	Applied	02/14/2018
EPA 625.1		Nitrobenzene	Applied	02/14/2018
EPA 625.1		Pentachlorophenol (PCP)	Applied	02/14/2018
EPA 625.1		Phenanthrene	Applied	02/14/2018
EPA 625.1		Phenol	Applied	02/14/2018
EPA 625.1		Pyrene	Applied	02/14/2018
EPA 625.1		bis(2-Chloroethoxy)methane	Applied	02/14/2018
EPA 625.1		bis(2-Chloroethyl) ether	Applied	02/14/2018
EPA 625.1		bis(2-Ethylhexyl) phthalate (DEHP)	Applied	02/14/2018
EPA 8081	A	Organochlorine pesticides by GC/ECD	Withdrawn	03/21/2019
EPA 8270	C	SOCs by GC/MS	Withdrawn	03/21/2019
EPA 8270 SIM		1,4-Dioxane (1,4-Diethyleneoxide)	Withdrawn	03/21/2019
EPA 8270 SIM		Acenaphthene	Withdrawn	03/21/2019
EPA 8270 SIM		Acenaphthylene	Withdrawn	03/21/2019

EPA 8270 SIM	Anthracene	Withdrawn	03/21/2019
EPA 8270 SIM	Benzo[a]anthracene	Withdrawn	03/21/2019
EPA 8270 SIM	Benzo[a]pyrene	Withdrawn	03/21/2019
EPA 8270 SIM	Benzo[b]fluoranthene	Withdrawn	03/21/2019
EPA 8270 SIM	Benzo[ghi]perylene	Withdrawn	03/21/2019
EPA 8270 SIM	Benzo[k]fluoranthene	Withdrawn	03/21/2019
EPA 8270 SIM	Chrysene (Benzo[a]phenanthrene)	Withdrawn	03/21/2019
EPA 8270 SIM	Dibenzo[a,h]anthracene	Withdrawn	03/21/2019
EPA 8270 SIM	Fluoranthene	Withdrawn	03/21/2019
EPA 8270 SIM	Fluorene	Withdrawn	03/21/2019
EPA 8270 SIM	Indeno(1,2,3-cd)pyrene	Withdrawn	03/21/2019
EPA 8270 SIM	Naphthalene	Withdrawn	03/21/2019
EPA 8270 SIM	Phenanthrene	Withdrawn	03/21/2019
EPA 8270 SIM	Pyrene	Withdrawn	03/21/2019
EPA 8270 SIM	Quinoline	Withdrawn	03/21/2019

Matrix: Solid and Chemical Materials

Method	Revision	Analyte	Status	Effective Date
EPA 3051	A	Microwave digestion of solids (HNO3 + HCl)	Withdrawn	03/21/2019
EPA 3051		Microwave digestion of solids (HNO3 only)	Withdrawn	03/21/2019
EPA 6010	D	Metals by ICP/AES	Applied	02/14/2018
EPA 8081	A	Organochlorine pesticides by GC/ECD	Withdrawn	03/21/2019
EPA 8270	C	SOCs by GC/MS	Withdrawn	03/21/2019
EPA 8270 SIM		1,4-Dioxane (1,4-Diethyleneoxide)	Withdrawn	03/21/2019
EPA 8270 SIM		Acenaphthene	Withdrawn	03/21/2019
EPA 8270 SIM		Acenaphthylene	Withdrawn	03/21/2019
EPA 8270 SIM		Anthracene	Withdrawn	03/21/2019
EPA 8270 SIM		Benzo[a]anthracene	Withdrawn	03/21/2019
EPA 8270 SIM		Benzo[a]pyrene	Withdrawn	03/21/2019
EPA 8270 SIM		Benzo[b]fluoranthene	Withdrawn	03/21/2019
EPA 8270 SIM		Benzo[ghi]perylene	Withdrawn	03/21/2019
EPA 8270 SIM		Benzo[k]fluoranthene	Withdrawn	03/21/2019
EPA 8270 SIM		Chrysene (Benzo[a]phenanthrene)	Withdrawn	03/21/2019
EPA 8270 SIM		Dibenzo[a,h]anthracene	Withdrawn	03/21/2019
EPA 8270 SIM		Fluoranthene	Withdrawn	03/21/2019
EPA 8270 SIM		Fluorene	Withdrawn	03/21/2019
EPA 8270 SIM		Indeno(1,2,3-cd)pyrene	Withdrawn	03/21/2019
EPA 8270 SIM		Naphthalene	Withdrawn	03/21/2019
EPA 8270 SIM		Phenanthrene	Withdrawn	03/21/2019
EPA 8270 SIM		Pyrene	Withdrawn	03/21/2019
EPA 8270 SIM		Quinoline	Withdrawn	03/21/2019

pennsylvania
DEPARTMENT OF ENVIRONMENTAL PROTECTION

03/22/2019

William Billings
Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601

Re: Certificate of Accreditation
DEP Lab ID No. 65-00282

Dear Laboratory Supervisor:

Enclosed is your new Certificate of Accreditation to operate as a Pennsylvania Accredited Laboratory. This Certificate of Accreditation expires **03/31/2020** unless suspended or revoked earlier. As a laboratory accredited in accordance with the Environmental Laboratory Accreditation Act of June 29, 2002 (P.L 596, No 90) (27 Pa C.S. §§ 4101 – 4113) and The Environmental Laboratory Accreditation Regulations of 25 Pa. Code Chapter 252 you are responsible for continual compliance with the accreditation Act and regulations promulgated thereunder. Failure to comply with all applicable Federal and Departmental laws and regulations may result in suspension or revocation of your laboratory's accreditation.

Your DEP laboratory identification number is **65-00282**. Please use this number on all correspondence with the PA Department of Environmental Protection (Department).

Your laboratory is accredited to perform only the analyses by the methods listed on the Scope of Accreditation that accompanies the Certificate of Accreditation. The Certificate of Accreditation remains the property of the Department and must be displayed in the laboratory.

Please note this certification must be renewed annually. Renewal applications must be submitted to the Department *no later than 60 days prior to the expiration of the certification*. Failure to submit a renewal application within this time period may result in a lapse of the laboratory's accreditation. Should this occur, the laboratory may not conduct any further analyses for which accreditation is required and, if the laboratory is accredited to perform analyses on drinking water, the laboratory must notify the public water suppliers served by the laboratory of the laboratory's failure to renew its certificate of accreditation. Copies of the renewal application may be found on the Department's web site (www.depweb.state.pa.us/labs).

If you have any questions concerning your certificate, you may contact your laboratory's accreditation officer Mike Azar at 717-346-8206 or miazar@pa.gov.

Sincerely,

Aaren Alger

Aaren S. Alger, Chief
Laboratory Accreditation Program

Enclosures

Recd 3-28-19

pennsylvania
DEPARTMENT OF ENVIRONMENTAL PROTECTION

03/21/2019

William Billings
Pace Analytical Services LLC - Pittsburgh PA
1638 Roseytown Suites 2, 3, & 4
Greensburg, PA 15601

Re: Accreditation Status Change (A19-00282-01)
DEP Lab # 65-00282

Dear Laboratory Supervisor:

On January 22, 2019, the Laboratory Accreditation Program of the Pennsylvania Department of Environmental Protection ("Department") received a Part 4—Add FOA Application from your laboratory. The Department reviewed this application and associated materials for EPA 608.3 in the non-potable matrix. Your accreditation status in the Pennsylvania Environmental Laboratory Accreditation Program has changed due to your application request. Your current accreditation status is as shown on the attached listing. That list of accredited fields of testing replaces all previous lists.

Your laboratory shall not use this Scope of Accreditation to imply endorsement by the Department. In order to maintain accreditation, your laboratory must remain in compliance with Departmental regulations.

Any person aggrieved by this action may appeal, pursuant to Section 4 of the Environmental Hearing Board Act, 35 P.S. Section 7514, and the Administrative Agency Law, 2 Pa.C.S. Chapter 5A, to the Environmental Hearing Board, Second Floor, Rachel Carson State Office Building, 400 Market Street, P.O. Box 8457, Harrisburg, PA 17105-8457, 717-787-3483. TDD users may contact the Board through the Pennsylvania Relay Service, 800-654-5984. Appeals must be filed with the Environmental Hearing Board within 30 days of receipt of written notice of this action unless the appropriate statute provides a different time period. Copies of the appeal form and the Board's rules of practice and procedure may be obtained from the Board. The appeal form and the Board's rules of practice and procedure are also available in braille or on audiotape from the Secretary to the Board at 717-787-3483. This paragraph does not, in and of itself, create any right of appeal beyond that permitted by applicable statutes and decisional law.

IF YOU WANT TO CHALLENGE THIS ACTION, YOUR APPEAL MUST REACH THE BOARD WITHIN 30 DAYS. YOU DO NOT NEED A LAWYER TO FILE AN APPEAL WITH THE BOARD.

IMPORTANT LEGAL RIGHTS ARE AT STAKE, HOWEVER, SO YOU SHOULD SHOW THIS DOCUMENT TO A LAWYER AT ONCE. IF YOU CANNOT AFFORD A LAWYER, YOU MAY QUALIFY FOR FREE PRO BONO REPRESENTATION. CALL THE SECRETARY TO THE BOARD (717-787-3483) FOR MORE INFORMATION.

If you have any questions please contact your laboratory's accreditation officer Mike Azar at 717-346-8206 or miazar@pa.gov.

Sincerely,

Aaren Alger

Aaren S. Alger, Chief
Laboratory Accreditation Program

Enclosure

ATTACHMENT A-7

ANAB CERTIFICATE OF ACCREDITATION, U.S. DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V5.1.1)
PACE ANALYTICAL SERVICES, LLC-PITTSBURGH

CERTIFICATE OF ACCREDITATION

ANSI National Accreditation Board

11617 Coldwater Road, Fort Wayne, IN 46845 USA

This is to certify that

Pace Analytical Services, LLC – Pittsburgh PA

1638 Roseytown Road, Suites 2, 3 & 4
Greensburg, PA 15601

has been assessed by ANAB and meets the requirements of international standard

ISO/IEC 17025:2005

and the

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V5.1.1)

while demonstrating technical competence in the field of

TESTING

Refer to the accompanying Scope of Accreditation for information regarding the types of activities to which this accreditation applies

L2417
Certificate Number

ANAB Approval

Certificate Valid Through: 04/02/2022
Version No. 003 Issued: 03/18/2019

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005 AND U.S DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V5.1.1)

Pace Analytical Services, LLC - Pittsburgh PA

1638 Roseytown Road, Suites 2, 3 & 4
Greensburg, PA 15601
Nasreen DeRubeis
724-850-5630

TESTING

Valid to: **April 2, 2022** Certificate Number: **L2417**

Environmental

Drinking Water		
Technology	**Method**	**Analyte**
Alpha Spec	HASL 300 U-02-RCm	Isotopic Uranium (232, 233/234, 235/236, 238)
GFPC	EPA 900.0	Gross Alpha/Beta
GFPC	SM 7110C	Gross Alpha
Gamma Spec	EPA 901.1	Gamma Emitters
Gamma Spec	EPA 901.1	Barium-133, Cesium-134, Cesium-137, Cobalt-60 and Zinc-65
GFPC	EPA 903.0	Total Alpha Radium
Alpha scintillation Counter	EPA 903.1	Radium 226
GFPC	EPA 904.0	Radium-228
GFPC	EPA 905.0	Strontium 90
Liquid Scintillation Counter	EPA 906.0	Tritium
KPA	ASTM D5174-97	Uranium-Total
Liquid Scintillation Counter	SM 7500-Rn B	Radon-222

Non-Potable Water		
Technology	**Method**	**Analyte**
Alpha Spec	HASL 300 Am-04 RCm	Am-241, Am-243, Cm- 243/244, Cm-245/246, Cm-248
Alpha Spec	HASL 300 Pu-02-RCm	Isotopic Plutonium (236, 238, 239, 240, 241, 242, 244)
Liquid Scintillation	HASL 300 Pu-02-RCm	Pu-241
Alpha Spec	HASL 300 Th-01-RCm	Isotopic Thorium (228, 229, 230, 232)
Alpha Spec	HASL 300 U-02-RCm	Isotopic Uranium (232, 233/234, 235/236, 238)
GFPC	EPA 900.0	Gross Alpha/Beta
GFPC	SM 7110C	Gross Alpha
GFPC	EPA 9310	Gross Alpha/Beta
Gamma Spec	EPA 901.1	Gamma Emitters
Gamma Spec	EPA 901.1	Barium-133, Cesium-134, Cesium-137, Cobalt-60 and Zinc-65
GFPC	EPA 903.0	Total Alpha Radium
GFPC	EPA 9315	Total Radium
GFPC	EPA 9315	Radium 226
Alpha scintillation Counter	EPA 903.1	Radium 226
GFPC	EPA 904.0	Radium-228
GFPC	EPA 9320	Radium-228
GFPC	EPA 905.0	Strontium 90
GFPC	Eichrom Method SRW01	Strontium 90
Liquid Scintillation Counter	EPA 906.0	Tritium
KPA	ASTM D5174-97	Uranium-Total
Liquid Scintillation Counter	SM 7500-Rn B	Radon-222
Liquid Scintillation Counter	Liquid Scintillation	Carbon-14
GFPC	RP 280 DOE	Lead-210
Alpha Spec	HASL 300 Po-01-RC and Po-02-RC	Polonium-210

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1	Gamma Emitters
Alpha Spec	HASL 300 Am-04-RCm	Am-241, Am-243, Cm- 243/244, Cm-245/246, Cm-248
Alpha Spec	HASL 300 Pu-02-RCm	Isotopic Plutonium (236, 238, 239, 240, 242, 244)
Liquid Scintillation	HASL 300 Pu-02-RCm	Pu-241

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1	Ra-226
Gamma Spec	EPA 901.1	Ra-228
Alpha Spec	HASL 300 Th-01-RCm	Isotopic Thorium (228, 229, 230, 232)
Alpha Spec	HASL 300 U-02-RCm	Isotopic Uranium (232, 233/234, 235/236, 238)
GFPC	Eichrom Method SRW01	Strontium 90
Liquid Scintillation Counter	Liquid Scintillation	Carbon-14
GFPC	EPA 900.0	Gross Alpha/Beta
GFPC	EPA 9310	Gross Alpha/Beta
Liquid Scintillation Counter	EPA 906.0 modified	Tritium
GFPC	RP 280 DOE	Lead-210
Alpha Spec	HASL 300 Po-01-RC and Po-02-RC	Polonium-210

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2417

Vice President

ATTACHMENT A-8

LABORATORY ACCREDITATION & CERTIFICATION
PACE ANALYTICAL ENERGY SERVICES, LLC, PITTSBURGH

Pace Analytical Energy Services, LLC
220 William Pitt Way
Pittsburgh, PA 15238
(412) 826-5245

LABORATORY ACCREDITATIONS & CERTIFICATIONS

ACCREDITOR:	Pennsylvania Department of Environmental Protection, Bureau of Laboratories
REGISTRATION NO.:	02-00538
SCOPE:	NELAP Non-Potable Water
EXPIRATION DATE:	November 30, 2019

ACCREDITOR:	NELAP: State of Virginia, Dept. of General Services/Div. of Consolidated Lab Serv.
ACCREDITATION ID:	460201
SCOPE:	Non-Potable Water
EXPIRATION DATE:	December 14, 2019

ACCREDITOR:	South Carolina Department of Health and Environmental Control, Office of Environmental Laboratory Certification
ACCREDITATION ID:	Certificate No. 89009003
SCOPE:	Clean Water Act (CWA); Resource Conservation and Recovery Act (RCRA)
EXPIRATION DATE:	November 30, 2019

ACCREDITOR:	NELAP: New Hampshire, Environmental Laboratory Accreditation Program
ACCREDITATION ID:	Certificate No. 299409
SCOPE:	Non-potable water
EXPIRATION DATE:	August 5, 2019

ACCREDITOR:	NELAP: New Jersey, Department of Environmental Protection
ACCREDITATION ID:	PA026
SCOPE:	Non-Potable Water;
EXPIRATION DATE:	June 30, 2019

ACCREDITOR:	NELAP: New York, Department of Health Wadsworth Center
ACCREDITATION ID:	11815
SCOPE:	Non-Potable Water;
EXPIRATION DATE:	April 1, 2020

ACCREDITOR:	NELAP: Connecticut, Department of Public Health, Division of Environmental Health
ACCREDITATION ID:	Certificate No. PH-0263
SCOPE:	Clean Water Act (CWA) Resource Conservation and Recovery Act (RCRA)
EXPIRATION DATE:	December 31, 2020

ACCREDITOR:	NELAP: Texas, Commission on Environmental Quality
ACCREDITATION ID:	Certificate No. T104704453-09-TX
SCOPE:	Non-Potable Water
EXPIRATION DATE:	December 31, 2019

ACCREDITOR:	West Virginia, Dept. of Environmental Protection
ACCREDITATION ID:	Certificate No. 395
SCOPE:	Non-Potable Water
EXPIRATION DATE:	May 31, 2019

SCHEDULE 1.1 (i)
Pace Analytical Energy Services, LLC
220 William Pitt Way
Pittsburgh, PA 15238
(412) 826-5245

LABORATORY ACCREDITATIONS & CERTIFICATIONS

COMMERCIAL LABORATORY STIPULATION: Georgia Rules for Commercial Environmental Laboratory Accreditation Chapter 391-3-26: As per the Georgia EPD Rules and Regulations for Commercial Laboratories, Pace Analytical Energy Services, LLC is accredited by the Pennsylvania Department of Environmental Protection Bureau of Laboratories under the National Environmental Laboratory Approval Program (NELAC). If you have any further questions regarding accreditation status for Pace Analytical Energy Services, LLC please contact your Pace Analytical Energy Services, LLC Project Manager.

ATTACHMENT A-9

PERRY JOHNSON LABORATORY ACCREDITATION, INC., CERTIFICATE OF ACCREDITATION, MATERIAL AND CHEMISTRY LABORATORY, INC., OAK RIDGE, TENNESSEE

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

Materials and Chemistry Laboratory, Inc. (MCLinc)

2010 Highway 58, Suite 1000, Oak Ridge, TN 37830

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005) "General Requirements for the competence of Testing and Calibration Laboratories" and the DOE Quality Systems Manual for Environmental Laboratories Version 5.1.1, 2018 and is accredited is accordance with the:

United States Department of Energy Consolidated Audit Program-Accreditation Program (DOECAP-AP)

This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system
(as outlined by the joint ISO-ILAC-IAF Communiqué dated April 2017):

Industrial Hygiene and Environmental Testing
(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
July 27, 2018	July 27, 2018	August 31, 2020

Accreditation No.:	*Certificate No.:*
99882	L18-352

Tracy Szerszen
President/Operations Manager

Perry Johnson Laboratory Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DOECAP-AP

Materials and Chemistry Laboratory, Inc. (MCLinc)

2010 Highway, Suite 1000, Oak Ridge, TN 37830

Contact Name: Jack R. Hall Phone: 865-576-4138

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Filters and Wipes	NIOSH 7902, MCL-7775 Appendix 29	Specific Ion Electrode	Fluorides
Liquids	EPA 3620B, MCL-7740, EPA 8082	Florisil Cleanup	PCBs
Liquids	EPA 3665A, MCL-7740 and EPA 8082	Sulfuric Acid Cleanup	PCBs
Solids, liquids, filters ,and wipes	EPA 6020 and EPA 6010, and *related ASTM methods	ICP/OES and ICP/MS	Metals Assay
Solids	Internal Published Method referenced in MCL-7738	Pyrolysis Apparatus	Anion Prep
Solids	SM 2450	Drying, filtering	Solids
Solids	UCOR Method	Radionuclides and Metals	Acid Leachates
Water	EPA 100.2; and *related ASTM methods	Transmission Electron Microscopy	Asbestos
Water	SM 4500-Cl modified for micro	Microtitrator	Total Residual Chlorine
Liquids, Filters, and Wipes	EPA 900, ASTM 2459,D 7902, MCL-7734	Gas Proportional Counting	Gross Alpha/Beta
Liquids and Solids	ASTM C-1771; MCL-7768	ICP/MS of Hydrolyzed Uranium Hexafluoride	Boron Silica and Tc-99
Liquids and Solids	*Related ASTM methods, Client	Thermal Gravimetric Analysis (TGA)/MS	Weight Loss on heating Compound ID
Liquids and Solids	ASTM C-1267, MCL-7737, MCL7737A	Davies-Gray Titration	Total Uranium
Liquids and Solids	DOE Methods Compendium RP550 and MCL-7754	Liquid Scintillation Counting	Tc-99
Liquids and Solids	EPA 1311, MCL-7743	TCLP	Leachates
Liquids and Solids	EPA 6020M, MCL-7768 and *related ASTM methods	ICP/MS	Tc-99
Liquids and Solids	M -EPA 6020, MCL-7768	ICP/MS	Lithium Isotopic
Liquids and Solids	M-EPA 6020, MCL-7768	ICP/MS	Boron Isotopic
Liquid, Solids, Filters and Wipes	EPA 3050B, MCL-7746 and MCL-7752	Acid Digestion Hot Block	Metals Sample Preparation
Liquid, Solids, Filters and Wipes	EPA 6010; ASTM D-1076, MCL-7751	Inductively Coupled Plasma - Optical Emission Spectroscopy	Metals
Liquid, Solids, Filters and Wipes	EPA-8082; NIOSH 5503; ASTM 5175, MCL-7740	GC/EC	PCBs
Solids, Filters and Wipes	*Related ASTM methods, Client specified	Scanning Electron Microscopy/EDS	Elemental Analysis
Solids, Filters and Wipes	NIOSH 7400, MCL-7721	Phase Contrast Microscopy	Asbestos
Solids, Filters and Wipes	NIOSH 7402, MCL-7708	Transmission Electron Microscopy	Asbestos
Solids, Filters and Wipes	NIOSH 7500; and *related ASTM methods	X-Ray Diffraction	Silica

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DOECAP-AP

Materials and Chemistry Laboratory, Inc. (MCLinc)

2010 Highway, Suite 1000, Oak Ridge, TN 37830
Contact Name: Jack R. Hall Phone: 865-576-4138

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Water and Solids	SM 2310, 2320	pH meter	pH, titrations
Water, Liquids, and Solids	MCL-7748,ASTM 412, D7042 and Instrument Manuals	Regular and Micro Kinematic Viscosity	Viscosity
Water, Liquids, and Solids	Modified EPA 1010; ASTM D3278, ASTM D3828	Micro Flashpoint, closed cup	Flashpoint
Water, Liquids, and Solids	Nicolet, *related ASTM methods	Fourier Transform Infrared (FTIR)	Organic Analysis
Water, Solids, Filter and Wipes	EPA 300; 9056; ASTM D-808; NIOSH 7903 and MCL-7739	Ion Chromatography	Anions
Water, Solids, Filter and Wipes	EPA 6010, MCL-7751 OSHA ID-125G	Inductively Coupled Plasma - Optical Emission Spectroscopy	Beryllium Testing + BeO
Water, Solids, Filter and Wipes	EPA 6020, MCL-7768	Inductively Coupled Plasma - Mass Spectroscopy	Beryllium Testing + BeO
Water, Solids, Filter and Wipes	EPA 6020, MCL-7768 NIOSH 7303	Inductively Coupled Plasma - Mass Spectroscopy	Metals
Water, Solids, Filter and Wipes	NIOSH 6009, EPA and *related ASTM methods	Cold Vapor Atomic Absorption	Mercury
Water, Solids, Filter and Wipes	NIOSH 7300, NIOSH 7301, NIOSH 7303, MCL-7751	Inductively Coupled Plasma - Optical Emission Spectroscopy	Metals
Water, Solids, Filter and Wipes	NIOSH 9002, MCL-7720	Polarized Light Microscopy	Asbestos
Water, Solids, Filter and Wipes	OSHA-215, ASTM 5267, EPA 7199, and MCL-7770	Ion Chromatography	Hexavalent Chromium
Water, Liquids, Solids, Filters and Wipes	EPA 6020, ASTM C-1345, C1474, MCL-7769	Inductively Coupled Plasma - Mass Spectrometry	Isotopic Uranium
Water, Liquids, Solids, Filters and Wipes	MCL-7759, ASTM D7283, C1539, D4922, ANSI 42.15 and EPA 913	Liquid Scintillation Counting	Gross Alpha/Beta

1. * "Accreditation is granted through a technology based flexible scope criteria. Additional methods other than listed above may fall under the accreditation of the laboratory. A complete listing of method capabilities can be derived from the laboratory upon request."

PERRY JOHNSON LABORATORY ACCREDITATION, INC .
755 West Big Beaver Road, Suite 1325
Troy, MI 48084

CONTRACT AMENDMENT

As evidenced by the signatures of the parties below, Perry Johnson Laboratory Accreditation, Inc., (PJLA) and Materials and Chemistry Laboratory, Inc. (MCLinc) (ORGANIZATION), agree to amend certain terms of their contract for services dated 02/13/2018. All terms of the contract not referenced below remain in full force and effect as if fully restated herein.

CONTRACT SECTION(s) TO BE AMENDED:

Section(s)	Amendment to read:
ALL	During the 2019 Surveillance assessment, laboratory will be transitioning to QSM 5.2. No additional time is required and current contracted time will remain the same. However, a $285.00 accreditation fee will be applied to this assessment.

This amendment must be signed and returned within 14 business days or it will become invalid.
Effective date for amendments: 04/11/2019

04/11/2019

Signatory
Perry Johnson Laboratory Accreditation, Inc.

4/15/19

(Signature) (Date)
For ORGANIZATION

Reviewed by:_____

ATTACHMENT B

DATA VALIDATION VARIANCE DOCUMENTATION

Attachment B. Data Validation Variance Documentation

Most data are evaluated in accordance with the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFGs) documentation and USEPA Methods; however, professional judgment, outside of the NFGs, is often necessary when making decisions regarding data quality. The tables below are intended to specifically document how Trihydro Corporation's Chemical Data Evaluation Service's (CDES) group interprets the areas where professional judgment is recommended in the NFGs or where Trihydro may validate differently from the specific rules noted in the NFGs. The tables are grouped by compound type (i.e., organic and inorganic).

- Data for organic analyses were evaluated in general accordance with validation criteria set forth in the USEPA Contract Laboratory Program (CLP) NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017 with additional reference to the USEPA CLP NFGs for Organic Data Review, document number EPA 540/R-99/008, October 1999. When additional reference is necessary for interpretation of the analytical data, the analytical method may be used as an additional resource although analytical methods do not provide guidance for qualification of data.
- Data for inorganic analyses were evaluated in general accordance with the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017 with additional reference to the USEPA CLP NFGs for Inorganic Data Review, document number EPA-540-R-04-004, October 2004. When additional reference is necessary for interpretation of the analytical data, the analytical method may be used as an additional resource although analytical methods do not provide guidance for qualification of data.
- Review of field duplicates was conducted in accordance with the USEPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures, EQADR-Supplement0, April 2013.

The following table contains the flags used during the data validation process and the associated flag definitions.

Flag Code	Flag Definition
J	Estimated concentration
J+	The result is an estimated concentration, but may be biased high
J-	The result is an estimated concentration, but may be biased low
UJ	Estimated reporting limit
U	Evaluated to be undetected at the reporting limit
JB	Estimated concentration due to blank contamination
R	Rejected, data not usable
NJ	Tentative identification and estimated concentration

Updated: February 2019

Attachment B. Data Validation Variance Documentation

Organic Analyses

The organic guidelines used to determine the quality of the data are in accordance with the NFGs and USEPA Methods for the analysis of volatile organic compounds, semivolatile organic compounds, pesticide compounds, and Aroclor compounds.

Preservation Criteria

Review Items:

a) pH
b) Sample Temperature
c) Holding Time
d) Other Sample Conditions (e.g., headspace)

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. The variances listed below are applicable to the preservation review items. Items noted in bold type indicate variances due to professional judgment.

Out of control review items noted above will result from the use of technical holding times. Sample holding times defined in days will be evaluated in days (independent of hours and minutes elapsed), sample holding times published in hours will be measured to the hour, etc.

TABLE 1. HOLDING TIME ACTIONS FOR TRACE VOLATILE ANALYSES

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	Yes	Greater than 14 days	**J-**	**R**
Aqueous*	Yes, but the VOA contains headspace greater than a dime size.	Greater than 7 days**	**J -**	**R**
Aqueous	No, pH > 2 at the time of analysis.	Greater than 7 days	**J -**	**R**

*** Qualification regarding sample headspace is subject to professional judgment. Hence, if sample condition does not allow zero headspace other action may be taken.**

**** Qualification regarding sample headspace is subject to professional judgment. Sample holding time may not be considered.**

NFG Section E Items 1a), b), c), and d): Gross holding time exceedances (doubling the technical hold and/or extraction time) will result in the rejection (R) of all data for the sample.

Attachment B. Data Validation Variance Documentation

TABLE 2. HOLDING TIME ACTIONS FOR LOW/MEDIUM VOLATILE ANALYSES

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	Yes	Greater than 14 days	**J-**	**R**
Aqueous*	Yes, but the VOA contains headspace greater than a dime size.	Greater than 7 days**	**J-**	**R**
Aqueous	No, pH > 2 at the time of analysis.	Greater than 7 days	**J-**	**R**

*** Qualification regarding sample headspace is subject to professional judgment. Hence, if sample condition does not allow zero headspace other action may be taken.**

**** Qualification regarding sample headspace is subject to professional judgment. Sample holding time may not be considered.**

NFG Section E Items 1a), b), c), and d): Gross holding time exceedances (doubling the technical hold and/or extraction time) will result in the rejection (R) of all data for the sample.

TABLE 3. HOLDING TIME ACTIONS FOR SEMIVOLATILES/PESTICIDE ORGANIC ANALYSES

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	Yes / No	Greater than 7 days (for extraction) and/or Greater than 40 days (for analysis)	**J**	**UJ**
Non-Aqueous	Yes / No	Greater than 14 days (for extraction) and/or Greater than 40 days (for analysis)	**J**	**UJ**
Aqueous	Yes / No	Grossly Exceeded (more than two times the holding time)	**J**	**R**
Non-Aqueous	Yes / No	Grossly Exceeded (more than two times the holding time)	**J**	**R**

Some analytical extraction and holding times are defined only in units of days, such as the 14-day holding time for preserved volatiles samples. In determining if samples were extracted/analyzed within acceptable holding times, a holding time of 14 days is interpreted to be equal to 14 days after the date of sampling, independent of sampling time. Data validation actions based on differences between laboratory acceptable holding times and the method holding times, as defined above, are described in Table 4, below.

Apply bias indicators (J+, J-) only if behavior of analyte(s) is known and documented.

NFG Section E Items 1a), b), c), and d): Gross holding time exceedances (doubling the technical hold and/or extraction time) will result in the rejection (R) of all data for the sample.

Aroclor holding times are now one year for properly preserved samples.

Attachment B. Data Validation Variance Documentation

Preservation Criteria

TABLE 4. HOLDING TIME ACTIONS FOR DISCREPANCIES BETWEEN LABORATORY HOLDING TIMES AND METHOD HOLDING TIMES

Analysis Within Laboratory Hold/Extraction Time?	Analysis Within Method Hold/Extraction Time?	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds
Yes	Yes	None	None
Yes	No	**J**	**UJ**
No	No	**J**	**UJ or R**

TABLE 5. TEMPERATURE ACTIONS FOR ORGANIC ANALYSES

	< 0°C Frozen	0-2° Not Frozen	2-6°C	6-10°C	10-20°C	>20°C
Volatiles	Reject	no qual	no qual	J- / UJ*	J- / UJ / REJECT*	Reject
SVOCs	Reject	no qual	no qual	J / UJ*	J / UJ*	Reject
Metals	no qual	no qual	no qual	no qual	no qual	no qual
Inorganics	Analyte dependent	no qual	no qual	Analyte dependent	Analyte dependent	Analyte dependent

***Temperatures >6˚C but ≤10˚C may be accepted based on the professional judgment of the validator. Application and selection of qualifiers for volatile and semivolatile is based on validator's professional judgment.**

Validator may elect to not qualify samples if they were hand delivered to the laboratory and did not have sufficient time to cool (less than 24 hours from last collection time). Qualification is at the discretion of the validator.

TABLE 6. SOIL GAS VAPOR INTRUSION HELIUM CONCENTRATION ACTIONS

Matrix	Criteria	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds
Soil Gas	Sample had helium concentration greater than 10% of the concentration in the shroud *	**R if analyzed**	**R if analyzed**
Soil Gas	Sample had helium concentration greater than 5%, but less than or equal to 10% of the concentration in the shroud *	**J**	**UJ**
Soil Gas	Sample had concentration of helium less than 5% of the concentration in the shroud *	No qualification	No qualification

*** Unless other limits are specified by project requirements.**

Attachment B. Data Validation Variance Documentation

Gas Chromatograph/Mass Spectrometer (GC/MS) and GC Electron Capture Detector (ECD) Instrument Performance Check
Review Items: a) Bromofluorobenzene (BFB) mass spectra and mass listing. b) Semi-Volatiles, decafluorotriphenylphosphine (DFTPP) mass spectra, and mass listing. c) Chromatograms and data system printouts.
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Initial Calibration

Review Items:

a) Initial Calibration Verification Result Recoveries (if applicable)
b) Time of Analyses for Initial Calibration
c) Mean Relative Response Factor (RRF) Results
d) Relative Standard Deviation (%RSD) Results
e) Chromatograms and Quantitation Reports

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.

Trihydro action variances from standard U.S. EPA criteria:

Data validation criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

TABLE 7. INITIAL CALIBRATION ACTION FOR PESTICIDE ANALYSES

Criteria	Action**	
	Detected Associated Compounds	Non-Detected Associated Compounds
Initial Calibration was not performed in the proper sequence	R	R
%RSD exceeds allowable limits*	J	UJ

* %RSD ≤ 20.0% for single component target compounds except alpha-BHC and delta-BHC.
* %RSD ≤ 25.0% for alpha-BHC and delta-BHC. %RSD ≤ 30.0% for Toxaphene peaks.
* %RSD ≤ 30.0% for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

** - If the confirmation column is not used for either identification or quantification, no qualification will be necessary.

TABLE 8. INITIAL CALIBRATION ACTION FOR AROCLOR ANALYSES

Criteria	Action**	
	Detected Associated Compounds	Non-Detected Associated Compounds
Initial Calibration was not performed in the proper sequence	R	R
%RSD exceeds allowable limits*	J	UJ

* %RSD ≤ 20.0% for Aroclors.
* %RSD ≤ 20.0% for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

** - If the confirmation column is not used for either identification or quantification, no qualification will be necessary.

Attachment B. Data Validation Variance Documentation

Initial Calibration Verification
Review Items: a) Initial Calibration Verification Result Recoveries (if applicable) b) Mean Relative Response Factor (RRF) Results c) Percent Difference (%D) Results d) Chromatograms and Quantitation Reports (if necessary)
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section. If the confirmation column is not used for either identification or quantification, no qualification will be necessary.

Continuing Calibration Verification
Review Items: a) Continuing Calibration Verification Result Recoveries (if applicable) b) Time of Analyses for Continuing Calibration Verification Results c) Mean Relative Response Factor (RRF) Results d) Percent Difference (%D) Results e) Chromatograms and Quantitation Reports (if necessary)
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section. If the confirmation column is not used for either identification or quantification, no qualification will be necessary. **For Method 8260 and Method 8270 analytes not included in the tables in the NFGs, the limit for %D should be 25%.**

Attachment B. Data Validation Variance Documentation

Blanks

Review Items:

a) Method, Trip, Equipment, and Field Blank Concentrations

b) Comparable Sample Results

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.

Trihydro action variances from standard U.S. EPA criteria:

The below mentioned data validation variances are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. Items noted in bold type indicate variances due to professional judgment.

Please note that if a project requires that the samples be reported to the method detection limit as the reporting limit, the U flag will not be used and the JB will be utilized instead.

TABLE 9. BLANK ACTIONS FOR TRACE VOLATILES, LOW-LEVEL VOLATILES SEMIVOLATILES, PESTICIDES, AND AROCLOR ANALYTES

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field, Equipment, Trip	Detect below the laboratory reporting limit	Detect below the laboratory reporting limit and/or below the blank concentration	**Report result with a U qualifier at the laboratory reporting limit**
Method, Field, Equipment, Trip	Detect below the laboratory reporting limit	Non-detect	No Qualification
Method, Field, Equipment, Trip	Detect below the laboratory reporting limit	Detect above or equal to the laboratory reporting limit but below or equal to 10 times the blank concentration	**Report result with a JB qualifier**
Method, Field, Equipment, Trip	Detect above or equal to the laboratory reporting limit	Detect below the laboratory reporting limit	**Report result with a U qualifier at the laboratory reporting limit**
Method, Field, Equipment, Trip	Detect above or equal to the laboratory reporting limit	Non-detect	No Qualification
Method, Field, Equipment, Trip	Detect above or equal to the laboratory reporting limit	Detect above or equal to the laboratory reporting limit but below or equal to 10 times the blank concentration	**Report result with a JB qualifier**
Method, Field, Equipment, Trip	Detect above or equal to the laboratory reporting limit	Detect below or equal to the blank concentration	**Report result with a U qualifier at the detection amount**

Attachment B. Data Validation Variance Documentation

Deuterated Monitoring Compounds (DMC) and Surrogate Spike Compounds

Review Items:

a) Surrogate or Deuterated Monitoring Compound Recovery Results

b) Surrogate or Deuterated Monitoring Compound Results

c) Chromatograms and Quantitation Reports

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. Items noted in bold type indicate variances due to professional judgment.

TABLE 10. DMC RECOVERY ACTIONS FOR VOLATILE ANALYSES

Criteria*	Action*	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R Less than Lower Acceptance Limit	**J -**	**UJ**
%R Less than 20%	**J -**	**R**

***If a list of associated analytes is not available from the laboratory, flag all analytes for the analytical method if one or more surrogate(s) is outside the laboratory quality control limits.**

TABLE 11. DMC RECOVERY ACTIONS FOR SEMIVOLATILE ANALYSES

Criteria	Action*	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R Less than Lower Acceptance Limit	**J -**	**UJ**
%R Less than 10%	**J -**	**R**

***If a list of associated analytes is not available from the laboratory, flag analytes according to the type of surrogate exceeding the laboratory quality control limits. Flag data only if two of the three base/neutral surrogates are outside of the laboratory quality control limits. In this case, flag all associated base/neutral analytes. If two of the three acid surrogates are outside of the laboratory quality control limits, qualify all associated acid analytes.**

*** If available, determine if diluted surrogate concentration is below the calibration range. If the surrogate is below the calibration range in the diluted sample, do not qualify for surrogate recovery.**

Attachment B. Data Validation Variance Documentation

Deuterated Monitoring Compounds (DMC) and Surrogate Spike Compounds

TABLE 12. SURROGATE ACTIONS FOR PESTICIDE ANALYSES

Criteria	Action*	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R Greater than 200%	**J +**	No qualification
%R Less than 10% (when sample dilution is not a factor*)	**J -**	**R**

***If a list of associated analytes is not available from the laboratory, flag all analytes for the analytical method.**

*** If available, determine if diluted surrogate concentration is below the calibration range. If the surrogate is below the calibration range in the diluted sample, do not qualify for surrogate recovery.**

TABLE 13. SURROGATE ACTIONS FOR AROCLOR ANALYSES

Criteria	Action*	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R Greater than 200%	**J +**	No qualification
%R Less than 10% (when sample dilution is not a factor)	**J -**	**R**

***If a list of associated analytes is not available from the laboratory, flag all analytes for the analytical method.**

*** If available, determine if diluted surrogate concentration is below the calibration range. If the surrogate is below the calibration range in the diluted sample, do not qualify for surrogate recovery.**

Attachment B. Data Validation Variance Documentation

Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

Review Items:

a) MS/MSD Recoveries
b) MS/MSD Relative Percent Difference (RPD) Values
c) MS/MSD Preparation Samples
d) MS/MSD Raw Results
e) Chromatograms and Quantitation Reports

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.

The NFGs provide control limits for only a small number of the organic target analytes; therefore, laboratory-generated control limits should be used to evaluate performance of MS/MSD analyses.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. Items noted in bold type indicate variances due to professional judgment.

TABLE 14. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ACTIONS FOR TRACE VOLATILES LOW-LEVEL VOLATILES, SEMIVOLATILES, PESTICIDES, AND AROCLOR ANALYTES

Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R above the Upper Acceptance Limit**	**J +**	No Qualification
%R value below the Lower Acceptance Limit**	**J -**	**UJ**
For Volatiles, %R Less than 20%**	**J -**	**UJ and R***
For Semivolatiles, %R Less than 10%**	**J -**	**UJ and R***
MS/MSD RPD value above the Upper Acceptance Limit**	**J**	**UJ**

***Flag non-detect results for the MS/MSD parent sample with an R flag. Flag other associated non-detect results as UJ.**

****** Qualify ALL samples in the preparation batch.**

Attachment B. Data Validation Variance Documentation

Laboratory Control Samples/Laboratory Control Sample Duplicates (LCS/LCSD)

Review Items:

a) LCS/LCSD Recoveries
b) LCS/LCSD Relative Percent Difference (RPD) Values (If applicable)
c) LCS/LCSD Raw Results
d) Chromatograms and Quantitation Reports

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017 (pesticides and PCBs) and in USEPA CLP NFGs for Organic Data Review; document number EPA 540/R-99/008, October 1999 (volatiles and semivolatiles).

The NFGs provide control limits for only a small number of the organic target analytes; therefore, laboratory-generated control limits should be used to evaluate performance of LCS/LCSD analyses.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. The variances listed below are applicable to the LCS/LCSD review items. Items listed in bold type indicate variances due to professional judgment.

TABLE 15. LCS/LCSD ACTIONS FOR TRACE VOLATILES, LOW-LEVEL VOLATILES, SEMI-VOLATILES PESTICIDES, AND AROCLOR ANALYTES

Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R above the Upper Acceptance Limit*	**J +**	No Qualification
RPD value above the Upper Acceptance Limit (if applicable)	**J**	**UJ**
%R below the Lower Acceptance Limit*	**J -**	**UJ**
%R Less than 30% (Volatiles)	**J -**	**R**
%R Less than 10% (Semi-Volatiles)	**J -**	**R**

*** But above 30% for volatiles and 10% for semi-volatiles.**

Associations are based on preparation batches not analytical batches.

Attachment B. Data Validation Variance Documentation

Laboratory Duplicates

Review Items:

a) Chromatogram Results

b) Quantitation Reports

c) RPD Values

Criteria:

Use laboratory limits or limits defined in project QAPP, if available.

Criteria for duplicate analyses are not defined in the USEPA CLP NFGs or the USEPA New England Environmental Data Review Supplement.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment. Validator professional judgment may vary due to specific project data quality objectives. The variances listed below are applicable to the Laboratory Duplicate review items. Items noted in bold type indicate variances due to professional judgment.

If acceptance limits are not defined in the laboratory report or the project QAPP, use professional judgement to qualify data.

TABLE 16. LABORATORY DUPLICATE ACTIONS FOR TRACE VOLATILES, LOW-LEVEL VOLATILES SEMI-VOLATILES, PESTICIDES, AND AROCLOR ANALYTES

Criteria	Action	
	Detected Compounds	Non-Detected Compounds
RPD value is greater than laboratory limits or QAPP limits and both results are greater than five times the reporting limit **	**J**	**UJ**
RPD value is greater than laboratory limits or QAPP limits and one or both results are less than five times the reporting limit	No Qualification	
RPD value is less than laboratory limits or QAPP limits	No Qualification	

**** Qualify ALL samples in the preparation batch.**

Attachment B. Data Validation Variance Documentation

Field Duplicates

Review Items:

a) Chromatogram Results

b) Quantitation Reports

Criteria:

The criteria are identical to those noted in the USEPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures, EQADR-Supplement0, April 2013.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. The variances listed below are applicable to the Field Duplicate review items. Items noted in bold type indicate variances due to professional judgment.

TABLE 17. FIELD DUPLICATE ACTIONS FOR TRACE VOLATILES, LOW-LEVEL VOLATILES SEMI-VOLATILES, PESTICIDES, AND AROCLOR ANALYTES

Matrix		
Water		
Criteria	**Action**	
	Detected Compounds	**Non-Detected Compounds**
The analyte RPD value is greater than 100%*	**J**	**UJ**
RPD value is greater than 30% and one or both results are greater than two times the reporting limit (Flag the parent and duplicate samples only)	**J**	Not Applicable
One sample is non-detect and the other is detect. The detected value is greater than two times the reporting limit (Flag the parent and duplicate samples only)	**J**	**UJ**
One sample is non-detect and the other is detect. The detected value is less than or equal to two times the reporting limit	No Qualification	
RPD value is greater than 30% and both results are less than two times the reporting limit	No Qualification	
RPD value is less than 30%	No Qualification	

***All samples for this day of sampling will be qualified.**

This guidance is applicable unless other regulatory or project–specific) guidance is available (e.g. TRRP or QAPP).

If multiple field duplicates are collected in a sample set, associations are based on date of collection.

Attachment B. Data Validation Variance Documentation

Field Duplicates

TABLE 18. FIELD DUPLICATE ACTIONS FOR TRACE VOLATILES, LOW-LEVEL VOLATILES, SEMI-VOLATILES PESTICIDES, AND AROCLOR ANALYTES

Matrix		
Soil		
Criteria	Action	
	Detected Compounds	Non-Detected Compounds
The analyte RPD value is greater than 100%*	J	UJ
RPD value is greater than 50% and one or both results are greater than two times the reporting limit (Flag the parent and duplicate samples only)	J	UJ
One sample is non-detect and the other is detect. The detected value is greater than two times the reporting limit (Flag the parent and duplicate samples only)	J	UJ
One sample is non-detect and the other is detect. The detected value is less than or equal to two times the reporting limit	No Qualification	
RPD value is greater than 50% and both results are less than two times the reporting limit	No Qualification	
RPD value is less than 50%	No Qualification	

***All samples for this analyte will be qualified.**

This guidance is applicable unless other regulatory or project–specific) guidance is available (e.g. TRRP or QAPP).
If multiple field duplicates are collected in a sample set, associations are based on date of collection.

Attachment B. Data Validation Variance Documentation

Field Duplicates

TABLE 19. FIELD DUPLICATE ACTIONS FOR TRACE VOLATILES, LOW-LEVEL VOLATILES, SEMI-VOLATILES PESTICIDES, AND AROCLOR ANALYTES

Matrix		
Air		
Criteria	Action	
	Detected Compounds	Non-Detected Compounds
The analyte RPD value is greater than 100%*	J	UJ
One sample is non-detect and the other is detect. The detected value is greater than two times the reporting limit (Flag the parent and duplicate samples only)	J	UJ
RPD value is greater than 25% and one or both results are greater than two times the reporting limit (Flag the parent and duplicate samples only)	J	UJ
RPD value is greater than 25% and both results are less than two times the reporting limit	No Qualification	
RPD value is less than 25%	No Qualification	

***All samples for this analyte will be qualified.**

This guidance is applicable unless other regulatory or project–specific) guidance is available (e.g. TRRP or QAPP).

If multiple field duplicates are collected in a sample set, associations are based on date of collection.

Attachment B. Data Validation Variance Documentation

Internal Standards
Review Items: a) Retention Time Variance b) Area Counts
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Target Compound Identification
Review Items: a) Relative Retention Times b) Mass Spectra Results c) Retention Times d) Chromatograms e) Data System Printouts
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Tentatively Identified Compounds (TICs)
Review Items: a) Chromatograms b) Library search printouts c) Spectra for the TIC candidates
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Gel Permeation Chromatography (GPC) Performance Check
Review Items: a) Two Ultraviolent (UV) Traces b) GPC Quantitation Blank Quantitation Reports c) Chromatograms
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the respective NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Inorganic Analyses

The inorganic guidelines used to determine the quality of the data are in accordance with the NFGs and USEPA Methods for the analysis of metals, cyanide, and other inorganic analytes.

Preservation Criteria

Review Items:

a) pH
b) cooler temperature
c) holding time
d) other sample conditions

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.

Trihydro action variances from standard U.S. EPA criteria:

Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

a) b) c) d) Gross holding time exceedances (doubling the technical hold and/or extraction time) will result in the rejection (R) of all data for the sample. Sample holding times are evaluated based on defined limit units (e.g., a holding time of 14 days is interpreted to be equal to 14 days after the date of sampling, independent of sampling time, and a 48 hour holding time is calculated to the hour).

TABLE 20a. TEMPERATURE ACTIONS FOR INORGANIC ANALYSES

Matrix	Preservation	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous and Non-Aqueous	Yes/No	Samples were received with temperatures above 6°C but below 20˚C*	**J**	**UJ**
Aqueous and Non-Aqueous	Yes/No	Samples were received with temperatures below 2˚C, sample bottles were not intact and the samples were frozen	**R if analyzed**	**R if analyzed**
Aqueous and Non-Aqueous	Yes/No	Samples were received with temperatures below 2˚C and were frozen but sample bottles were intact	No qualification	No qualification
Aqueous and Non-Aqueous	Yes/No	Samples were received with temperatures above 20˚C	**Reject all results****	
Aqueous	No	Samples were digested/analyzed over twice the holding time from the time of sampling	**Reject all results**	

**Temperatures > 6ºC but ≤ 10ºC for mercury and cyanide may be accepted based on the professional judgment of the validator.*

**Metals (except mercury) samples received above 20ºC may not require rejection due to the chemical stability of the metals and may be accepted based on the professional judgment of the validator. Use professional judgement to evaluate analytes not addressed in NFGs.

Attachment B. Data Validation Variance Documentation

Please refer to the categories below for general guidance on relative sensitivity to elevated temperatures in coolers. This list is provided for general guidance only and professional judgement should be applied when determining qualifications of sample data based on cooler temperatures.

TABLE 20b. TEMPERATURE SENSITIVITIES FOR INORGANIC ANALYSES

Very Sensitive	Moderately Sensitive	Insensitive
Acidity	Ammonia	Hardness
Alkalinity	Bromide	Metals
BOD	Chloride	TDS
COD	Fluoride	TSS
Hexavalent Chromium	Kjeldahl Nitrogen (TKN)	
Color	Nitrate+Nitrite	
Cyanide	Phenolics	
Ferrous Iron	Conductivity	
pH	Sulfate	
Nitrate	Turbidity	
Nitrite	Mercury	
DO		

Attachment B. Data Validation Variance Documentation

ICP-MS Tune Analysis
Review Items: a) Instrument Printouts b) Raw Data
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Calibration
Review Items: a) Instrument Printouts b) Raw Data
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017 with additional reference to the USEPA CLP NFGs for Inorganic Data Review, document number EPA-540-R-04-004, October 2004.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Blanks

Review Items:

a) Method, Calibration, Trip, Equipment, and Field Blank Concentrations

b) Associated Sample Results

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. Items noted in bold type indicate variances due to professional judgment. **Please note that if a project requires that the samples be reported to the method detection limit as the reporting limit, the U flag will not be used and the JB will be utilized instead.**

TABLE 21. BLANK ACTIONS FOR INORGANIC ANALYTES

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field, Equipment, Trip, Calibration	Detect below the laboratory reporting limit	Detect below the laboratory reporting limit and/or below the blank concentration	**Report result with a U qualifier at the laboratory reporting limit**
Method, Field, Equipment, Trip, Calibration	Detect below the laboratory reporting limit	Non-detect	No Qualification
Method, Field, Equipment, Trip, Calibration	Detect below the laboratory reporting limit	Detect above or equal to the laboratory reporting limit but below or equal to 10 times the blank concentration	**Report result with a JB qualifier**
Method, Field, Equipment, Trip, Calibration	Detect above or equal to the laboratory reporting limit	Detect below the laboratory reporting limit	**Report result with a U qualifier at the laboratory reporting limit**
Method, Field, Equipment, Trip, Calibration	Detect above or equal to the laboratory reporting limit	Non-detect	No Qualification
Method, Field, Equipment, Trip, Calibration	Detect above or equal to the laboratory reporting limit	Detect above or equal to the laboratory reporting limit but below or equal to 10 times the blank concentration	**Report result with a JB qualifier**
Method, Field, Equipment, Trip, Calibration	Detect above or equal to the laboratory reporting limit	Detect below or equal to the blank concentration	**Report result with a U qualifier at the sample amount**
Calibration	Negative result with absolute value > reporting limit	Detect less than 10x the laboratory reporting limit	**J -**
Calibration	Negative result with absolute value > reporting limit	Non-detect	**UJ**

Attachment B. Data Validation Variance Documentation

Inductively Coupled Plasma – Interference Check Sample (ICP-ICS)
Review Items: a) Instrument Printouts b) Raw Data
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Laboratory Control Samples/Laboratory Control Sample Duplicates (LCS/LCSD)
Review Items: a) LCS/LCSD Recoveries b) LCS/LCSD Relative Percent Difference (RPD) Values (If applicable) c) LCS/LCSD Raw Results
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017. NOTE: Specific LCS/LCSD percent recovery control limits are provided in the NFGs for ICP-AES metals and ICP-MS metals; however, LCS/LCSD RPD control limits are not defined. In addition, LCS/LCSD percent recovery and RPD control limits are not provided for mercury or cyanide. If specific control limits are not defined in the NFGs, then laboratory-generated control limits should be used.
Trihydro action variances from standard U.S. EPA criteria: Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

Attachment B. Data Validation Variance Documentation

Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

Review Items:

a) MS/MSD Recoveries

b) MS/MSD Relative Percent Difference (RPD) Values

c) MS/MSD Preparation Samples

d) Post-Digestion Spike Recoveries (if any)

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.

NOTE: Specific MS/MSD percent recovery control limits (75%-125%) are provided in the NFGs for ICP-AES metals, ICP-MS metals, mercury, and cyanide; however, MS/MSD RPD control limits are not provided. Laboratory-generated control limits should be used for the evaluation of the MS/MSD RPD results.

Trihydro action variances from standard U.S. EPA criteria:

Data validator criteria are in general accordance with the criteria noted in the NFG listed in the criteria section.

When Post-Digestion Spike results for metals are required and available, the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017 should be used to qualify the sample results.

When Post-Digestion Spike results for metals are not available and/or not required, Table 22 below should be used to qualify the sample results. If Post-Digestion Spike analyses are not required, but are still performed and reported by the laboratory, the Post-Digestion Spike results may be ignored and Table 22 should be used to qualify the sample results.

TABLE 22. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ACTIONS FOR INORGANICS

Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R above the Upper Acceptance Limit**	**J+**	No Qualification
%R value below the Lower Acceptance Limit**	**J-**	**UJ**
%R Less than 30%**	**J-**	**UJ and R***
MS/MSD RPD value above the Upper Acceptance Limit**	**J**	**UJ**

*Flag non-detect results for the MS/MSD parent sample with an R flag. Flag other associated non-detect results with a UJ flag.

** Qualify ALL samples in the preparation batch.

Attachment B. Data Validation Variance Documentation

Laboratory Duplicates

Review Items:

a) Sample Results
b) Reporting Limits
c) RPD Values

Criteria:

The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.

NOTE: For analyses not included in the NFG, laboratory limits will be used for evaluation of laboratory performance.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. The variances listed below are applicable to the laboratory duplicate review items. Items noted in bold type indicate variances due to professional judgment.

TABLE 23. LABORATORY DUPLICATE ACTIONS FOR INORGANIC ANALYTES

Criteria	Action	
	Detected Compounds	Non-Detected Compounds
RPD value is greater than 20% and both results are greater than five times the reporting limit **	**J**	**UJ**
Original sample or duplicate sample is less than or equal to five times the reporting limit and absolute difference between sample and duplicate is greater than the reporting limit. **	**J**	**UJ**

** Qualify ALL samples in the preparation batch.

Attachment B. Data Validation Variance Documentation

ICP Serial Dilution
Review Items: a) Method Detection Limits (MDLs) b) Percent Difference (%D) Values
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.
Trihydro action variances from standard U.S. EPA criteria: The data validation variances listed below are based on validator professional judgment and may slightly vary from the USEPA CLP National Functional Guidelines but are found to be more conservative. Validator professional judgment may vary due to specific project data quality objectives. Items noted in bold type indicate variances due to professional judgment. **If serial dilution samples are not prepared from client samples, the serial dilution results will be examined but will not be flagged since matrix similarity could not be guaranteed.**

Attachment B. Data Validation Variance Documentation

Field Duplicates

Review Items:

a) Sample Results

b) Reporting Limits

Criteria:

The criteria are identical to those noted in the USEPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures, EQADR-Supplement0, April 2013.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment. Validator professional judgment may vary due to specific project data quality objectives. The variances listed below are applicable to the field duplicate review items. Items noted in bold type indicate variances due to professional judgment.

TABLE 24. FIELD DUPLICATE ACTIONS FOR INORGANIC ANALYSES

Matrix		
Water		
Criteria	Action	
	Detected Compounds	Non-Detected Compounds
The analyte RPD value is greater than 100%*	**J**	**UJ**
RPD value is greater than 30% and one or both results are greater than two times the reporting limit (Flag the parent and duplicate samples only)	**J**	**UJ**
RPD value is greater than 30% and both results are less than two times the reporting limit	No Qualification	
RPD value is less than 30%	No Qualification	

*** If the RPD is > 100%, qualify the analyte results in all associated samples.**

This guidance is applicable unless other regulatory or project–specific guidance is available (e.g. TRRP or QAPP).

If multiple field duplicates are collected in a sample set, associations are based on date of collection.

Attachment B. Data Validation Variance Documentation

Field Duplicates

TABLE 25. FIELD DUPLICATE ACTIONS FOR INORGANIC ANALYSES

Matrix		
Soil		
Criteria	Action	
	Detected Compounds	Non-Detected Compounds
The analyte RPD value is greater than 100%*	J	UJ
RPD value is greater than 50% and one or both results are greater than two times the reporting limit (Flag the parent and duplicate samples only)	J	UJ
RPD value is greater than 50% and both results are less than two times the reporting limit	No Qualification	
RPD value is less than 50%	No Qualification	

*** If the RPD is > 100%, qualify the analyte results in all associated samples.**
This guidance is applicable unless other regulatory or project–specific guidance is available (e.g. TRRP or QAPP).
If multiple field duplicates are collected in a sample set, associations are based on date of collection.

Attachment B. Data Validation Variance Documentation

ICP-MS Internal Standards
Review Items: a) Instrument printouts b) Raw data c) Relative Intensities
Criteria: The criteria are identical to those noted in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.
Trihydro action variances from standard U.S. EPA criteria: The data validation variances listed below are based on validator professional judgment. Validator professional judgment may vary due to specific project data quality objectives. **The National Functional Guidelines listed in the criteria section above specifies that a minimum of five of the following internal standards are required to be added to each sample: ^{6}Li, Sc, Y, Rh, In, Tb, Ho, Lu and Bi. However, the analytical methods may allow other internal standards to be used. The appropriateness of the internal standards will be evaluated based on method requirements.** **Requirements for internal standard intensity evaluation in some methods differ from the criteria National Functional Guidelines. Where method requirements are more strict than requirements in the National Functional Guidelines, method criteria will be used.**

Attachment B. Data Validation Variance Documentation

Total and Dissolved Metals

Review Items:

a) Laboratory reports

b) Electronic Data Deliverable (EDD)

c) Raw data (if available)

Criteria:

The criteria are implied in "Overall Assessment of Data" sections in the USEPA CLP NFGs for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017.

Trihydro action variances from standard U.S. EPA criteria:

The data validation variances listed below are based on validator professional judgment. Validator professional judgment may vary due to specific project data quality objectives.

In data sets with analyses of both total and dissolved metals for submitted samples, ensure that the total metals concentrations were greater than the associated dissolved metals results

Calculate the total – dissolved metal concentrations for each analyte for each sample. If the result is greater than or equal to 0 (total concentration ≥ dissolved result), no further action is required. If the result is less than 0 (total concentration < dissolved result), actions are defined in the table below.

TABLE 26. TOTAL AND DISSOLVED ANALYSES

Criteria	Action	
	Detected Compounds	Non-Detected Compounds
Total metal concentration is less than the associated dissolved metal result.	**J**	**-----**
Total metal concentration is less than 5 times the applicable RL, the associated dissolved metal result is greater than 5 times the RL, and the difference between the results is greater than the value of the RL.	**J**	**UJ**
Total metal concentration is less than 5 times the applicable RL, the associated dissolved metal result is greater than 5 times the RL, and the difference between the results is less than the value of the RL.	No Qualification	
Total metal concentration is less than the associated dissolved metal result and the difference is greater than the measurement uncertainty (RPD > 30% for water samples).	**J**	**-----**
Total metal concentration is greater than the associated dissolved metal result.	No Qualification	
Both total metal concentration and the associated dissolved metal result are less than 5 times the applicable RLs	No Qualification	

Total metal concentration is less than the associated dissolved metal result but within measurement uncertainty (RPD < 30% for water samples).	No Qualification

If the dissolved metals concentration was greater than the associated total metals result for an analyte,

1) **Contact the laboratory to identify the issue for resolution.**
2) **If the laboratory confirms the reported results, contact the project manager or responsible project personnel to notify of the potential error.**
3) **Assign appropriate qualifiers with reason code DIS-TOT (Dissolved metals concentration was greater than the associated total metals result) or LE (Laboratory Error).**

Professional judgement: If total metal concentration is less than the associated dissolved result, calculate the RPD for the two values. Measurement uncertainty can be the laboratory duplicate criteria of 20% (from NFG) or the laboratory-specific limit for lab duplicates or the MS/MSD RPD for the specific analyte or the field duplicate limit for the appropriate matrix.

References

USEPA. 2004. Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, document number EPA 540-R-04-004. October.

USEPA. 2017. Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001. January.

USEPA. 2017. Contract Laboratory Program National Functional Guidelines for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002. January.

USEPA. 1999. Contract Laboratory Program National Functional Guidelines for Organic Data Review, document number EPA 540/R-99/008. October.

USEPA. 2013. USEPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures, EQADR-Supplement0. April.

ATTACHMENT C

STANDARD OPERATING PROCEDURES

ATTACHMENT C-1

ACID DIGESTION OF AQUEOUS SAMPLES FOR ICP AND ICP-MS ANALYSIS
PACE, INDIANAPOLIS

Document Information

Document Number: ENV-SOP-IND1-0035 **Revision:** 01

Document Title: Acid Digestion of Aqueous Samples for ICP and ICP-MS Analysis

Department(s): Metals

Previous Document Number: S-IN-M-030-rev.13

Date Information

Effective Date: 20 Nov 2018

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All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-IND1-0035 **Revision:** 01
Title: Acid Digestion of Aqueous Samples for ICP and ICP-MS Analysis

All dates and times are in Central Time Zone.

ENV-SOP-IND1-0035

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Elizabeth Schrage (008534)	Quality Manager	18 Nov 2018, 08:11:27 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Steven Sayer (004775)	General Manager	19 Nov 2018, 07:54:31 AM	Approved
Felicia Walker (005354)	Manager - Lab Services	20 Nov 2018, 11:20:02 AM	Approved

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for acid digestion of aqueous samples for metals analysis while meeting the requirements specified in EPA methods 3005A and 3010A for analysis by ICP and in EPA Method 200.2 for analysis by ICP-MS.

2. Summary of Method

2.1. A portion of sample is digested with strong acid and heat in a block digester and then brought to volume with reagent water.

3. Scope and Application

3.1. This procedure is used to determine total metals and dissolved metals.

3.2. Volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of metals digestion equipment. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This digestion procedure is used for the preparation of aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection	Preservation	Storage	Hold time
Aqueous - Total	250mL in plastic container	- HNO_3 to pH <2 - Samples received at pH>2 must be preserved to pH<2 with HNO_3 and be allowed to equilibrate for 24 hours before being prepared for analysis. Acidification date and time are recorded in the Sample Preservation Logbook.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Aqueous - Dissolved	250mL in plastic container	- Filter; HNO_3 to pH<2 - For all dissolved elements by methods 200.7 or 200.8, samples must be filtered within 15 minutes of collection and before adding HNO_3, or data must be qualified that filtration occurred beyond 15 minutes of collection. Samples filtered in the lab are preserved to pH<2 with HNO_3 and allowed to equilibrate for 24 hours before being prepared for analysis. Filtration and acidification date and time are recorded in the metals digestion prep log.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Aqueous – Drinking Water	1L plastic container for Pb/Cu Rule compliance.	-Samples must be acidified to pH<2 with HNO_3 as soon as possible but not more than 14 days after sample collection. -Samples must stand in the original container used for collection for at least 28 hours after acidification.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Description / Comments
Hot Block Digester	Environmental Express or equivalent, adjustable and capable of maintaining a temperature of 92°C to 98°C.
Centrifuge	Fisher Centrific centrifuge Model 225 or equivalent
Vacuum pump	For lab filtration for dissolved elements.

9.2. General Supplies

Item	Description
Volumetric Flasks	Class A , various capacities
Volumetric Pipettors	Eppendorf or equivalent, various sizes
Digestion Tubes	Environmental Express or equivalent, volumetrically certified and contaminant free
Thermometer	Ever Safe or equivalent, calibrated, used for monitoring Hot Block temperature
Plunger Filters	Environmental Express or equivalent
Graduated Cylinders	Class A, various capacities
pH strips	Fisher or equivalent, full range
Filtration system	FlipMate or equivalent 0.45 um fiber filter disc caps and cups for lab filtration for dissolved elements.

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Nitric acid	Concentrated, trace metal analyzed or equivalent
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Table 10.1 Standard Definitions

Standard	Description	Comments
Spiking Standard	This solution contains the target analytes and is generally prepared using a standard source secondary to the standards used for calibration.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
ICP Stock Spiking Standards	Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
ICP Working Spiking Standard	Refer to Section 10.2.3.1.	Expires 6 months from date of preparation.	Same as stock standard
ICP-MS Stock Spiking Standard #1	Inorganic Ventures; catalog #HERT-CAL-2A or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
ICP-MS Stock Spiking Standard #2	Inorganic Ventures; catalog #HERT-CAL-2B or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions

10.2.3. Preparation Procedures

Table 10.3 – ICP Stock Spiking Standard Details

Analyte	Concentration (mg/L)
Inorganic Ventures PA-STD-1B	
Arsenic	200
Barium	200
Beryllium	200
Cadmium	200
Chromium	200
Cobalt	200
Copper	200
Lead	200
Lithium	200
Manganese	200
Nickel	200
Phosphorus	200
Selenium	200
Strontium	200
Thallium	200
Vanadium	200
Zinc	200
Inorganic Ventures PA-STD-2B	
Antimony	200
Boron	200
Molybdenum	200
Silicon	1000
Inorganic Ventures PA-STD-2B continued	
Silver	100
Tin	200
Titanium	200
Inorganic Ventures PA-STD-3B	
Aluminum	2000
Calcium	2000
Iron	2000
Magnesium	2000
Potassium	2000
Sodium	2000

Table 10.4 – ICP-MS Stock Spiking Standard Details

Analyte	Concentration (mg/L)
Inorganic Ventures HERT-CAL-2A	
Antimony	2
Molybdenum	2
Tin	2
Titanium	2
Inorganic Ventures HERT-CAL-2B	
Aluminum	20
Arsenic	2
Barium	2
Beryllium	2
Boron	2
Cadmium	2
Chromium	2
Cobalt	2
Copper	2
Lead	2
Manganese	2
Nickel	2
Selenium	2
Silver	2
Strontium	2
Thallium	2
Thorium	2
Uranium	2
Vanadium	2
Zinc	2

10.2.3.1. Working Spiking Standard Preparation

Dilute 25mL of each stock spiking standard (solutions 1B, 2B and 3B) to 100mL with reagent water for a final nominal concentration of 50mg/L.

11. Calibration

11.1. Not applicable to this SOP.

12. Procedures

12.1. If lower reporting limits are required, digestate concentration may be performed provided that final acid concentration and final spike concentration remain consistent with unconcentrated digestates. Refer to Section 12.4.

12.2. Lab Filtration for Dissolved Elements

12.2.1. Prepare the filtration apparatus by attaching a filter disc cap to a sample cup for each sample to be filtered.

12.2.2. To filter, attach the filtration apparatus to the vacuum pump and turn the pump on. Turn the vacuum pump off when filtration is complete and an adequate volume of filtrate has been collected.

12.2.3. Prepare a Method Blank by filtering reagent water through the filter disc cap and into a labeled sample cup. Filter enough volume to support all analyses requested.

12.2.4. Prepare an LCS by filtering an LCS prepared as described in Section 12.3.3 or 12.4.3 through the filter disc cap and into a labeled sample cup. Filter enough volume to support all analyses requested.

12.2.5. Filter samples by pouring the sample from the original sample container into the filter disc cap and collecting approximately 100mL of the sample filtrate in a labeled sample cup.

12.2.6. Preserve all filtrates to pH<2 with concentrated nitric acid. Hold preserved samples for a minimum of 24 hours before digestion and/or analysis.

12.2.7. Record all filtration information including sample cup lot number, filter disc cap lot number, and date and time of preservation in the metals digestion log.

12.3. Aqueous Sample Digestion for ICP

12.3.1. Transfer 50mL of well-mixed sample into a labeled digestion tube.

12.3.2. Prepare a Method Blank by transferring 50mL of reagent water to a digestion tube.

12.3.3. Prepare an LCS by adding 1mL of the ICP Working Spiking Standard (50mg/L nominal) to 50mL of reagent water for a nominal spike concentration of 1mg/L.

12.3.4. Prepare a Matrix Spike by adding 1mL of the ICP Working Spiking Standard (50mg/L nominal) to 50mL of sample for a nominal spike concentration of 1mg/L.

12.3.5. Add 2.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes.

12.3.6. If digestate is generating brown fumes, add another 2.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

12.3.7. Evaporate without boiling to approximately 10mL. Do not allow samples to go dry.

12.3.8. Cool the samples then add 2mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

12.3.9. Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates by using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered. Alternatively, reduce the effect of particulates by placing the samples into a centrifuge for approximately 5 minutes at 3500 rpm.

12.4. Aqueous Sample Digestion for ICP-MS

12.4.1. Transfer 50mL of well-mixed sample into a labeled digestion tube.

12.4.2. Prepare a Method Blank by transferring 50mL of reagent water to a digestion tube.

12.4.3. Prepare an LCS by adding 1mL of the ICP-MS Stock Spiking Standard #1 (2mg/L) and 1mL of the ICP-MS Stock Spiking Standard #2 (2mg/L nominal) to 50mL of reagent water for a nominal spike concentration of 0.04mg/L.

12.4.4. Prepare a Matrix Spike by adding 1mL of the ICP-MS Stock Spiking Standard #1 (2mg/L) and 1mL of the ICP-MS Stock Spiking Standard #2 (2mg/L nominal) to 50mL of sample for a nominal spike concentration of 0.04mg/L.

12.4.5. Add 0.5mL concentrated nitric acid and 0.25mL concentrated hydrochloric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of

95°C (+/- 3°C) in the digestion tubes.

12.4.6. If digestate is generating brown fumes, add another 0.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

12.4.7. Evaporate without boiling to approximately 10mL. Do not allow samples to go dry.

12.4.8. Remove the samples from the hot block and allow them to cool.

12.4.9. Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, reduce the effect of particulates by placing the samples into a centrifuge for approximately 5 minutes at 3500 rpm.

12.5. Aqueous Sample Digestion with Concentration for ICP

12.5.1. Transfer 50mL of well-mixed sample into a labeled digestion tube.

12.5.2. Prepare a Method Blank by transferring 50mL of reagent water to a digestion tube.

12.5.3. Prepare an LCS by adding 0.2mL of the ICP Working Spiking Standard (50mg/L nominal) to 50mL of reagent water for a nominal spike concentration of 1mg/L.

12.5.4. Prepare a Matrix Spike by adding 0.2mL of the ICP Working Spiking Standard (50mg/L nominal) to 50mL of sample for a nominal spike concentration of 1mg/L.

12.5.5. Add 0.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester and set the temperature to achieve 95°C (+/- 3°C) in the digestion tubes.

12.5.6. If digestate is generating brown fumes, add another 0.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

12.5.7. Evaporate without boiling to approximately 10mL. Do not allow samples to go dry.

12.5.8. Cool the samples then add 0.4mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

12.5.9. Dilute the digestates to 10mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered. Alternatively, reduce the effect of particulates by placing the samples into a centrifuge for approximately 5 minutes at 3500 rpm.

12.6. Record all preparation information including standard numbers, reagent numbers, digestion tube lot numbers, filter lot numbers, Hot Block number, thermometer ID and correction factor, and digestion temperature in the metals digestion log and deliver the digestates to the ICP analyst.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Refer to the SOP for the determinative method.	Refer to the SOP for the determinative method
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	Refer to the SOP for the determinative method.	Refer to the SOP for the determinative method.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Refer to the SOP for the determinative method.	Refer to the SOP for the determinative method.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Action for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Samples are digested for the ICP analysis of Antimony, Boron, Lithium, Silicon, Silver, Strontium, Tin and Titanium in addition to the analytes listed in the method.

19.2. Samples are digested for the ICP-MS analysis of Titanium in addition to the analytes listed in the method.

19.3. Volumes of acid used for ICP digestion vary from those in the methods.

19.4. Method modified for use with Hot Block digesters and digestion tubes are never capped while heating.

19.5. A digestion temperature range of 95°C +/-3°C is observed.

19.6. Samples are verified to be pH<2 prior to digestions but this verification may not take place "immediately" prior to digestion.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and should be handled with extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

22.3. **Equipment**: Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Preventions

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Methods 3005A and 3010A.

25.2. U.S. EPA, EMSL Method 200.2, Revision 2.8, 1994.

25.3. Pace Analytical Quality Manual; latest revision.

25.4. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-M-030-rev.11	1. Cover Page: changed phone number, changed method reference to 3010A and 200.2, add ICP-MS reference updated effective date format and changed SOP naming format from inorganics to metals. 2. Table of Contents: added new Section 14, Method Modifications. 3. Section 1.1: changed method reference to 3010A and 200.2 and added ICP-MS reference. 4. Table 7.1: added preservation for lab-filtered samples. 5. Table 8.1: updated required temperature range of Hot Block. 6. Section 9: added ICP-MS spiking standards. 7. Section 11: revised to include batch QC preparation, added guidance for non-standard final volumes, revised acid-addition to reflect current process, and separated process for ICP and ICP-MS digestion. Added specific procedure for digestate concentration to achieve lower limits. 8. Table 12.1: updated Method Blank corrective action. 9. Section 13: removed MDL requirement. 10. Section 14: new Method Modifications section added. 11. Section 16.1: changed method reference to 3010A and added 200.2 reference.	17Sep2015
S-IN-M-030-rev.12	1. Converted to 27 section format. 2. Table 7.1: added requirement to filter within 15 minutes of collection for methods 200.7 and 200.8 and revised storage temperature format. 3. Section 9.1: added centrifuge. 4. Section 12.2: changed final evaporation volume from 5mL to 10mL and added centrifuge as option to filter. 5. Section 12.3: changed final evaporation volume from 5mL to 10mL and changed filtration to centrifugation. 6. Section 12.4: changed final evaporation volume from 5mL to 10mL and added centrifuge as option to filter. 7. Table 13.1: referred to SOP for determinative method for acceptance criteria. 8. Section 25.4: added years 2003 and 2009 to TNI reference.	08Oct2017
S-IN-M-030-rev.13	1. Cover page: added reference to method 3005A. 2. Section 1.1: added reference to method 3005A. 3. Table 7.1: added requirement that all West Virginia samples for dissolved elements must be filtered within 15 minutes of collection. 4. Section 9.1: added vacuum pump. 5. Section 9.2: added filtration apparatus. 6. Section 12.2: added section to describe lab filtration procedure for dissolved elements. 7. Table 13.1: referred to determinative method for corrective actions. 8. Section 25.1 added reference to method 3005A.	24Oct2017
ENV-SOP-IND1-0035-rev.01	1. Removed cover, table of contents and headers for use in Master Control. 2. Table 7.1: added preservation and handling for drinking water samples to comply with the Pb/Cu rule. 3. Section 19.6: added a modification to indicate that sample pH is not always checked "immediately" prior to digestion. 4. Section 27: updated Document Number to Master Control number.	11Nov2018

ATTACHMENT C-2

NITRATE/NITRITE
PACE, INDIANAPOLIS

Document Information

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Document Title: Nitrate/ Nitrite

Department(s): Wet Chemistry

Previous Document Number: S-IN-I-042-rev.14

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Signature Manifest

Document Number: ENV-SOP-IND1-0045 **Revision:** 01
Title: Nitrate/ Nitrite

All dates and times are in Central Time Zone.

ENV-SOP-IND1-0045

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Elizabeth Schrage (008534)	Quality Manager	18 Nov 2018, 08:04:37 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Steven Sayer (004775)	General Manager	19 Nov 2018, 07:54:44 AM	Approved
Anne Troyer (008754)	Manager - Lab Services	19 Nov 2018, 02:58:52 PM	Approved

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining Nitrate/Nitrite Nitrogen in aqueous samples while meeting the requirements specified in EPA method 353.2 rev. 2.0.

2. Summary of Method

2.1. This method is based upon an aqueous sample being passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Separate, rather than combined, nitrate/nitrite values are readily obtained by carrying out the procedure first with, and then without, the Cu-Cd reduction step.

3. Scope and Application

3.1. The applicable range for this method is 0.1-5mg/L of nitrate/nitrite. The reporting limit for water samples is 0.1mg/L and for soil samples is 5mg/Kg. Refer to the LIMS for method detection limits.

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of nitrate-nitrite analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to the measurement of nitrate/nitrite in ground water, drinking, surface and saline waters, domestic and industrial wastes, and aqueous extracts of solid samples.

5. Limits of Detection and Quantitation

5.1. Refer to LIMS for method detection limits. Reporting limits are shown below:

Analyte	Aqueous mg/L	Solid mg/kg
Nitrogen, Nitrate	0.1	5
Nitrogen, NO2 plus NO3	0.1	5
Nitrogen, Nitrite	0.1	5

6. Interferences

6.1. Suspended matter in the reduction column will restrict sample flow. Samples may be pre-filtered.

6.2. Low results may be obtained from samples containing high concentrations of metals, such as iron or copper. EDTA can be added to eliminate this interference.

6.3. Residual chlorine can produce a negative interference by limiting reduction efficiency. Before analysis, samples should be checked and if required, dechlorinated with sodium thiosulfate. NOT DOING THIS.

6.4. Samples that contain large amounts of oil and grease can interfere with the cadmium in the procedure. This can be eliminated by pre-extracting the sample using an organic solvent. Oily samples are generally rejected for analysis.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	250mL in plastic container	For combined nitrate/nitrite analysis, H_2SO_4 to pH<2 For nitrate or nitrite individually, unpreserved.	Cool to ≤6°C	For preserved samples, analysis must be completed within 28 days of collection date. For unpreserved samples, analysis must be completed within 48 hours of collection.
Solid	50-100g in a glass jar	None required	Cool to ≤6°C	Sample preparation must be completed within 14 days of collection. Analysis must be completed within 48 hours of sample preparation.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
Autoanalyzer	Lachat Quikchem 8000, 8500 or equivalent	Equipped with autosampler, flow cell spectrophotometer for use at 520nm and data reduction system.

9.2. General Supplies

Item	Description
Graduated cylinders	Various sizes, Class A
Automatic-pipettors	Eppendorf or equivalent, various sizes
Volumetric flasks	Class A, various sizes
Beakers	125mL glass, 50mL disposable or equivalent
Filter paper	Whatman 4 or equivalent
Syringe filter	Environmental Express 0.45um or equivalent
Stir plate and stir bars	For preparation of solid samples
Balance	Accurate to 0.1g or equivalent
Chlorine Test Strips	HF Scientific Micro Check or equivalent
Sand	Or equivalent to be used as a simulated soil matrix.

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Sodium Thiosulfate	Reagent grade crystals
Sulfuric acid	Concentrated, Fisher catalog #A510-P212, or equivalent.
Sodium Hydroxide	Reagent grade pellets or equivalent.
10N Sodium Hydroxide solution	Reagent grade, Fisher catalog #S5410-4 or equivalent
Hydrochloric acid	Concentrated, JT Baker catalog #9530-33 or equivalent
Ammonium Hydroxide	Spectrum Chemical catalog #AM180 or equivalent
Ammonium Chloride	Reagent grade crystals or equivalent.
Disodium EDTA	Fisher catalog # O2793-500 or equivalent
Ammonium Chloride Buffer (pH 8.5) **for QuickChem 8000**	CAUTION: prepare this under a hood! To a 1L flask, add 500mL reagent water, 105mL conc. hydrochloric acid, 95mL ammonium hydroxide, and 1g disodium EDTA. Dissolve and dilute to mark with reagent water. Invert to mix. After cooling, adjust the pH to 8.5 using HCl or 10N sodium hydroxide solution. Solution expires 6 months from date of preparation.
Ammonium Chloride Buffer (pH 8.5) **for QuickChem 8500**	CAUTION: prepare this under a hood! To a 1L flask, add 500mL reagent water, 85g ammonium chloride, 1g disodium EDTA, and 9.2g sodium hydroxide. Dissolve and dilute to mark with reagent water. Invert to mix. After cooling, adjust the pH to 8.5 using HCl or 10N sodium hydroxide solution. Solution expires 6 months from date of preparation.
Phosphoric acid	85% solution, Fisher/ catalog # A242-4 or equivalent
Sulfanilamide	Fisher catalog #O4525 or equivalent
N-(1-naphthyl)-ethylenediamine dihydrochloride	Acros catalog # 42399-250 or equivalent
Sulfanilamide color reagent	In a 1L flask, add approx. 600mL of reagent water, add 100mL of phosphoric acid, 40g of sulfanilamide, and 1g of N-(1-naphthyl)-ethylenediamine dihydrochloride. Stir for 30 minute to dissolve and then dilute to the mark with reagent water. Invert to mix and then store in a dark bottle. This solution is stable for one month from preparation.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This standard is used for spiking MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Nitrate calibration standard	Ricca; catalog # R5307900-120A; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Stock Nitrite calibration standard	Ricca; catalog # R5444900-120C; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate #1 Nitrate/Nitrite calibration standards	Refer to Section 10.2.3.1	Must be prepared fresh monthly	Same as stock standard.
Intermediate #2 Nitrate/Nitrite calibration standards	Refer to Section 10.2.3.2	Must be prepared fresh daily	Same as stock standard.
Working Nitrate/Nitrite calibration standards	Refer to Section 10.2.3.3	Must be prepared fresh daily.	Must be prepared fresh daily.
Stock Nitrate ICV standard	SPEX; catalog # AS-NO3N9-2Y; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Stock Nitrite ICV standard	SPEX; catalog # AS-NO2N9-2Y; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Nitrate/Nitrite ICV Standard	Refer to Section 10.2.3.4	Must be prepared fresh monthly	Same as stock standard.
Working Nitrate/Nitrite ICV standard	Refer to Section 10.2.3.5	Must be prepared fresh daily.	Must be prepared fresh daily.
Working Nitrate Check Standard	Refer to Section 10.2.3.6	Must be prepared fresh daily	Must be prepared fresh daily.
Working Nitrite Check Standard	Refer to Section 10.2.3.7	Must be prepared fresh daily	Must be prepared fresh daily.

10.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for additional instructions regarding preparation of standards for Nitrate/Nitrite analysis. Instructions for preparation of fresh daily standards are detailed below.

10.2.3.1. Intermediate #1 Nitrate/Nitrite Calibration Standard Preparation

Dilute 10mL of each of the Stock Nitrate calibration standard (1000mg/L) and Stock Nitrite calibration standard (1000mg/L) to 100mL with reagent water for a final concentration of 100mg/L for each Nitrate and Nitrite or 200mg/L Nitrate+Nitrite.

10.2.3.2. Intermediate #2 Nitrate/Nitrite Calibration Standard Preparation

Dilute 5mL of the Intermediate #1 Nitrate/Nitrite Calibration Standard (200mg/L Nitrate+Nitrite) to 100mL with reagent water for a final concentration of 10mg/L Nitrate+Nitrite.

10.2.3.3. Working Nitrate/Nitrite Calibration Standard Preparation

Working calibration standards for the **QuickChem 8000** must be prepared fresh daily in reagent water using either the Intermediate #1 Nitrate/Nitrite Calibration Standard (100mg/L each NO3,NO2) or the CAL7 Nitrate/Nitrite Calibration Standard (2.5mg/L each NO3, NO2). Examples of possible calibration standards are as follows but may vary:

Standard	Standard Volume	Intermediate Standard used	Final Volume	Final Conc. each NO3, NO2	Final Conc. NO3+NO2
CAL0	0 mL	N/A	50mL	0 mg/L	0 mg/L
CAL1	0.2 mL	CAL7	50mL	0.01 mg/L	0.02 mg/L
CAL2	0.4 mL	CAL7	50mL	0.02 mg/L	0.04 mg/L
CAL3	2.0 mL	CAL7	50mL	0.1 mg/L	0.2 mg/L
CAL4	0.2 mL	Intermediate #1	50mL	0.4 mg/L	0.8 mg/L
CAL5 (CCV)	1.0 mL	Intermediate #1	100mL	1 mg/L	2 mg/L
CAL6	1.0 mL	Intermediate #1	50mL	2 mg/L	4 mg/L
CAL7	2.5 mL	Intermediate #1	100mL	2.5 mg/L	5 mg/L

Working calibration standards for the **QuickChem 8500** must be prepared fresh daily and are auto-diluted by the instrument using the Intermediate #2 Nitrate/Nitrite Calibration Standard (10mg/L Nitrate+Nitrite). Examples of possible calibration standards are as follows but may vary:

Standard	Auto-Dilution Factor	Final Conc. NO3+NO2
CAL0	N/A	0 mg/L
CAL1	500	0.02 mg/L
CAL2	250	0.04 mg/L
CAL3	50	0.2 mg/L
CAL4	12.5	0.8 mg/L
CAL5 (CCV)	5	2 mg/L
CAL6	2.5	4 mg/L
CAL7	2	5 mg/L

10.2.3.4. Intermediate Nitrate/Nitrite ICV Standard Preparation

Dilute 10mL of each of the Stock Nitrate ICV standard (1000mg/L) and Stock Nitrite ICV standard (1000mg/L) to 100mL with reagent water to give a final concentration of 100mg/L for each Nitrate and Nitrite or 200mg/L Nitrate+Nitrite.

10.2.3.5. Working Nitrate/Nitrite ICV Standard Preparation

Dilute 1mL of the Intermediate Nitrate/Nitrite ICV standard (100mg/L) to 100mL with reagent water to give a standard concentration of 2mg/L Nitrate+Nitrite. This standard must be prepared fresh daily.

10.2.3.6. Working Nitrate Check Standard Preparation

Dilute 0.1mL of the Stock Nitrate calibration standard (1000mg/L) to 50mL with reagent water to give a standard concentration of 2mg/L. This standard must be prepared fresh daily.

10.2.3.7. Working Nitrite Check Standard Preparation

Dilute 0.1mL of the Stock Nitrite calibration standard (1000mg/L) to 50mL with reagent water to give a standard concentration of 2mg/L. This standard must be prepared fresh daily.

11. Calibration and Standardization

11.1. Initial Calibration: A minimum of five initial calibration standards are analyzed in decreasing order of concentration. The lowest calibration standard must be at or below the reporting limit. A new initial calibration curve is run on each working day. Refer to the Quality Manual for more information regarding calibration curves.

11.2. Linear Calibration: Using the Lachat software, prepare a standard curve by plotting area versus concentration. The analyst may employ a regression equation that does not pass through the origin. Weighting factors of $1/x$ or $1/x^2$ may be used to gain accuracy at lower concentrations. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = y-intercept of the line

The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient or r must be ≥ 0.995.

11.3. Non-linear Calibration: In situations where a linear model does not meet the acceptance criteria, a non-linear calibration model may be employed provided that a minimum of six initial calibration standards have been analyzed. Weighting factors of $1/x$ or $1/x^2$ may be used to gain accuracy at lower concentrations. The non-linear or quadratic model produces the following equation:

$$y = ax^2 + bx + c$$

The coefficient of the determination (COD) or r^2 can be used at a measure of the "goodness of fit." In order to be used for quantitative purposes, the COD or r^2 must be ≥ 0.99.

11.4. Initial Calibration Corrective Action: If the initial calibration does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered.

11.5. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy of the calibration, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately after an initial calibration curve. The acceptable range for the ICV is +/-10% Difference, which is equivalent to 90-110% Recovery.

$$\% \text{ Difference} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration})}{\text{Theoretical concentration}} \times 100$$

$$\% \text{ Recovery} = \frac{\text{Calculated concentration}}{\text{Theoretical concentration}} \times 100$$

11.6. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.7. Initial Calibration Blank (ICB): The ICB consists of reagent water. A ICB must be analyzed after each ICV. If any ICB result is above the reporting limit, sample analysis must be stopped. Samples associated with a failed ICB must be reanalyzed. Exception: If the ICB is >RL, associated samples determined to be <RL are reportable.

11.8. Nitrite Check Standard: A 2mg/L nitrite (NO2) check standard must be analyzed after each initial calibration curve.

11.9. Nitrate Check Standard: A 2mg/L nitrate (NO3) check standard must be analyzed after each initial calibration curve. This standard is used to verify the efficiency of the cadmium reduction column. Calculate the ratio of the nitrate and nitrite check standards observed concentrations as follows to determine the percent efficiency of the cadmium column:

$$\frac{\text{NO3 Check Standard conc.}}{\text{NO2 Check Standard conc.}} \times 100$$

The calculated cadmium column efficiency must be $\geq$90%.

11.10. Nitrate/Nitrite Check Standard Corrective Action: If the calculated percent efficiency of the cadmium column fails the acceptance criteria, another set of nitrate/nitrite check standards may be analyzed. If the percent efficiency of the cadmium column fails again, then a new cadmium reduction column must be installed and a new initial calibration curve must be analyzed.

11.11. Continuing Calibration Verification (CCV): A CCV must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable range for these standards is +/-10% Difference, which is equivalent to 90-110% Recovery. NOTE: certain clients or programs may require that CCVs at two concentrations, low and high, be analyzed when a non-linear or quadratic calibration model is used.

11.12. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.13. Continuing Calibration Blank (CCB): The CCB consists of reagent water. A CCB must be analyzed after each ICV/CCV. If any CCB result is above the reporting limit, sample analysis must be stopped. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable.

12. Procedures

12.1. Aqueous Sample Preparation

12.1.1. Filter any samples that contain suspended solids using a 0.45um syringe filter. Alternatively, samples can be centrifuged.

12.1.2. Check each sample for residual chlorine using a chlorine test strip. Remove any residual chlorine detected using sodium thiosulfate.

12.1.3. **QuickChem 8000:** for preserved samples, adjust sample pH to 5-9 with HCl or NaOH prior to analysis. NOTE: No pH adjustment of preserved samples is required for QuickChem 8500.

12.1.4. Prepare a Method Blank by adding 10mL reagent water to an autosampler tube for analysis. Method Blank should be filtered if associated samples in the batch required filtration.

12.1.5. Prepare an LCS by diluting 0.1mL of the Intermediate Nitrate/Nitrite ICV Standard (100mg/L) to 10mL with reagent water for a final concentration of 1mg/L each NO3or NO2 or 2mg/L NO3+NO2. LCS should be filtered if associated samples in the batch required filtration.

12.1.6. Prepare a Matrix Spike by diluting 0.1mL of the Intermediate Nitrate/Nitrite ICV Standard (100mg/L) to 10mL with sample for a final concentration of 1mg/L each NO3or NO2 or 2mg/L NO3+NO2. MS should be filtered if associated parent sample required filtration.

12.2. Solid Sample Preparation

12.2.1. Weigh 10g of well-mixed sample into a beaker and add 100mL reagent water. Stir for 30 minutes and then allow it to settle before gravity filtering. If necessary, filtrate may be further filtered through a 0.45um syringe filter before analysis.

12.2.2. Prepare a Method Blank by weighing 10g of sand into a beaker and adding 100mL reagent water. Stir for 30 minutes and then allow it to settle before gravity filtering. If necessary, filtrate may be further filtered through a 0.45um syringe filter before analysis.

12.2.3. Prepare an LCS by diluting 0.1mL of the Intermediate Nitrate/Nitrite ICV Standard (100mg/L) to 10mL with the filtrate from Section 12.2.2 for a final concentration of 10mg/kg each each NO3or NO2 or 20mg/kg NO3+NO2.

12.2.4. Prepare a Matrix Spike by diluting 0.1mL of the Intermediate Nitrate/Nitrite ICV Standard (100mg/L) to 10mL with the sample filtrate from Section 12.2.1 for a final concentration of 10mg/kg each each NO3or NO2 or 20mg/kg NO3+NO2.

12.3. Configure the instrument according to manufacturer's instructions. Allow the colorimeter and recorder to warm up. Run a baseline with all reagents, using reagent water to flush the tubing. Whenever new tubing is used, allow ample time to flush residual compounds from the tubing.

12.4. Establish initial calibration as described in Sections 11.1 through 11.11.

12.5. Once initial calibration is established, analyze 10mL portions of each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration standards
ICV
ICB
NO2 Check Standard

NO3 Check Standard
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB

12.6. Sample concentrations exceeding the linear range must be diluted and reanalyzed or the result must be qualified as estimated.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples.	Target analyte must be less than reporting limits	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	90-110% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per batch plus an additional MS if >10 samples in the batch.	90-110% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

Aqueous Sample (mg/L) = $(X_s)(D)$

Solid Sample (mg/kg) = $\frac{(X_s)(V_f)(D)}{(W_s)}$

Where: X_s = Nitrate/Nitrite concentration, mg/L
V_f = Final sample volume in milliliters
D = Dilution factor
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

14.2. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Section 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. MDLs must be determined per EPA *Definition and Procedure for the Determination of the Method Detection Limit, Revision 2*; December 2016.

18.2. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. This procedure has been adapted for the analysis of solid samples.

19.2. Cadmium reduction column is purchased and not prepared in the lab.

19.3. Stock Nitrate and Nitrite standards are purchased as certified solutions and not prepared from dry chemicals.

19.4. Initial calibration acceptance based on correlation coefficient (r) or coefficient of the determination (COD or r^2) rather than the difference between the measured value of the calibration solutions and the true value concentration.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. USEPA, Methods 353.2, Revision 2, "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, August 1993.

25.2. Lachat QuickChem Methods 10-107-04-1-A, July 2008, 10-107-04-1-C, August 2000 and 10-107-05-1-A, November 2007.

25.3. Pace Analytical Quality Manual; latest revision.

25.4. NELAC/TNI Standards; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not Applicable

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-042-rev.13	1. Table of Contents: added new Section 14, Method Modifications 2. Section 3: added references to solid samples and MDLs. 3. Table 7.1: added information for the collection of solid samples 4. Table 8.2: added supplies for the preparation of solid samples 5. Table 9.1: updated reagent details 6. Section 9.2.3: clarified final concentration of standards is for each NO3 and NO2. 7. Section 9.2.3.2: identified the standard used as the CCV 8. Sections 11.2-11.5: detailed the preparation of solid samples, MB, LCS and MS. 9. Section 11.9: 10. Section 11.10: added calculation of solid samples final concentration 11. Table 12.1: revised corrective action for Method Blank. 12. Section 13.1: revised MDL frequency to every 6 months and as necessary. 13. New Section 14, Method Modifications added. 14. Section 15.1: updated SOP reference.	09May2013

S-IN-I-042-rev.14	1. Cover page: revised format for effective date and document control. 2. Section 5.1: added table for water and soil RLs. 3. Table 7.1: revised storage temperature. 4. Section 9.1: added QuickChem 8500. 5. Section 9.2: added sand and chlorine test strips. 6. Section 10.1: added sodium thiosulfate, sulfuric acid, buffer and diluents for QuickChem 8500. Added expiration period for buffer. 7. Table 10.3: added Int. #2 standard and working NO3 check standard. 8. Section 10.2.3: added Int. #2 standard, separated working calibration standards for QuickChem 8000 and 8500, added ICV for QuickChem 8500 and added working NO3 check standard. 9. Section 11.2: specified that a minimum of 5 calibration standards is required. 10.Section 11.3: specified that weighting may be used for linear calibrations. 11.Section 11.4: added non-linear calibration specifications. 12.Section 11.8: added ICB 13.Section 11.9: revised NO2 check standard requirements 14.Section 11.10: added NO3 check standard and column efficiency equation. 15.Section 11.11: added NO3/NO2 check standard corrective action. 16.Section 11.12: added a note regarding CCVs associated with non-linear calibration. 17.Section 12: separated aqueous and solid sample handling procedures. 18.Section 12.1: added chlorine check and pH adjustment of aqueous samples. 19.Table 13.1: clarified MS frequency 20.Section 14.1: clarified aqueous sample calculation. 21.Section 19: added method modification for ICAL evaluation/acceptance. 22.Section 25: added reference to Lachat methods.	07Oct2016
ENV-SOP-IND1-0045-rev.01	1. Removed cover, table of contents and headers for use in Master Control. 2. Section 6.4: added language that oily samples are usually rejected for analysis. 3. Section 10.2.3.2: corrected standard ID used from Int #2 to Int #1. 4. Sections 12.2.1 and 12.2.2: corrected dilution procedure to match practice. 5. Table 13.1: updated LCS corrective action to include one rerun attempt. 6. Section 18.1: updated MDL procedure reference. 7. Section 25.4: updated reference to include 2003 and 2009. 8. Section 27: updated Document Number with Master Control number.	11Nov2018

ATTACHMENT C-3

SAMPLE MANAGEMENT
PACE, INDIANAPOLIS

Pace Analytical Services, LLC
7726 Moller Road
Indianapolis, IN 46268
Phone: 317.228.3100
Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

SAMPLE MANAGEMENT

Reference Methods: N/A

Local SOP Number:	S-IN-C-001-rev.09
Effective Date:	August 20, 2018
Supersedes:	S-IN-C-001-rev.08
SOP Template Number:	SOT-ALL-C-001-rev.06

APPROVALS

General Manager	August 15, 2018 Date
Quality Manager	August 7, 2018 Date
Client Services Manager	August 15, 2018 Date
Sample Receiving Manager	August 7, 2018 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

This is **COPY#**_____ distributed on _____ by _____

Table of Contents

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to outline the procedures involved with the receipt, login, storage, and disposal of samples received by Pace Analytical Services, LLC.

2. Summary of Method

2.1. Samples are delivered to the laboratory via several delivery mechanisms. Samples received are checked for adherence to the Sample Acceptance Policy (see Attachment I) with any discrepancies noted. Discrepancies are communicated to the client if necessary for their acknowledgement and decision making.

2.2. The Laboratory Information Management System (LIMS) assigns all samples with a unique sample number and manages the analyses assigned to each sample.

2.3. Samples are labeled with the appropriate information and staged in refrigerated sample storage coolers if temperature preservation is required or possibly stored on open shelves for samples not requiring sub-ambient temperature preservation. Samples will remain under these conditions until prepared and/or analyzed. Samples received under United States Department of Agriculture (USDA) protocols need to be stored separately (please refer to the lab's Regulated Soils SOP, if applicable).

2.4. Samples and associated sub-samples (digestates, extracts, etc.), are maintained for a minimum of 45 days from receipt of samples unless otherwise requested by the client or other regulatory agency.

2.5. Samples are disposed of in accordance with local laboratory regulatory requirements, waste handling procedures, and any USDA regulated soil requirements.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP apply to all personnel involved in the receipt, login, storage, and disposal of samples.

3.2. The Sample Acceptance Policy (Attachment I) contains the guidelines for acceptable sample conditions. Any deviation from these guidelines requires detailed documentation within the report, usually as a footnote, or on the chain-of-custody (COC), or Sample Condition Upon Receipt (SCUR) form and may require client contact.

3.3. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. Refer to Table 8.1 in this SOP for the applicable matrices.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Samples may be prone to cross contamination from others within the same delivery group or from other client projects. The sample receiving personnel must make every effort to minimize cross- contamination.

6.2. Preservation checks are one of the most likely situations where cross-contamination may occur. Materials used in the process must be specific to each sample and may not used for multiple samples or multiple containers of the same sample.

6.3. Samples are stored under specific conditions and in specific locations, typically per the requirements of the analytical method. However, consideration must be given to samples that are uniquely different from others. Samples that are anticipated to be severely contaminated must be segregated from others in anticipation that the high levels of contaminants may cross-contaminate others in close proximity. USDA samples must also be distinctly segregated for storage.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Acceptable sample preservation, containers, and hold times can be referenced in the Bottle and Preservation Table, available within the Pace Quality Assurance Manual, or as a separate document. Samples are stored separately from all standards and reagents and any known highly contaminated samples.

7.2. **NOTE**: To avoid contamination, no food or drink products can be located near the areas where samples are unpacked, labeled, or staged.

7.3. Sample Storage – See Section 12.3 for general storage guidelines.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual.

8.2. **Chain-of-Custody (COC):** a form used to record the field identification of samples collected, analyses requested, date and time of collection, sample preservation used, and traceability of samples from time of collection until delivery to the laboratory. This is a legal document. See example Attachment II.

8.3. **Laboratory Information Management System (LIMS):** a computer system used to manage the flow and traceability of environmental samples and associated data within the laboratory.

8.4. **Matrix:** the bulk characteristics of a sample. See Table 8.1 below.

8.5. **Safety Data Sheet (SDS):** contains information on chemicals used in the laboratory.

8.6. **Sample Custody:** a sample is considered to be in someone's custody if:

8.6.1. It is in one's physical possession;

8.6.2. It is in someone's view, after being in someone's physical possession;

8.6.3. It is kept in a secured area, restricted to authorized personnel only.

8.7. **Sample Condition Upon Receipt (SCUR) form:** a form used to record the condition of samples received in the laboratory.

8.8. **Sample Receipt Form (SRF):** form generated by LIMS system after a project is logged in. Contains sample and project information.

8.9. **UN Number** - identification numbers preceded by the letters UN are associated with proper shipping names considered appropriate for international and domestic transportation. These shipping names along with the identification numbers are located in the Federal Register (49CFR172.101).

Table 8.1

NELAC/TNI defined matrix	Corresponding EPIC Pro matrices
Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device.	Air (AR)
Aqueous: any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.	Water (WT)
Biological tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.	Tissue (TS) or Tissue Dry (TD)
Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.	Oil (OL) or Other (OT)
Drinking Water: any aqueous sample that has been designated a potable or potentially potable water source.	Drinking Water (DW)
Non-aqueous liquid: any organic liquid with < 15% settleable solids.	Other (OT)
Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.	Water (WT)- not assigned as a separate matrix.
Solids: includes soils, sediments, sludges and other matrices with > 15% settleable solids.	Solid (SL)
(No corresponding matrix to wipes; wipes would be included in with solids)	Wipe (WP) or Swab (SW)

9. Equipment and Supplies (Including Computer Hardware and Software)

Table 9.1

Equipment/Supplies	Description
Sample Labels	
Thermometers	Infrared, digital, NIST traceable
Sample storage cooling units	Capable of holding required storage temperatures
COC forms	Chain of Custody forms
SCUR forms	
pH paper	Wide range, 0-14
Label Printer	
LIMS computer system	EPIC Pro
Disposable pipettes	
Sample containers	
Residual chlorine strips	Capable of measuring 0.5mg/L of chlorine
Temperature blank	

10. Reagents and Standards

10.1. All reagents used in this procedure must be labeled with:

10.1.1. Laboratory reagent identification number;

10.1.2. Unless otherwise noted, the name and concentration of the reagent;

10.1.3. Date the reagent was received, opened and, as needed, prepared;

10.1.4. Person preparing reagent;

10.1.5. Expiration date.

10.2. **Reagents: Table 10.1**

Reagent	Formula	Concentration
Sulfuric Acid	H_2SO_4	1:1
Nitric Acid	HNO_3	1:1
Hydrochloric Acid	HCl	1:1
Sodium Hydroxide	NaOH	50% or Pellets
Sodium Thiosulfate	$Na_2S_2O_3 \cdot 5H_2O$	
Zinc Acetate Solution (for sulfide)		
Methanol	CH_3OH	Purge and Trap Grade
Hexane	C_6H_{14}	Pesticide Grade
Ascorbic Acid (for cyanide)		
Sodium Bisulfate	$NaHSO_4$	

10.3. For acids, bases and other reagents obtained from other laboratory departments, this information is located in the appropriate hardcopy or electronic standards/reagent preparation log. In the event that these reagents are managed within the Sample Receiving group, the department must maintain its own reagent preparation log.

10.4. Alternatively, pre-preserved sample containers can be used. In this case, documentation must be maintained for bottleware and preservation traceability.

11. Calibration and Standardization

11.1. Thermometers, IR-Guns, and other equipment used for measuring temperatures must be calibrated according to SOP S-IN-Q-157 **Support Equipment**, or its equivalent revision or replacement.

12. Procedure

12.1. **Sample Receipt**

12.1.1. The laboratory receives client samples via three major methods: mail/commercial delivery service, Pace Analytical courier/field services and hand delivery.

12.1.2. **Courier COC Procedures**: Pace labs use courier services that pick up client samples on either a regular schedule or on an as-needed basis as communicated by Project Managers (PMs) or by the client.

12.1.2.1. When the client is present during courier pick-up, the client signs the COC relinquishing custody to the courier. The courier signs the COC as accepting the samples and provides the client with a copy of the COC. When the courier returns to the lab with the client samples, the courier signs the COC as relinquishing the samples to the lab.

12.1.2.2. If the client is not present during courier pick-up, the courier signs the COC as accepting the samples and leaves a copy of the COC for the client. If a client also has a sample log, the courier must sign and date the log when the samples are picked up. When the courier returns to the lab with the client samples, the courier signs the COC as relinquishing the samples to the lab. The date/time of delivery to the lab by the courier is the official date/time received by the lab (analogous to the official date/time of receipt by an outside commercial carrier or courier).

12.1.2.3. To ensure the sample security, the Pace courier vehicle is locked at each client pick-up location. IMPORTANT: Pace Analytical courier/field services personnel must open the sample coolers and verify there is adequate ice in the coolers before transporting or shipping to the laboratory. An exception to this policy would be for coolers already custody-sealed by the client. These coolers are not to be opened except by the receiving lab personnel.

12.1.3. **Lab COC Procedures**: If the client drops off the samples, the COC is signed by laboratory receiving personnel and a copy of the signed COC is given to the client at that time. If samples are received via commercial carrier or mail delivery, the COC is signed when the cooler or package is opened and processed for login. The delivery date and time is considered the date/time received.

12.1.3.1. **Samples Dropped Off:** Sample receiving personnel must review the COC for any evidence of rush turnaround requests, analyses with short hold times, or samples with very little hold time remaining. Projects that fall under these conditions must be given immediate attention. The PM responsible for that client must be alerted in the event that they have not already alerted the laboratory to the project as it may be possible that the client did not pre-schedule the project. Once the samples are received and logged into the LIMS, the sample technician and project manager will coordinate the notification and delivery of samples to the laboratory.

12.1.3.2. Internal Chain-of-Custody: If a client or program requires internal chain-of-custody (ICOC) procedures, the PM must determine, prior to log-in, which projects require ICOC processing and must clearly communicate the requirement to the laboratory. Refer to SOP S-IN-C-055 **Internal Chain-of-Custody** for detailed information.

12.1.4. **Sample Acceptance Policy** - Copies of the Sample Acceptance Policy must be provided, in the form of a letter, fax, or e-mail to each client or sampler, as necessary. Samples are considered acceptable if they meet the criteria listed in the Sample Acceptance Policy (see Attachment I)

12.1.4.1. Some labs may have agreements with clients regarding exceptions to the client contact requirements for sample acceptance policy deviations. If a lab has such agreements, two conditions must be met: 1) the agreement must be a formal document showing client approval; 2) the lab must qualify the final report as appropriate to their applicable regulatory bodies.

12.1.4.2. For Wisconsin Drinking Water samples: Samples that do not meet the criteria in the Sample Acceptance Policy will be rejected by sample custody. Sample custody will notify the PM and the client will be notified before proceeding with login. If the client wishes to proceed with analysis, the project manager will retain documentation of the request to proceed.

12.1.5. **Measuring cooler temperature – temperature blank:** Open the cooler and verify the temperature of the samples by taking the temperature of the temperature blank. If there is no temperature blank in the cooler, proceed to Section 12.1.6. The temperature of the temperature blank must be determined using a NIST-traceable stick thermometer. Remove the temperature blank bottle from the cooler and sit it on the benchtop. Remove the lid of the temperature blank and place the stick thermometer into the bottle. If the observed temperature of the temperature blank is outside of the acceptable range of 0^{o}C to 6^{o}C, proceed to Section 12.1.6.

12.1.6. **Contingency - measuring the temperature of cooler melt water:** If there is no temperature blank in the cooler or if the observed temperature of the temperature blank is outside of the acceptable

range of 0°C to 6°C, measure the temperature of visible cooler melt water**.** The temperature must be determined using a NIST-traceable stick thermometer. Place the stick thermometer into the cooler melt water. If the observed temperature of the visible cooler melt water is outside of the acceptable range of 0°C to 6°C, proceed to Section 12.1.7.

12.1.7. **Contingency - measuring temperature of a centrally located sample container:** If there is no temperature blank in the cooler, if there is no visible cooler melt water, or if the observed temperature of the temperature blank and/or cooler melt water is outside of the acceptable range of 0°C to 6°C, measure the temperature of a representative sample container. A representative sample container will be centrally located within the cooler. The temperature must be determined using a NIST-traceable IR gun. Remove the representative sample container from the cooler. While holding the container by the lid, take the container temperature using an IR gun pointed at an opaque surface such as the bottle label. If the observed temperature of the representative sample container is outside of the acceptable range of 0°C to 6°C, proceed to Section 12.1.8.

12.1.8. **Contingency – all measured temperatures are outside of the acceptable range:** If the observed temperature of the temperature blank, cooler melt water, and representative sample container are all outside of the acceptable range of 0°C to 6°C, immediately consult with the Sample Receiving Manager, the Project Manager, or the Customer Service Manager to initiate client notification and the need for additional documentation that may include photos of the cooler conditions upon receipt.

12.1.9. **Measuring the temperature of West Virginia samples:** measure the temperature of each sample container using an IR gun as described in Section 12.1.7 and document any container that is outside of the acceptable range of 0°C to 6°C.

12.1.10. Record the uncorrected (observed) and corrected cooler temperatures on the COC (example in Attachment II) and/or SCUR form (example in Attachment III. In addition, record the type of "ice" used for packing the cooler (e.g., wet ice, "blue ice", gel packs, etc.).

12.1.11. If samples within a project are spread over multiple coolers and one or more of the coolers are outside of the temperature criteria, then the contents of the cooler must be itemized and the samples and sample containers affected by the out-of-control temperature must be documented on the SCUR form for communication to the client. This itemization must be retained in the project file for future reference.

12.1.12. Make a copy of the COC. Give the original COC to login personnel to begin live login while the cooler is being unpacked.

12.1.13. Carefully unpack the cooler and organize the samples, grouped by client sample ID, according to the order on the COC. Review COC against samples to make sure the bottles received match the analysis requested. All anomalies must be recorded on the SCUR form and/or the Sample Container Count form (example in Attachment IV).

12.1.13.1. If a cooler is received at the end of the day and will not be checked in until the next day, the temperature of the cooler must be determined and documented. The COC must be evaluated for any requested short hold analyses before the cooler is placed into the walk-in overnight. If there are short hold analyses requested, the supervisor of the affected department must be notified.

12.1.14. For USDA samples, the cooler and all contents must be decontaminated with a 10% bleach solution (refer to Regulated Soil SOP for procedure). For non-USDA samples, discard any ice or water that remains in the cooler and the packing material used to secure the samples. Water or ice should be discarded down a drain that connects to the local sewer. Packing materials should be placed in the garbage. If a sample container was broken, the contents remaining in the cooler MUST be discarded in a manner consistent with the hazardous waste handling standard operating procedure. Refer to SOP S-IN-C-007 **Regulated Soil Handling** for detailed instructions.

12.1.15. **pH Verification Instructions:**

12.1.15.1. The pH of the sample must be verified on all preserved sample bottles requiring pH preservation (see exceptions in Section 12.1.11.3).

12.1.15.2. Open each preserved bottle (except as noted below). Use a new disposable pipette, a stirring rod or another inert utensil to withdraw a small portion of the sample. Dispense the aliquot onto an unused pH strip and check the pH.

12.1.15.3. **NOTE:** Do not check the pH of samples for coliform, volatiles, Wisconsin Diesel Range Organics (WI-DRO), oil and grease, hexane extractable materials (HEM), or any bottle with a septum lid. These analyses will be checked by the analyst at the bench and must not be opened by sample management personnel.

Table 12.1 – General pH Preservation Requirements by Preservative

Sample Preservatives	Sample pH Requirement
Hydrochloric Acid (HCl)	must be less than 2
Nitric Acid (HNO_3)	must be less than 2
Sulfuric Acid (H_2SO_4)	must be less than 2
Sodium Hydroxide (NaOH)	must be greater than 12
Zinc Acetate and Sodium Hydroxide (NaOH)	must be greater than 9

12.1.15.4. If the pH for a sample container that is supposed to be preserved is not within the required range, indicate the anomaly on the SCUR form or on the COC and mark the container with a red dot. If a sample does not require preservation, write N/A in the applicable section of the SCUR form.

12.1.15.5. Any pH adjustments will be made by the analytical departments.

12.1.16. **Total Residual Chlorine Verification Instructions -** Total residual chlorine must be verified at the time of receipt for certain analyses (see Table 12.2). Do not check the sample bottles for those analyses listed in 12.1.11.3.

12.1.16.1. Open the appropriate sample container. Utilizing a new disposable pipette, stirring bar or other inert utensil; withdraw a small portion of the sample. Dispense the aliquot on an unused residual chlorine test strip.

12.1.16.2. If any chlorine is detected, regardless of amount, note the information on the COC, SCUR or analytical bench sheet.

12.1.16.3. Samples that are positive for residual chlorine are immediately taken to the appropriate department for dechlorination.

Table 12.2 – Analyses requiring Residual Chlorine Verification

Analyses
Cyanides SM4500 CN, EPA 335.4, 9012, 9014
EPA 608
EPA 625

12.1.17. Note any discrepancies pertaining to samples as defined by the sample acceptance policy detailed above on the COC, SCUR, or Sample Container Count form, as applicable. Any discrepancies involving temperature, preservation, hold time, collection dates and times, sample volume, sample containers, and unclear analysis, must be reported to project management as soon as possible.

12.1.18. For short hold samples, the laboratory is notified and the samples are staged per Table 12.3.

Table 12.3 – Analyses with Short Holding Times

24-48 Hr. Short Hold Analyses

Waters

Method/Analytes	ACODE(S) & Department	Holding Time
Biochemical Oxygen Demand (BOD, CBOD, SM5210B)	5210BW, 5210BWC / **Wet Chem**	48 Hour
Color (SM2120B)	2120B W / **Wet Chem**	48 Hour
Hexavalent Chromium (Cr+6, CrVI, Cr6+,7196, SM3500-Cr B)	7196 W, 3500CrDW / **Wet Chem Unpreserved**	24 Hour
Nitrate/Nitrite (NO3, NO2, 353.2, 300.0, 9056)	3000 S, 3000 W, 9056 S, 9056 W / **GC** 3532 W / **Wet Chem**	48 Hour
Ortho-Phosphate (O-Phos, PO4, SM4500-P E)	4500PE WO / **Wet Chem**	48 Hour
Settleable Matter/Solids (SM2540F)	2540F W / **Wet Chem**	48 Hour
Sulfide Unpreserved (SM4500-S2 D)	4500S2D W / **Wet Chem**	24 Hour
Surfactant (MBAS, SM5540C)	5540C W / **Wet Chem**	48 Hour
Turbidity (Turb, 180.1)	1801 W / **Wet Chem**	48 Hour

Solids

Method/Analytes	ACODE(S) & Department	Holding Time
BP Volatile Soils	8260 S	Subsample within 48 hours
Volatile (8260 Terracore 5035A)	8260E5035A, 8260TCUST / **RCVG Freezer** or **VOA Freezer**	Frozen within 48 Hours of sampling

12.2. **Sample Login**

12.2.1. All samples received by the laboratory must be logged into the LIMS. Rush projects and/or projects with short holds are prioritized into four categories during the triage process. The four categories are Critical A (HT or TAT ≤ 24 hrs.), Critical B (HT or TAT ≤48 hrs.), Critical C (HT or TAT ≤4 days), and Non-Critical (HT or TAT ≥5 days). Critical A projects should be processed first, followed by Critical B, Critical C, then Non-Critical.

12.2.1.1. Samples must be logged into the LIMS so the samples can be uniquely identified (lab sample identification numbers). These lab sample ID numbers are used to track the prep and analysis activities of the samples, as well as identify the sub-samples, digestates, extracts, and other sample byproducts. This laboratory code maintains an unequivocal link with the unique client field sample ID code assigned to each sample.

12.2.1.2. Using the COC and LIMS profile, login the container types, number of containers, matrix, and requested tests. Once saved, LIMS will give the project a workorder number and each sample will be given a sequential sample number. Any special instructions to the lab should be communicated as a comment at the time of login, such as Regulated Soils, OH VAP, WC, etc. Once logged in, a Sample Receipt Form (SRF) will be generated by LIMS.

12.2.1.3. For foreign or domestic regulated soils, the project must be commented in LIMS at the time of login and a LabTrack must be created in the Hazardous Disposal queue to alert the lab to the special handling and disposal requirements of regulated soils.

12.2.2. Cross-check the SRF with the COC and then generate labels for each sample container.

12.2.3. Cross-check the information on the sample container and the sample label and attach the sample label to the appropriate sample container. Inform the Project Coordinator or Project Manager if there are any discrepancies between the sample containers and the sample labels.

12.2.4. If any samples require analyses performed outside of the laboratory, prepare the samples for subcontracting according to the procedures listed in the SOP describing the subcontracting of analytical services, S-IN-C-003 **Subcontracting Samples**, or equivalent revision or replacement.

12.2.5. SRF Review: The Project Manager, Project Coordinator, or designated Client Services personnel must review and verify the following information by comparing the COC to the SRF. Some of this information may not be provided by the client and those fields should be left blank:

12.2.5.1. Report Recipient(s);

12.2.5.2. Invoice Recipient;

12.2.5.3. PO#;

12.2.5.4. Project Name;

12.2.5.5. Project Number;

12.2.5.6. Requested Due Date;

12.2.5.7. Sample ID;

12.2.5.8. Matrix;

12.2.5.9. Collection Date & Time;

12.2.5.10. Received Date & Time;

12.2.5.11. Analysis: Double check compound lists;

12.2.5.12. Comments for special instructions to the lab (Regulated Soils, OH VAP, WV, etc.);

12.2.5.13. Price;

12.2.5.14. Region Codes;

12.2.5.15. Work Region % Split (for Pace internal subcontracted work).

12.3. **Sample Storage**

12.3.1. Once unpacked, samples will be logged into the LIMS in a timely manner and returned to appropriate storage conditions as soon as possible. Labs must make every effort to keep samples under the required thermal conditions during the login process. For the exceptional case where samples are not logged in the day they were received, they must be stored under appropriate temperature-controlled conditions until login takes place. In all cases, the sample temperatures must be taken as soon after receipt as possible and the samples stored to maintain the required storage conditions while awaiting login or labeling.

12.3.1.1. For ESI BP-XA projects, samples must be kept in the cooler while being processed. If not kept in the cooler, the temperature must be checked and documented every 20 minutes during processing.

12.3.2. Once logged into the LIMS and labeled, samples are placed in the appropriate storage areas. Specific temperature requirements are outlined in the analytical methods, but general guidelines are outlined below:

12.3.2.1. Short hold samples are delivered directly to the laboratory.

12.3.2.2. Summa canisters and Tedlar bags are stored on designated shelving at ambient temperature.

12.3.2.3. Volatiles- Aqueous samples are stored by receiving date or by project number in segregated volatiles cooler. Associated trip blanks are stored with the samples.

12.3.2.4. Volatiles- Soil and other solid samples received preserved in methanol are stored by receiving date or by project number in a segregated volatile cooler or freezer. Associated trip blanks are stored with the samples.

12.3.2.5. Volatiles- Soil and other solid samples received preserved with a stir bar, or deionized water and a stir bar, are stored by receiving date or by project number in a segregated volatiles freezer. Associated trip blanks are stored with samples when compatible with storage conditions.

12.3.2.6. Volatiles- Soil and other solid samples received in 4oz containers or similar bottleware are stored by receiving date or by project number in a segregated volatile cooler. If required by client or program, these samples may be sub-sampled and preserved upon receipt. In order to preserve these samples when required, it is necessary to collect a 5g aliquot of the sample and transfer it to a 40mL vial. One of the following preservation options must be utilized:

12.3.2.6.1. Add 5mL of deionized water and a stir bar to the 5g aliquot and preserve by storing in a freezer until analysis, or;

12.3.2.6.2. Within 48 hours of collection in the field, the 5g aliquot must be immediately extracted with 5mL of methanol and stored in a segregated volatiles cooler until analysis, or;

12.3.2.6.3. Within 48 hours of collection in the field, the 5g aliquot can be preserved with 10mL of deionized water and a stir bar, stored in a segregated volatile cooler and analyzed within 48 hours of collection.

12.3.2.7. Volatiles- Soil and other solid samples received in Encore samplers must be managed within 48 hours of collection by freezing the Encore or extruding it.

12.3.2.7.1. If extruding the sample into a 40mL vial containing a stir bar or a stir bar and 10mL of deionized water, then the sample is stored in the segregated volatile freezer until analysis.

12.3.2.7.2. If extruding the sample into methanol, then the sample is extracted within 48 hours of collection and the sample is stored in a segregated volatile cooler until analysis.

12.3.2.7.3. NOTE: if samples are not received within 48 hours of collection or are not received with enough time to process the samples correctly within 48 hours of collection, this must be noted in a way that will be visible on the final report (e.g., footnote in LIMS).

12.3.2.8. General Chemistry/Semi-volatiles- Waters and other liquid samples are staged by receiving date or by project number on the shelves in the appropriate sample storage cooler.

12.3.2.9. General Chemistry/Semi-volatiles- Soils and other solid samples are staged by receiving date or by project number on the shelves in the appropriate sample storage cooler.

12.3.2.10. Metals Solids and Liquids: These samples are staged by receiving date or by project number on designated shelving in the laboratory or appropriate designated area. These samples may be stored at ambient temperature unless Mercury or Hexavalent Chromium analysis is needed. If Mercury or Hexavalent Chromium analysis will be performed, the samples are staged by receiving date or by project number in the appropriate sample storage cooler. Samples requiring low level mercury analysis by Method 1631 are taken to the clean room for ambient storage and preservation, as needed.

12.4. **Sample Retention and Disposal**

12.4.1. If samples must be returned to customers, the lab must take special care to ensure that the samples are not damaged during any handling, testing, storing, or transporting processes.

12.4.2. Samples may need to be retained longer than the normal sample retention time (45 days from sample receipt). Reasons for this extended sample retention include: customer, program, or contract requirements so that samples can be retained in a secure location for the customers that is designated as a long-term storage area. In these cases, the samples are noted in LabTrack, labeled, and segregated by extended hold.

12.4.3. Disposal of unconsumed samples: Refer to the laboratory SOPs regarding waste handling and disposal: **Waste Management and Handling S-IN-W-002**, and **Regulated Soil Handling S-IN-C-007**, or current revisions or replacements.

13. Quality Control

13.1. For any sample received at the laboratory that does not meet the sample acceptance, hold time or preservation criteria, the client must be contacted by project management and advised of the situation.

13.1.1. If the client instructs the laboratory to proceed with the analysis, all appropriate personnel/departments must be informed and the client approval must be documented on the SCUR or COC. Data will be appropriately qualified.

13.1.2. The client may also instruct the laboratory to preserve the samples at the laboratory prior to proceeding with analysis. This must be documented on the COC or the SCUR, and must be noted in the final laboratory report.

13.2. All supporting documentation related to sample custody must be retained by the laboratory. This includes: memorandums, fax transmissions, the original COC, all paperwork received with the COC, the completed SCUR form and copies of email transmissions. Please contact the laboratory QM/SQM for documentation retention time frames required.

13.3. Documenting discrepancies during receipt of samples:

13.3.1. The following are examples of client discrepancies that need to be documented on the appropriate paperwork (e.g., SCUR form):

13.3.1.1. Lost samples/insufficient sample volume;

13.3.1.2. Broken or missing bottles;

13.3.1.3. Missing COC;

13.3.1.4. Mislabeled bottles;

13.3.1.5. Preservation error;

13.3.1.6. Missing sample related details (date, time, sample type).

13.3.2. Pace sample management discrepancies will be documented on the SCUR form, COC or within the project files. Discrepancies attributable to errors and omissions on the part of the laboratory will be addressed and resolved through the formal corrective action process.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Hazards and Precautions - Use extreme caution in handling samples and wastes as they may be hazardous. Each reagent and chemical used in this method should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats, safety glasses, and ventilation hoods. SDS are on file and available to all personnel.

22.2. All personnel involved in sample management are responsible for complying with OSHA and DOT regulations. These regulations pertain to the safe handling and/or shipping of the chemicals specified in this procedure. Refer to the Sample Control Supervisor for any questions or concerns related to the safe handling and shipment of hazardous materials.

22.3. Other laboratory safety requirements are contained in the Chemical Hygiene Plan/Safety Manual. Immediate questions can also be addressed with the local Safety Officer.

23. Waste Management

23.1. Not applicable to this SOP.

24. Pollution Prevention

24.1. Not applicable to this SOP.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. The NELAC Institute (TNI) Standard- 2003 and 2009.

25.3. SW-846, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA, current revision.

25.4. American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1995, Standard Methods for the Examination of Water and Wastewater, A.E. Greenberg, L.W. Clesceri, A.D. Eaton and M.A.H. Franson, eds., 19th ed., American Public Health Association, Washington D.C.

25.5. U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

25.6. U.S. Environmental Protection Agency, 1988, Methods for Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

25.7. Code of Federal Regulations- most recent version.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Sample Acceptance Policy

26.2. Attachment II – Example Chain of Custody

26.3. Attachment III – Example Sample Condition Upon Receipt form

26.4. Attachment IV - Example Sample Container Count Form

27. Revisions

Document Number	Reason for Change	Date
S-IN-C-001-rev.07	1. Cover page: changed phone number, added SOP Template number and updated document control format. 2. Section 4.3: added that highly contaminated samples are double bagged. 3. Section 5.1: changed MSDS to SDS 4. Section 6: added Table 6.1 and updated the definition of matrix and MSDS to SDS. 5. Table 9.1: added ascorbic acid, sodium bisulfate and monochloroacetic acid. 6. Section 11.1.2: added more detail regarding courier procedures. 7. Section 11.5.7: added detail regarding required sample temperature upon receipt. 8. Section 11.1.5.8: added WI drinking water acceptance requirements. 9. Section 11.1.6: changed IR temp reading from an average of multiple bottles to a single reading of a representative sample. Added West Virginia sample temp requirements. 10. Section 11.3.1: added that chlorine checks should not be performed for containers upon which chemical preservation should not be checked as in Section 11.2.1. 11. Table 11.2: added 625, 608 and Cyanide. 12. Table 11.3: added bacteria, odor, color, MBAS and settleable solids. 13. Section 11.8: added section for sample retention and disposal. 14. Section 13.1: added that applicable personnel must read and understand this SOP. 15. Updated attachments.	13Oct2015
S-IN-C-001-rev.08	1. Adapted from SOT-ALL-C-001-rev.06. 2. Table 9.1: added requirements for residual chlorine test strips. 3. Table 10.1: added hexane 4. Section 12.1.3: added general statement regarding internal COC. 5. Section 12.1.7: added temperature measurement for West Virginia samples. 6. Section 12.1.11: added procedure for coolers received late in the day. 7. Section 12.1.13: removed TOC as a parameter not pH-checked at receipt. Added procedure to mark the lid of sample determined to be under-preserved and added statement that pH adjustments are made by the analytical departments. 8. Section 12.1.14: added details for handling samples determined to contain residual chlorine. 9. Table 12.2: added methods 9012 and 9014. 10. Section 12.2: added detail for current login procedures. 11. Section 12.3: added detail for current sample storage procedures. 12. Section 12.4: added use of LabTrack for tracking extended hold of samples. 13. Section 25.2: added years 2003 and 2009 to TNI reference. 14. Attachment I: removed TOC as parameter not pH-checked at time of receipt. 15. Attachment III: added example of SCUR.	30Oct2017
S-IN-C-001-rev.09	1. Cover page: added signature line for Sample Receiving Manager. 2. Section 12.1.3: revised language to match our process for signing the COC. 3. Section 12.1.5: added detail to procedure for measuring temperature blank. 4. Sections 12.1.6 – 12.1.8: added contingencies for measuring cooler temperature. 5. Section 12.1.9: added detail for measuring West Virginia sample temperatures. 6. Table 12.3: updated to current information. 7. Section 26.4: added Attachment VI for Sample Container Count 8. Attachments: updated all to current version.	6Aug2018

Attachment I – Sample Acceptance Policy

In accordance with regulatory guidelines, Pace Analytical facilities comply with the following sample acceptance policy for all samples received.

If the samples do not meet the sample receipt acceptance criteria outlined below, the Pace facility is required to document all non-compliances, contact the client, and either reject the samples or fully document any decisions to proceed with analyses of samples that do not meet these criteria. Any results reported from samples not meeting these criteria are appropriately communicated to the client.

Sample Acceptance Policy requirements**:**

1. Sample containers must have unique client identification designations, and dates and times of collection, that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC);
2. There must be clear documentation on the COC, or related documents such as the Sample Condition Upon Receipt (SCUR) form, that lists the unique sample identification, sampling site location (including state; some regulations may require city, county, etc.), date and time of sample collection, and name and signature of the sample collector;
3. There must be clear documentation on the COC, or related documents, that lists the requested analyses, the preservatives used, sample matrix, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.);
4. Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval;
5. Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval;
6. Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval;
7. Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval;
8. All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2^{o}C of the required temperature, or within the method-specified range. For samples with a required temperature of 4^{o}C, samples with a temperature ranging from just above freezing to 6^{o}C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
9. For all compliance **drinking water** samples, analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are improperly preserved (with the exception of VOA samples- tested for pH at time of analysis).
10. Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.

Attachment II – Example Chain-of-Custody Form

CHAIN-OF-CUSTODY Analytical Request Document

Pace Analytical

Chain-of-Custody is a LEGAL DOCUMENT - Complete all relevent fields

LAB USE ONLY- Affix Workorder/Login Label Here or List Pace Workorder Number or MTJL Log-in Number Here

ALL SHADED AREAS are for LAB USE ONLY

Company:	Billing Information:		
Address:			
Report To:	Email To:		
Copy To:	Site Collection Info/Address:		
Customer Project Name/Number:	State: / County/City: / Time Zone Collected: [ ]PT [ ]MT [ ]CT [ ]ET		
Phone: Email:	Site/Facility ID #:	Compliance Monitoring? [ ] Yes [ ] No	
Collected By (print):	Purchase Order # : Quote #:	DW PWS ID #: DW Location Code:	
Collected By (signature):	Turnaround Date Required:	Immediately Packed on Ice: [ ] Yes [ ] No	
Sample Disposal: [ ] Dispose as appropriate [ ] Return [ ] Archive: _____ [ ] Hold: _____	Rush: [ ] Same Day [ ] Next Day [ ] 2 Day [ ] 3 Day [ ] 4 Day [ ] 5 Day (Expedite Charges Apply)	Field Filtered (if applicable): [ ] Yes [ ] No Analysis: _____	

Container Preservative Type **

Lab Project Manager:

** Preservative Types: (1) nitric acid, (2) sulfuric acid, (3) hydrochloric acid, (4) sodium hydroxide, (5) zinc acetate, (6) methanol, (7) sodium bisulfate, (8) sodium thiosulfate, (9) hexane, (A) ascorbic acid, (B) ammonium sulfate, (C) ammonium hydroxide, (D) TSP, (U) Unpreserved, (O) Other _____

Analyses

Lab Profile/Line:

Lab Sample Receipt Checklist:
Custody Seals Present/Intact Y N NA
Custody Signatures Present Y N NA
Collector Signature Present Y N NA
Bottles Intact Y N NA
Correct Bottles Y N NA
Sufficient Volume Y N NA
Samples Received on Ice Y N NA
VOA - Headspace Acceptable Y N NA
USDA Regulated Soils Y N NA
Samples in Holding Time Y N NA
Residual Chlorine Present Y N NA
Cl Strips: _____
Sample pH Acceptable Y N NA
pH Strips: _____
Sulfide Present Y N NA
Lead Acetate Strips: _____

LAB USE ONLY:
Lab Sample # / Comments:

* Matrix Codes (Insert in Matrix box below): Drinking Water (DW), Ground Water (GW), Wastewater (WW), Product (P), Soil/Solid (SL), Oil (OL), Wipe (WP), Air (AR), Tissue (TS), Bioassay (B), Vapor (V), Other (OT)

Customer Sample ID	Matrix *	Comp / Grab	Collected (or Composite Start) Date	Collected (or Composite Start) Time	Composite End Date	Composite End Time	Res Cl	# of Ctns

Customer Remarks / Special Conditions / Possible Hazards:

Type of Ice Used: Wet Blue Dry None

Packing Material Used:

Radchem sample(s) screened (<500 cpm): Y N NA

SHORT HOLDS PRESENT (<72 hours) : Y N N/A

Lab Tracking #:

Samples received via: FEDEX UPS Client Courier Pace Courier

LAB Sample Temperature Info:
Temp Blank Received: Y N NA
Therm ID#: _____
Cooler 1 Temp Upon Receipt: __oC
Cooler 1 Therm Corr. Factor:___oC
Cooler 1 Corrected Temp: _____oC
Comments:

Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)	Date/Time:
Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)	Date/Time:
Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)	Date/Time:

MTJL LAB USE ONLY
Table #:
Acctnum:
Template:
Prelogin:
PM:
PB:

Trip Blank Received: Y N NA
HCL MeOH TSP Other

Non Conformance(s): YES / NO

Page: _____ of: _____

Attachment III – Example Sample Condition Upon Receipt Form

SAMPLE CONDITION UPON RECEIPT FORM

Pace Analytical

Project #:_____ **Date/Time and Initials of person examining**

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☐ Other _____

Tracking #: _____

Custody Seal on Cooler/Box Present: ☐ Yes ☐ No **Seals Intact:** ☐ Yes ☐ No

Packing Material: ☐ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other _____

Thermometer: 1 2 3 4 5 6 A B C D E F Ice Type: ☐ Wet ☐ Blue ☐ None | Samples collected today and on ice ☐ Yes ☐ No ☐ N/A

Cooler Temperature: _____ Ice Visible in Sample Containers?: ☐ Yes ☐ No ☐ N/A

(Initial/Corrected) Temp should be above freezing to 6°C If temp. is Over 6°C or under 0°C, was the PM Notified?: ☐ Yes ☐ No ☐ N/A

All discrepancies will be written out in the comments section below.

	Yes	No		Yes	No	N/A
Are samples from West Virginia? Document any containers out of temp.			checked?: **exceptions: VOA, coliform, LLHg, O&G, and any container with a septum cap or preserved with HCl.** All containers needing preservation are found to be in compliance with EPA recommendation (<2, >9, >12) unless otherwise noted.			
USDA Regulated Soils? (ID, NY, WA, OR,CA, NM, TX, OK, AR, LA, TN, AL, MS, NC, SC, GA, FL, or Puerto Rico)						
Chain of Custody Present:			Circle: HNO3 H2SO4 NaOH NaOH/ZnAc			
Chain of Custody Filled Out:			Dissolved Metals field filtered?:			
Short Hold Time Analysis (<72hr)?: Analysis:			Headspace Wisconsin Sulfide			
Time 5035A TC placed in Freezer or Short Holds To Lab:				Present	Absent	N/A
			Residual Chlorine Check (SVOC 625 Pest/PCB 608)			
			Residual Chlorine Check (Total/Amenable/Free Cyanide)			
Rush TAT Requested:			Headspace in VOA Vials (>6mm):			
Containers Intact?:			Trip Blank Present?:			
Sample Labels Match COC?: Except TCs, which only require sample ID			Trip Blank Custody Seals?:			

Comments:

F-IN-Q-290-rev.16,5Mar2018

Attachment IV – Example Sample Container Count Form

CLIENT: _____

COC PAGE ____ **of** ____

COC ID# _____ Project # _____

Sample Line Item	DG9H VG9H	AG0U	AG1H	AG1U	AG2U	AG3S	WGFU	SP5T	BP1U	BP2N	BP2S	BP2U	BP3B	BP3N	BP3S	BP3U	SBS DI Bulk Kit R				Matrix SI/Wt/NAL (Soil/Water/Non-Aqueous Liquid)	pH <2	pH >9	pH>12
1																								
2																								
3																								
4																								
5																								
6																								
7																								
8																								
9																								
10																								
11																								
12																								

Container Codes

Glass

DG9B	40mL Na Bisulfate amber vial	AG0U	100mL unpreserved amber glass
DG9H	40mL HCL amber voa vial	AG1H	1 liter HCL amber glass
DG9M	40mL MeOH clear vial	AG1S	1 liter H2SO4 amber glass
DG9P	40mL TSP amber vial	AG1T	1 liter Na Thiosulfate amber glass
DG9S	40mL H2SO4 amber vial	AG1U	1liter unpreserved amber glass
DG9T	40mL Na Thio amber vial	AG2N	500mL HNO3 amber glass
DG9U	40mL unpreserved **amber** vial	AG2S	500mL H2SO4 amber glass
VG9H	40mL HCL clear vial	AG2U	500mL unpreserved amber glass
VG9T	40mL Na Thio. clear vial	AG3S	250mL H2SO4 glass amber
VG9U	40mL unpreserved **clear** vial	AG3U	250mL unpreserved amber glass
VGFX	40mL w/hexane wipe vial	BG1H	1 liter HCL clear glass
VSG	Headspace septa vial & HCL	BG1S	1 liter H2SO4 clear glass
WGKU	8oz unpreserved clear jar	BG1T	1 liter Na Thiosulfate clear glass
WGFU	4oz clear soil jar	BG1U	1 liter unpreserved glass
JGFU	4oz unpreserved amber wide	BG3H	250mL HCl Clear Glass
		BG3U	250mL Unpreserved Clear Glass

Plastic / Misc.

BP1A	1 liter NaOH, Asc Acid plastic	BP3U	250mL unpreserved plastic
BP1N	1 liter HNO3 plastic	BP3Z	250mL NaOH, Zn Ac plastic
BP1S	1 liter H2SO4 plastic		
BP1U	1 liter unpreserved plastic	AF	Air Filter
BP1Z	1 liter NaOH, Zn, Ac	C	Air Cassettes
BP2A	500mL NaOH, Asc Acid plastic	R	Terra core kit
BP2N	500mL HNO3 plastic	SP5T	120mL Coliform Na Thiosulfate
BP2O	500mL NaOH plastic	U	Summa Can
BP2S	500mL H2SO4 plastic	ZPLC	Ziploc Bag
BP2U	500mL unpreserved plastic		
BP2Z	500mL NaOH, Zn Ac		
BP3B	250mL NaOH plastic		
BP3N	250mL HNO3 plastic		
BP3S	250mL H2SO4 plastic		

ATTACHMENT C-4

SUBCONTRACTING SAMPLES
PACE, INDIANAPOLIS

Pace Analytical Services
7726 Moller Road
Indianapolis, IN 46268
Phone: 317.228.3100
Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

SUBCONTRACTING SAMPLES

Reference Methods: N/A

Local SOP Number:	S-IN-C-003-rev.05
Effective Date:	October 2, 2017
Supersedes:	S-IN-C-003-rev.04
SOP Template Number:	SOT-ALL-C-003-rev.07

APPROVALS

General Manager — September 19, 2017 Date

Quality Manager — September 19, 2017 Date

Client Services Manager — September 19, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

This is **COPY#** _____ Distributed on _____ by _____ and is ____ **CONTROLLED**

Table of Contents

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to establish a uniform system in the event that samples must be transferred to another laboratory for analysis. The following procedures are intended to prevent any negative impact on data quality or turnaround time, maintain accurate records of shipped samples, and ensure proper revenue allocation.

2. Summary of Method

2.1. Samples are subcontracted to a Pace network laboratory or to an outside laboratory when the analysis cannot be performed by the owner region/laboratory. Samples are subcontracted only with the consent and approval of the client. The subcontracted laboratory must maintain current NELAC/TNI, or other federal program certification or other primary state accreditation for the state the samples originated from unless prior approval from the client is received to use an alternate laboratory. Whenever possible, arrangements for a subcontracted analysis must be made prior to start of the project.

2.2. Sample analysis may be subcontracted when senior lab management determines that the present workload of the laboratory prohibits the analysis of samples within the required hold times or project due date, the requested method/parameter has not been developed, or when the required certification or accreditation is not current.

2.3. All revenue must be properly allocated through the Laboratory Information Management System (LIMS) or alternate system. In the case of a new project with tests that must be subcontracted, the Account Executives should identify the subcontract labs during the quoting process.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP apply to all personnel involved in the process of subcontracting samples to another lab.

3.2. This SOP is applicable to all samples requiring transfer to another laboratory in order to meet holding time, certification, or method requirements.

3.3. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. Not applicable to this SOP.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Samples that will be subcontracted will be checked in using the same process as samples remaining in the owning lab. Adjustment of sample pH will be done by the work lab.

7.2. Samples to be shipped to another laboratory for analyses must be shipped according to the handling and preservation requirements of the analysis to be performed.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Quality Manual, Glossary Section.

8.2. **Sending Region** – This is the laboratory that originally received the samples and will be producing the invoice.

8.3. **Receiving Region** – This is the laboratory that receives the samples from another Pace laboratory.

8.4. **SI (Sub in) Code -** Subcontracting code used to define which area in the laboratory will receive the payment. This may have a different name depending on which LIMS is used.

8.5. **SO (Sub out) code** – Subcontracting code used to define that the samples were sent to a laboratory outside the Pace network. This may have a different name depending on which LIMS is used.

8.6. **Analysis code** - This is a group of data which describes a specific analysis. It is comprised of all the data necessary to perform procedures and report results.

8.7. **Sample Acceptance Form (SAF)**: form generated by LIMS system after a project is logged in and reviewed by the Project Manager. This form is sent to the client electronically. This may have a different name depending on which LIMS is used.

8.8. **Sample Receipt Form (SRF):** form generated by LIMS system after a project is logged in. Contains sample and project information. This may have a different name depending on which LIMS is used.

9. Equipment and Supplies

Table 9.1

Equipment/Supplies	Description/ Comments
Sample Labels	
Sample Containers and/or kits	
Coolers	
Plastic bags	
Blank Chains-of-Custody	
Bottle Order Database	
Preservatives	
Disposable pipettes	
Bubble wrap	
Absorbent Sheets or other packing materials	
USDA labels	

10. Reagents and Standards

10.1. Not applicable to this SOP.

11. Calibration and Standardization

11.1. Not applicable to this SOP.

12. Procedure

12.1. When it is determined that a Pace laboratory cannot perform an analysis, the Project Manager (PM) or Project Coordinator (PC) associated with those samples must locate and secure the services of another facility.

12.1.1. If the analysis is routine to the sending region and the sample must be shipped (e.g., due to capacity issues, equipment failure, etc.), then the department manager is responsible for notifying the PM that the samples must be shipped.

12.2. The PM/PC must obtain client approval before subcontracting the samples.

12.3. Contractual obligations must be considered in the decision of where to send samples. Client permission may be obtained verbally, but must be received via e-mail, facsimile, and/or writing prior to the submission of results. **Note:** Copies of telephone logs may be used as a form of documentation and should be copied into the project file.

12.3.1. All subcontract laboratories must submit proof of applicable accreditation prior to receiving samples from a Pace lab. There must also be a subcontract lab information form and proof of insurance on file.

12.3.2. Only pre-approved subcontract labs will be used unless a client requires Pace to use a specific subcontract lab.

12.3.3. If Pace wants to add another subcontract lab to the approved list of labs, refer to the procedures listed in SOP **S-IN-Q-027, Evaluation and Qualification of Vendors.**

12.3.4. If the subcontract laboratory is not approved to perform the work, Pace can either refrain from sending samples or initiate a formal inspection of the proposed facility.

12.3.5. The sending lab must communicate any extra information necessary for the subcontract lab to perform the work properly (i.e., technical specifications involved, specific storage requirements needed, etc.).

12.4. When subcontracting samples to another laboratory, the PM/PC must discuss the following information with the subcontracting laboratory:

12.4.1. Analyses/Methods;

12.4.2. Number of samples;

12.4.3. Matrix;

12.4.4. Receipt Date;

12.4.5. Due Date;

12.4.6. Dry weight for soil samples;

12.4.7. Holding Time Constraints;

12.4.8. Required reporting limits;

12.4.9. QC Deliverables;

12.4.10. Certification requirements;

12.4.11. USDA Soil Permit requirements (as necessary);

12.4.12. Required sample volumes and preservatives.

12.5. If samples are sent to another Pace laboratory (inter-regionally), the sending region will perform the following tasks:

12.5.1. Log the samples into the LIMS using the appropriate 'Sub-In' analysis codes inherent to the lab's LIMS, however named.

12.5.2. Obtain an Inter-Laboratory Work Order (IRWO) form (example Attachment I) or similar form, depending on the LIMS used.

12.5.3. Assign Inter-Laboratory Work Order (IRWO) number and complete some form of Interregional Sub-out log (this is optional).

12.5.4. Enter the following information into the IRWO Form (example Attachment I) or similar form. Keep a copy in the project file.

12.5.4.1. Today's date in the space marked "Date prepared" and the date the results are due to the sending region is noted in "Requested Completion Date".

12.5.4.2. Sending region, receiving region, state of sample origin, the type of QC deliverable, external client and sending Project Manager must be filled in on the IRWO form.

12.5.4.3. Sending Project number/ Work Order Number– assigned by LIMS in sending laboratory.

12.5.4.4. Check off what type of work is being sent, the requested reportable units, and whether to report the data moisture corrected (dry weight).

12.5.4.5. Enter method description, container type, quantity of containers, preservative, quantity of samples, and unit price.

12.5.4.5.1. Total Price, which is split between the two laboratories. The system will default to 80/20 for all tests (except for dioxin which defaults to a 90/10 split); if different enter the correct split. This means that 80% of the revenue goes to the work region and 20% of the revenue goes to the owner region.

12.5.4.5.2. Mark if the samples are to be returned to the sending laboratory; if checked no, the receiving laboratory is responsible for final disposition of the remaining sample volume.

12.5.4.5.3. Mark the matrix of the samples.

12.5.5. Attach a copy of the sub-in COC printed from LIMS (if applicable) and mark the appropriate box on the IRWO form.

12.5.6. Attach a copy of the Sample Condition Upon Receipt (SCUR) form that was completed during the sample staging process.

12.5.7. A copy of the Inter-Regional Work Order (or IRWO/Sub-COC) form must be placed in the project folder.

12.5.8. If sending extracts, include prep batch logs and standards prep information. Include compound list and reporting limits when clients request a special list.

12.5.9. All paperwork being shipped must be placed in a sealable bag, and may be emailed or faxed prior to shipment.

12.5.10. Once the samples are received in the receiving region, the following information is entered into the LIMS:

12.5.10.1. Project number – this will be the receiving lab's project number and is assigned by LIMS.

12.5.10.2. Sample number(s) - must be the sending lab's sample number. These numbers must appear on the report run from LIMS by the sending lab.

12.5.10.3. Client ID – from the sending region.

12.5.10.4. Receipt date – must be the date received by the receiving lab, not that of the original sending lab.

12.5.10.5. Split between the two labs. System will default to 80/20; if different enter the correct split. The percent entered should be the receiving region's share of the revenue.

12.5.10.6. Full charge for the work being completed.

12.5.10.7. Analysis code - will be the receiving lab's method specific analysis code.

12.5.11. Project completion- Receiving Region:

12.5.11.1. Deliver results to the sending lab.

12.5.11.2. Enter a Ship Date in the Project Edit screen, this closes out the project.

12.5.11.3. Sends the IRWO form to the ABM and may keep a copy in the project file.

12.5.12. Project completion- Sending Region:

12.5.12.1. Batch the interregional schedules (analysis codes) and validate, thus forcing them to completion.

12.6. If samples are sent outside of Pace, the following tasks must be performed once the subcontract laboratory has been approved:

12.6.1. Create new COC in LIMS using Client project and sample identifications but do not put Client name on owner COC (example Attachment II). Retain copy of COC and file with PM/PC paperwork.

12.6.2. Note: Attach Compound List and Reporting Limits if clarification is needed.

12.7. Once the reports are received from the subcontract labs (internal or external labs), the reports are collated with any information from the sending laboratory. All information pertaining to the analysis of the samples is fully disclosed to the client.

12.7.1. The subcontract lab must be noted clearly somewhere on the final report to the client (e.g., the cover letter). A comment may be added to the final report with wording such as "The samples were subcontracted to <Full Name and Address of the Subcontracted Laboratory> for <specific tests> analysis. Results of this analysis are reported on the <Full Lab Name> final report".

12.7.2. At a minimum, the subcontract lab must provide Pace with a method blank and LCS for all target analytes, where applicable to the test. This is the minimum amount of quality control necessary to evaluate the subcontract lab data.

12.8. The sending lab must make all pertinent sample receipt information available to the lab performing the actual sample analysis. If the sending lab has already performed sample receipt activities (e.g., preservation checks, etc.), this must be fully documented on the COC or SCUR that is sent to the lab running the samples. Adjustment of sample pH will be done by the work lab.

13. Quality Control

13.1. Minimum data review requirements: The sending lab must review the following information when receiving a final report from a subcontract lab. If any of these items is incorrect or lacking information, the PM/PC must contact the subcontract lab to obtain the correct information or to obtain a revised final report:

13.1.1. Verify analytical tests are correct per sample number;

13.1.2. Verify analyte lists are correct per test;

13.1.3. Verify that the minimum amount of quality control has been completed for each test (method blank and LCS).

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Not applicable to this SOP.

23. Waste Management

23.1. Not applicable to this SOP.

24. Pollution Prevention

24.1. Not applicable to this SOP.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. The NELAC Institute (TNI) Standard- 2003 and 2009.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Example Inter-Regional Work Order (IRWO).

26.2. Attachment II: Example Chain of Custody.

27. Revisions

Document Number	Reason for Change	Date
S-IN-C-003-rev.02	1. Cover page: removed reference to SOP Template. Revised format to Periodic Review from Annual Review format. Updated copyright information. 2. Table of Contents: removed reference to attachments. 3. Section 7 Responsibilities and Distribution removed because this information is contained in the Quality Manual. Removal caused section numbering to change. 4. Section 11 Procedure (previously Section 12): reorganized entire section and simplified the information in old section 12.5.4. 5. Section 15: added TNI reference. 6. Section 16: updated list of attachments. 7. Attachments: updated attachment versions and added two new attachments.	14Jun2011
S-IN-C-003-rev.03	1. Cover Page: added address to upper right corner of page 2. Table of Contents: added new Section 14, Method Modifications 3. New Section 14 added, Method Modifications.	17Jun2013
S-IN-C-003-rev.04	1. Cover page: changed phone number 2. Section 11.6.4: added as requirement for sub lab QC. 3. Attachment 1: updated	10Sep2015
S-IN-C-003-rev.05	1. Adapted from SOT-ALL-C-003-rev.07. 2. Section 25.2: added years 2003 and 2009 to TNI reference.	18Sep2017

Attachment I- Example Inter-Regional Work Order

INTER-LABORATORY WORK ORDER #_____
(To be completed by sending lab)

Sending Project No:	
Receiving Project No:	
Check Box for Consolidated Invoice:	☐
Date Prepared:	
REQUESTED COMPLETION DATE:	

Sending Region		Sending Project Mgr.	
Receiving Region		External Client	
State of Sample Origin		QC Deliverable	

All questions should be addressed to sending project manager.

Type of Work: Analytical ☐ Other (Identify) _____
Requested Reportable Units _____ Report Wet or Dry Weight? _____

WORK REQUESTED

Method Description	Container Type (include volume)	Quantity of containers	Preservative	Quantity of Samples	Unit Price	Amount
						$ -
						$ -
						$ -
						$ -
						$ -
						$ -
					TOTAL	$ -

Special Requirements: _____

Receiving Region Department	Acctg. Code	Totals from above	Revenue Allocation	
			Receiving Region (80%)	Client Services Dept. Sending Region (20%)
Microbiology	17		$ -	$ -
Metals	20		$ -	$ -
Wet Chem.	21		$ -	$ -
ICPMS	22		$ -	$ -
SV GCMS	30		$ -	$ -
SV GC & LC	31		$ -	$ -
V GC	33		$ -	$ -
V GCMS	34		$ -	$ -
Dioxin* (Includes Coplanar PCBs & PBDEs)	35		$ -	$ -
Radiation*	38		$ -	$ -
Air	39		$ -	$ -
Asbestos	60		$ -	$ -
Other(Specify)			$ -	$ -
Total			$ -	$ -

*Revenue Allocation (Receiving Region (90%) and Sending Region (10%))

FOR ANALYTICAL WORK COMPLETE THIS SECTION ALSO

Chain of Custody Included: ☐ Yes ☐ No Return Samples to Sending Region: ☐ Yes ☐ No

Matrix: ☐ Soil ☐ Water ☐ Air ☐ Other (Identify) _____

CONFIRMATION OF WORK COMPLETED

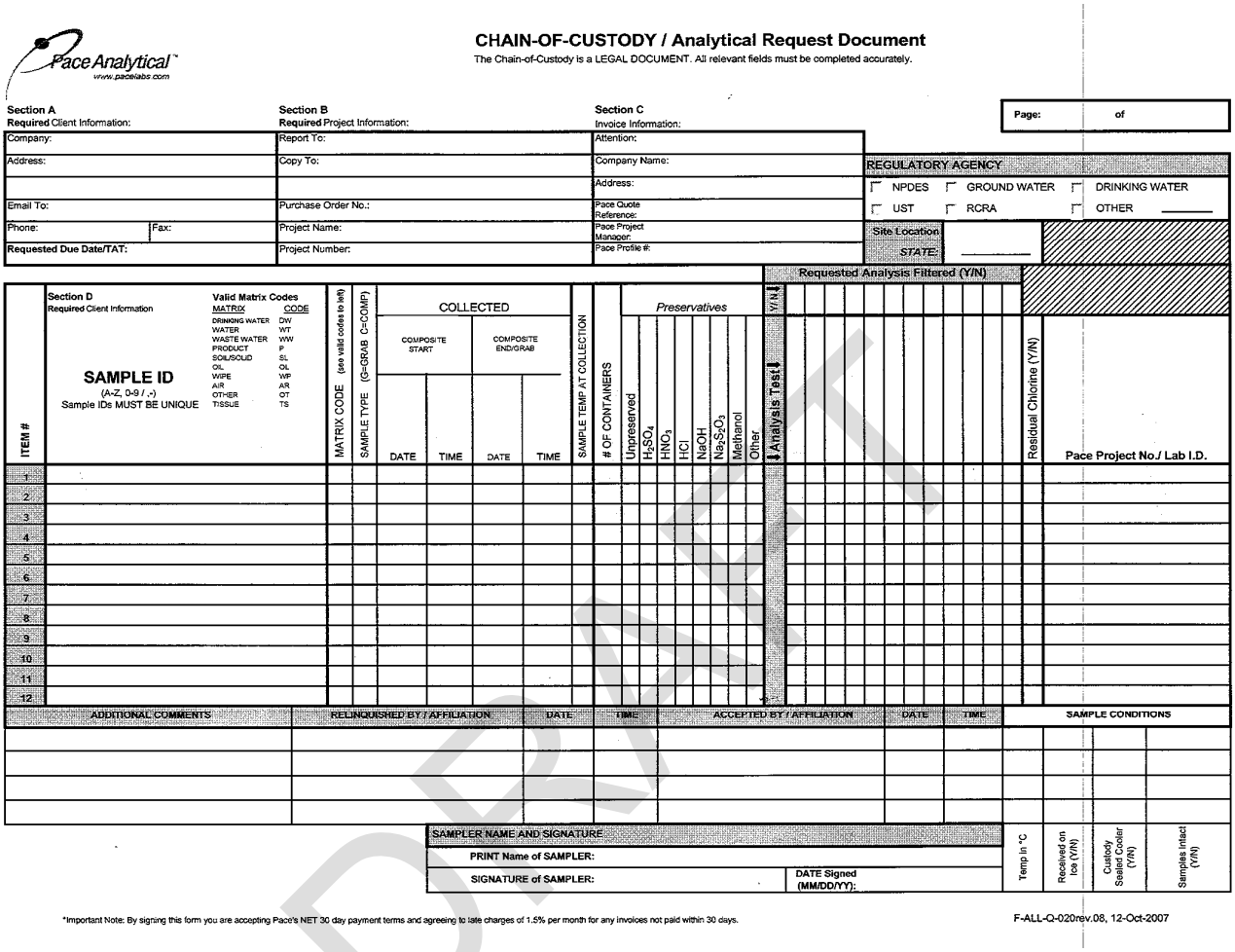
Date Completed: _____ Receiving Project Manager: _____

DISPOSITION of FORM

Original sent to the receiving lab - Copy kept at the sending lab.
When work completed: Original sent to the ABM at the receiving laboratory. Copies are made to corporate as needed.

FALLC002rev.07, 2December2009

Attachment II- Example Chain-of-Custody



Pace Analytical
www.pacelabs.com

CHAIN-OF-CUSTODY / Analytical Request Document
The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Page: of

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:
Company:	Report To:	Attention:
Address:	Copy To:	Company Name:
		Address:
Email To:	Purchase Order No.:	Pace Quote Reference:
Phone: Fax:	Project Name:	Pace Project Manager:
Requested Due Date/TAT:	Project Number:	Pace Profile #:

REGULATORY AGENCY
NPDES GROUND WATER DRINKING WATER
UST RCRA OTHER
Site Location
STATE:

Requested Analysis Filtered (Y/N)

ITEM #	Section D Required Client Information — SAMPLE ID (A-Z, 0-9 / ,-) Sample IDs MUST BE UNIQUE	Valid Matrix Codes: MATRIX / CODE — DRINKING WATER DW; WATER WT; WASTE WATER WW; PRODUCT P; SOIL/SOLID SL; OIL OL; WIPE WP; AIR AR; OTHER OT; TISSUE TS	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Y/N	Analysis Test	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1																					
2																					
3																					
4																					
5																					
6																					
7																					
8																					
9																					
10																					
11																					
12																					

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS

SAMPLER NAME AND SIGNATURE
PRINT Name of SAMPLER:
SIGNATURE of SAMPLER: DATE Signed (MM/DD/YY):

Temp in °C | Received on Ice (Y/N) | Custody Sealed Cooler (Y/N) | Samples Intact (Y/N)

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

F-ALL-Q-020rev.08, 12-Oct-2007

ATTACHMENT C-5

BOTTLE PREPARATION
PACE, INDIANAPOLIS

Pace Analytical Services, LLC
7726 Moller Road
Indianapolis, IN 46268
Phone: 317.228.3100
Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

BOTTLE PREPARATION

Reference Methods: N/A

Local SOP Number:	S-IN-C-004-rev.05
Effective Date:	January 8, 2018
Supersedes:	S-IN-C-004-rev.04
SOP Template Number:	SOT-ALL-C-004-rev.06

APPROVALS

General Manager — January 2, 2018 Date

Quality Manager — December 28, 2017 Date

Sample Receiving Supervisor — December 28, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to outline the procedures involved with bottle preparation and shipment.

2. Summary of Method

2.1. Bottle orders are prepared according to client needs and are purchased with or without preservatives only from approved vendors. All bottles are stored appropriately to avoid contamination.

2.2. Bottles, preservatives, field blanks, and trip blanks are prepared, packaged, labeled, and shipped following DOT regulation and client requests. An information packet is included with each box or cooler.

2.3. Bottles must be stored under specific conditions and in specific locations, typically by type of preservative or container. Bottles must be segregated from potential sources of contamination, including target analytes.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP apply to all personnel involved in the preparation and shipment of bottles used for sample collection.

3.2. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. Not applicable to this SOP.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Acceptable sample preservation, containers, and hold times are listed in Attachment II of this SOP. They may also be located in the Pace Quality Assurance Manual, the laboratory's method SOPs or in the applicable test method. Samples are stored separately from all standards and reagents and any known highly contaminated samples.

7.2. **NOTE**: To avoid contamination, no food or drink products can be located near the areas where samples are unpacked, labeled, or staged or where outgoing bottles are prepared.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual.

8.2. **Chain-of-Custody (COC):** a form used to record the field identification of samples collected, analyses requested, date and time of collection, sample preservation used, and traceability of samples from time of collection until delivery to the laboratory. This is a legal document.

8.3. **Laboratory Information Management System (LIMS):** a computer system used to manage the flow and traceability of environmental samples and associated data within the laboratory.

8.4. **Matrix:** the bulk characteristics of a sample. See Table 8.1 below.

8.5. **Safety Data Sheet (SDS):** contains information on chemicals used in the laboratory.

8.6. **Sample Custody:** a sample is considered to be in someone's custody if:

8.6.1. It is in one's physical possession;

8.6.2. It is in someone's view, after being in someone's physical possession;

8.6.3. It is kept in a secured area, restricted to authorized personnel only.

8.7. **Sample Condition Upon Receipt (SCUR) form:** a form used to record the condition of samples received in the laboratory.

8.8. **Sample Receipt Form (SRF):** form generated by LIMS system after a project is logged in. Contains sample and project information.

8.9. **UN Number** - identification numbers preceded by the letters UN are associated with proper shipping names considered appropriate for international and domestic transportation. These shipping names along with the identification numbers are located in the Federal Register (49CFR172.101).

Table 8.1

NELAC/TNI defined matrix	Pace Analytical defined matrix
Aqueous: any liquid sample not defined as drinking water. Includes surface water, groundwater, effluents, TCLP, and other extracts.	Waters: includes groundwater, wastewaters, drinking waters, effluents, and any free-flowing liquids.
Drinking water: any aqueous sample that has been designated as potable or potentially potable.	Not assigned as a separate matrix, but samples are assigned to drinking water methods.
Non-aqueous liquid: any organic liquid with <15% settleable solids.	Other or Non-aqueous Liquid: Assigned as a separate matrix from waters.
Biological tissue: any sample from a biological origin such as fish tissue or plant material.	Tissue: would include tissue and plant samples.
Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids	Soils: includes soils, sediments, sludges; other solid materials such as wood, metal, etc. may fall under another heading.
Chemical waste: a product or by product of an industrial process that results in a matrix not defined above.	Other or Non-aqueous Liquid: Assigned as a separate matrix from waters.
Air: vapor samples including those contained within sorbent tubes, filters or other devices.	Air: vapor samples including those contained within sorbent tubes, filters, or other devices.
No corresponding matrix to wipe; wipes would be included in with solids.	Wipe: includes wipe samples or swabs taken to check for surface contamination.

9. Equipment and Supplies (Including Computer Hardware and Software)

Table 9.1

Equipment/Supplies	Description/ Comments
Sample Labels	
Sample Containers and/or kits	
Coolers	
Plastic bags	
Blank COCs	
Bottle Order Program	
Preservatives	
Disposable pipettes	
Bubble wrap	
Absorbent Sheets or other packing materials	
LIMS Computer System	Epic Pro

10. Reagents and Standards

10.1. All reagents used in this procedure must be labeled with:

10.1.1. Laboratory reagent identification number;

10.1.2. Unless otherwise noted, the name and concentration of the reagent;

10.1.3. Date the reagent was received, opened and, as needed, prepared;

10.1.4. Person preparing reagent;

10.1.5. Expiration date.

10.2. **Reagents: Table 10.1**

Reagent	Formula	Concentration
Sulfuric Acid	H_2SO_4	1:1
Nitric Acid	HNO_3	20%
Hydrochloric Acid	HCl	1:1
Sodium Hydroxide	NaOH	50% or Pellets
Sodium Thiosulfate	$Na_2S2O_3 \cdot 5H_2O$	
Zinc Acetate Solution (for sulfide)		
Methanol	MeOH	Purge and Trap Grade
Ascorbic Acid (for cyanide)		
Sodium Bisulfate		
Hexane for PCB Wipes		Pesticide Grade

10.3. For acids, bases and other reagents obtained from other laboratory departments, this information is located in the department reagent preparation log. In the event that these reagents are managed within the Sample Receiving group, the department must maintain its own reagent preparation log.

10.4. Some Pace labs use pre-preserved sample containers. In this case, documentation from the vendor must be maintained for bottleware and preservation traceability.

11. Calibration and Standardization

11.1. Pipettes and other equipment used for measuring volumes must be calibrated according to S-IN-Q-157, **Support Equipment** or its equivalent revision or replacement.

12. Procedure

12.1. A Bottle Order Request form is completed after it is determined what the client needs for a specific project or field event.

12.2. The bottle order form is delivered electronically to the bottle prep area and printed on the day the bottle kit is to be assembled and shipped.

12.2.1. If the order needs to be shipped out or picked up within 24 hours, or if the order is very large, the Project Manager (PM) should tell the bottle prep personnel that the order will require immediate attention and enter the bottle order into the bottle order database as a RUSH order.

12.2.2. If the order does not require expedited handling, the order is entered into the bottle order database using the date the bottle order is due to the customer.

12.3. Sample management personnel will review the bottle order to determine if there is a sufficient stock of bottles to fill the order as written, and to clarify any special instructions listed.

12.3.1. Any problems or questions should be directed to the person who completed the bottle order or a PM or the Client Services Manager (CSM) if they are not available.

12.3.2. Each bottle order is assigned a unique ID number to use for future tracking of bottles and reagents used.

12.4. Purchasing the bottles or containers:

12.4.1. Only pre-cleaned, new, certified bottles are used to fill orders for containers, where available. These must be purchased from an approved vendor.

12.4.2. Containers can be purchased by the following two options: 1) Pre-preserved by the supplier, and 2) Unpreserved.

12.4.3. Where certified bottles are not available, the lab may be required to demonstrate that the containers are free from interferences and contamination when compared to the analytes of interest. This demonstration will be dependent on the regional or client driven quality assurance requirements. If the laboratory staff is unsure of these requirements, consult the local Senior Quality Manager (SQM)/Quality Manager (QM) for more information.

12.4.4. Containers that have been returned from clients must not be reused to prepare bottle orders unless the COC seal has not been broken on the outer container box. The packing material must not be reused. If the containers are returned to the laboratory outside of the sealed box and the COC seal on the individual bottles has not been broken, they may be reused only when clearly segregated for the client returning the bottles.

12.4.5. The Certificate of Analysis (COA) for each bottle lot is filed in the appropriate folder for future reference. Record the lot number for each bottle type used in the bottle kit on the bottle order form.

12.5. Sample Container Labels:

12.5.1. Sample labels can be the standard Pace Analytical blank roll or sheet labels, or can be preprinted using the laboratory's label printing procedure.

12.5.2. Sample labels may be affixed to each container or provided unattached depending on the needs of the client. When unspecified by the client, do not attach the labels to the bottles:

12.5.2.1. When sending the labels attached to the container, affix a sample label to each container before the preservative is added or, as the bottles are prepared for shipment or delivery.

12.5.2.2. Always place the label on the bottle as close to the bottom of the bottle as possible. Make sure the label is not wrinkled or creased, and that it is as straight as possible.

12.5.2.3. If a client requests that labels be shipped separate from the bottles, make sure the bottles are marked with the preservative used.

12.6. Absorbent Material – Shipments being prepared with one or more preservative (Table 10.1) must have an absorbent sheet placed on the bottom of the cooler, in order to absorb all the hazardous materials in case of a spill. **This is a DOT requirement.**

12.7. Container Preparation with Preservative - Containers requiring the addition of preservatives prior to shipment must be preserved according the preservation chart listed in Attachment II.

12.7.1. Purchase the required preservatives at the appropriate concentrations from an approved vendor, or prepare the appropriate reagents from stock solutions according to the specifications in Table 10.1.

12.7.1.1. Each new bottle of reagent must be recorded in the Sample Receiving Reagents and Standards Logbook or in the relevant department reagent logbook.

12.7.1.2. Remove the lids from the containers to be preserved, taking care to place the lids on the counter with the inside facing up.

12.7.1.3. Add the appropriate amount of preservative to each bottle, and replace the lid. Make sure the lid is tight.

12.7.1.4. Wipe any excess preservative that remains on the outside of the container using a paper towel. Discard the towel after use.

12.7.1.5. Containers with corrosive preservatives require a positive means of securing the lid or cap to prevent leakage (e.g., tape over cap). **This is a DOT requirement.**

12.8. Preparation for Preservation at time of Sample Collection - A client may also request that the preservatives be shipped separately from the bottles, for preservation at the time of sample collection. This is not the preferred approach for Pace Analytical and the client should be discouraged from doing so wherever possible. Nevertheless, if the client insists on using this approach, prepare the container kit as follows:

12.8.1. Prepare the preservatives for shipping by pouring them into an appropriate shipping container (consult the laboratory DOT trained shipping specialist), labeling the container with the contents, the date filled and the initials of the person preparing the solution. Also include the appropriate equipment (e.g., disposable pipettes) for field preparation.

12.8.2. Include any instructions for adding the preservatives to the sample containers in the field.

12.8.3. Laboratory personnel cannot add more than **30mL** of preservative to any containers due to the rules for small quantity exception. **This is a DOT requirement.**

12.9. Select the prepared bottles needed to fill the bottle order and gather them on the counter.

12.9.1. Group the bottles by bottle type, or by sampling location, depending on the request on the bottle order.

12.9.2. If there are preprinted sample labels provided, affix them to the appropriate bottles. These can be placed over unmarked labels if the bottles are already pre-labeled.

12.10. Field Blank Preparation

12.10.1. Prepare a set of empty bottles defined on the bottle order form.

12.10.2. Include enough reagent grade laboratory water to fill all of the bottles requested for the field blank. This should be shipped in separate approved containers, per method, labeled with date prepared.

12.11. Trip Blank Preparation

12.11.1. Prepare a set of empty bottles defined on the bottle order form.

12.11.2. Aqueous trip blank samples are usually provided only for volatile analyses. However, clients may request a trip blank for all bottle types that they are using in the field.

12.11.2.1. For aqueous trip blanks, fill each of the bottles with reagent grade laboratory water and label them with the following information: LABORATORY TRIP BLANK – DO NOT OPEN – RETURN WITH SAMPLES. Volatile vials must be filled so that there is no headspace in the vials.

12.11.3. Methanol trip blanks may be prepared to document the transport of methanol preserved volatile samples. These may be taken directly from the same kit or source that the sample containers are taken from.

12.11.4. Mark the date that the bottles were filled at the laboratory on each bottle and affix a custody seal on the vials to assure the bottles are not tampered with.

12.11.5. The shelf life for trip blanks preserved in the laboratory will be consistent with the shelf life of the sample vials (see corresponding expiration date on the COA), or 1 year, whichever is sooner for aqueous trip blanks, or 3 months, whichever is sooner for methanol trip blanks.

12.11.6. The holding time for trip blanks will be based on the date of sample collection from the first sample collected in that project, unless the client has documented a date on the COC.

12.11.7. The preservation for trip blanks must be consistent with the preservatives utilized in sample collection.

12.12. Wrap all containers in appropriate packing material to prevent breakage, typically foam, bubble bags, or more bubble wrap. When shipping glass containers in the original box, provide sufficient bubble bags or bubble wrap for the return of samples.

12.13. Select a shipping container for the samples:

12.13.1. If a box is requested, pack the samples as firmly as possible into the box. The contents of the box should not move around when shaken.

12.13.2. If a cooler is requested, the cooler must be large enough to allow room for the sample containers to be returned with enough ice to cool the samples to $\leq 6^{\circ}$ during return shipment.

12.13.3. Choose a cooler that is clean and dry.

12.14. Pack the containers for shipment or delivery to the client:

12.14.1. Place a layer of absorbent sheet material on the bottom of the cooler. Place a layer of bubble wrap on top of the absorbent sheet.

12.14.2. Open a large trash bag and line the cooler with it. All sample containers should be placed inside the trash bag liner in the cooler.

12.14.3. If the bottles are to be packed by sampling location (i.e., well, outfall, etc.), select the requested number of each type of bottle for each sample and bag them in a Ziploc bag.

12.14.3.1. If the bottles are pre-labeled with the location, make sure that all of the bottles chosen have the same client ID. Place glass containers inside bubble bags before packing the cooler.

12.14.3.2. Mark the bag with the location ID if requested.

12.14.3.3. Place the bag in the cooler, making sure that any bottles containing preservative are upright to avoid spillage.

12.14.3.4. Add additional sets of bottles to the cooler, as space will allow.

12.14.4. If the bottles are to be packed by bottle type, pack the cooler or box with the bottles. If the bottles contain preservatives, pack the bottles upright to help prevent spillage. If the bottles are to be packed by bottle type, pack the cooler or box with the bottles.

12.14.5. Allow room for the sample containers to be returned with enough ice to cool the samples. This space should be filled with extra packing material to prevent breakage during shipment of the empty containers.

12.15. Place a temperature blank in the cooler.

12.15.1. Temperature blanks are prepared by filling a small plastic bottle with tap water and replacing the cap.

12.15.2. The bottle should be labeled "LABORATORY TEMPERATURE BLANK – RETURN WITH SAMPLES – DO NOT OPEN". A brightly colored label should be used to call the sampler's attention to the temperature blank.

12.16. Add an information packet to the cooler or box which contains the following information in a Ziploc bag:

12.16.1. COC Forms (F-ALL-Q-020) (number specified on the bottle request form);

12.16.2. Additional sample labels, if required;

12.16.3. Cooler Custody Seals, if required;

12.16.4. Sampling instructions, if required;

12.16.5. 'Tips for Packing Your Cooler' sheet;

12.16.6. Pace Analytical return address label;

12.16.7. Appropriate description of contents of sample containers (e.g., preservatives);

12.16.8. Copy of the bottle request form;

12.16.9. Pre-paid return shipping label, if required;

12.16.10. Sample Acceptance Policy (F-ALL-C-002).

12.16.11. Short Hold/RUSH stickers as needed.

12.17. Seal the cooler or box with packing tape or bands for shipment:

12.17.1. Tape the lid down tightly or band the cooler or box with an auto band-sealer so that it will not come open during shipment.

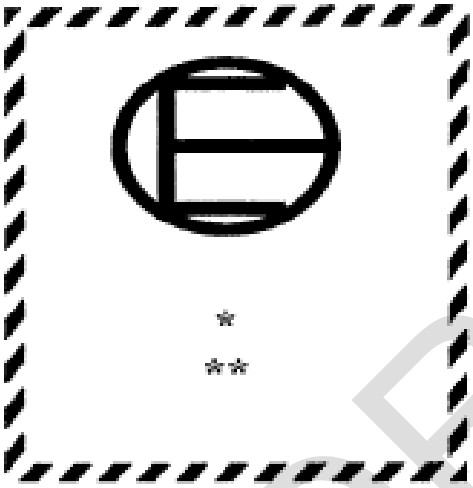
12.17.2. Wrap a continuous tape strip completely around the cooler (or use an auto band sealer).

12.17.3. If using tape, seal the cooler with one custody seal if requested by the client, and sign and date the seal.

12.18. Label the outside of the cooler with appropriate client information, i.e., project name, project manager's name, delivery date, etc. If the cooler is being shipped all necessary information should be present on the shipping label.

12.19. Preservatives are able to be shipped without hazardous material labeling or restrictions so long as 49 CFR 173.4 and 49 CFR 173.4a are obeyed (regulations commonly referred to as Small Quantity Exemptions). These regulations include restrictions already discussed such as having a positive means of securing the cap (Section 12.7.1.5) and volume limit of 30mL per container (Section 12.8.3). These shipments must also be labeled according to these regulations to properly designate the containers as exempt from DOT regulations.

12.19.1. All preservative shipments designated for air transport must have the following label:



12.19.1.1. The "*" must be replaced by the primary hazard class, or when assigned, the division of each of the hazardous materials contained in the package. The "**" must be replaced by the name of the shipper or consignee if not shown elsewhere on the package. Refer to Attachment I for Hazard Classes.

12.19.1.2. This label must be not less than 100mm (3.9inches) x 100mm (3.9inches) and must be durable and clearly visible.

12.19.2. Preservative shipments are able to be sent without hazardous material restrictions or hazardous material labeling because steps are taken to ensure that the package conforms to 49CFR 173.4a (the exception for limited quantities). When preparing a preservative shipment, the shipper must understand 49 CFR 173.4 and obey all parts of the regulation. See Attachment III for a list of these rules. All preservative shipments are designated for ground transport only and must have a label that states, "**This package conforms to 49 CFR 173.4 for domestic highway or rail transport only".** Here is an example:

12.20. Drop shipment of sample containers:

12.20.1. If a drop shipment of sample containers is required by a client, that is if the bottles are not going to be prepared and shipped from the laboratory, Pace Analytical will order the sample containers from a certified vendor. The vendor will ship the preserved containers directly to the client along with a Certificate of Analysis for the containers.

12.20.2. **Note: Nitric acid preserved containers drop-shipped by air must contain nitric acid at a concentration less than 20%.**

12.21. Courier Delivery:

12.21.1. Place the bottle kit in the appropriate location for courier deliveries, and notify the courier of the scheduled delivery date, contact, and location.

12.21.2. A label is affixed to the top of the cooler with the delivery information or the information is documented and placed in a specific location for the courier to know where to go and what to pick up.

12.22. Outside Carrier Shipments:

12.22.1. Schedule the package shipment and affix the shipping label to the container to be shipped.

12.22.2. Place the cooler or package to be shipped in the appropriate area for pickup by the outside carrier.

13. Quality Control

13.1. All supporting documentation related to sample custody must be retained by the laboratory. This includes; memorandums, fax transmissions, all paperwork received copies of email transmissions.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Hazards and Precautions - Use extreme caution in preparing bottles with preservatives (i.e. nitric acid) as they may be hazardous. Each reagent and chemical used in this method should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats, safety glasses and ventilation hoods. SDS are on file and available to all personnel.

22.2. All personnel involved in bottle preparation and shipment are responsible for complying with OSHA and DOT regulations. These regulations pertain to the safe handling and/or shipping of the chemicals specified in this procedure. A reference file of SDSs is available to all personnel. Refer to the Sample Control Supervisor for any questions or concerns related to the safe handling and shipment of hazardous materials.

22.3. Other laboratory safety requirements are contained in the Chemical Hygiene Plan/Safety Manual. Immediate questions can also be addressed with the local Safety Officer.

23. Waste Management

23.1. Not applicable to this SOP.

24. Pollution Prevention

24.1. Not applicable to this SOP.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. The NELAC Institute (TNI); "Quality Systems"- 2003 and 2009.

25.3. Chapter 3, "Inorganic Analytes;"SW-846, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA, Rev. 3, 1996.

25.4. Code of Federal Regulations, Chapter 40, Part 136.3, Table II.

25.5. American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1995, Standard Methods for the Examination of Water and Wastewater, A.E. Greenberg, L.W. Clesceri, A.D. Eaton and M.A.H. Franson, eds., 19th ed., American Public Health Association, Washington D. C.

25.6. U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water And Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

25.7. U.S. Environmental Protection Agency, 1996, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Office of Solid Waste and Emergency Response, Washington D.C.

25.8. U.S. Environmental Protection Agency, 1988, Methods for Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – List of Preservatives and Hazard Classes.

26.2. Attachment II – Sample Containers, Preservation, and Holding Time

26.3. Attachment III – Regulation 49 CFR 173.4.

26.4. Attachment IV – Regulation 49 CFR 173.4a.

27. Revisions

Document Number	Reason for Change	Date
S-IN-C-004-rev.04	1. Adapted from Corporate SOT-ALL-C-004-rev.05 2. Cover page: added lab address and revised document control format 3. Table 10.1: added hexane 4. Section 12.4.5: added requirement that only one CofA from each bottle lot be maintained. 5. Section 12.5: removed recommendation to place the label as low as possible on the bottle and added instructions for printing pre-printed labels. 6. Section 12.11.3: added Terracore and bulk supply to clarify. 7. Attachment II: added Indiana RISC and Ohio VAP allowance for preservation of water for Hexavalent Chromium analysis.	03Dec2015
S-IN-C-004-rev.05	1. Adapted from Corporate SOT-ALL-C-004-rev.06. 2. Table 8.1: removed saline matrix and revised Pace matrix for non-aqueous liquids and chemical waste. 3. Table 10.1: revised to match Pace Indy reagents. 4. Section 12: updated to reflect electronic bottle order database. 5. Section 12.14: updated to match Pace Indy cooler packing procedures. 6. Section 12.15: added that brightly colored temperature blank labels should be used. 7. Section 12.16: added short hold/rush stickers. 8. Section 25: added years 2003 and 2009 to TNI reference. 9. Attachment I: updated.	26Dec2017

Attachment I – List of Preservatives and Hazard Classes

CHEMICAL	CLASS/DIVISION	UN NUMBER
Acetone	3	1090
Hexane	3	1208
Methanol	3	1230
Hydrochloric Acid	8	1789
Nitric Acid	8	2031
Sodium Bisulfate	8	2837
Sodium Hydroxide	8	1824
Sulfuric Acid	8	1830
Trisodium Phosphate	8	3262
Sodium thiosulfate	none	none

Class 3 = Flammable liquid
Class 8 = Corrosive material

UN Number - Identification numbers preceded by the letters UN are associated with proper shipping names considered appropriate for international transportation as well as domestic transportation.

Attachment II – Sample Containers, Preservation, and Holding Times

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acid Base Accounting	Sobek	Solid	Plastic/Glass	None	N/A
Acidity	SM2310B	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	$\leq 6^oC$	14 Days
Actinides	HASL-300	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid	Plastic/Glass	None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass (NY requires separate bottle filled to the exclusion of air)	$\leq 6^oC$	14 Days
Alkylated PAHs		Water	1L Amber Glass	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid	8oz Glass	$\leq 10^oC$	1 Year/40 Days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4 , bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	$\leq 6^oC$; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4 , bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	$\leq 6^oC$	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	$\leq 6^oC$	48 hours
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Asbestos	EPA 600/R-93/116	Solid	Plastic/Glass; bulk- 2" square; popcorn ceiling- 2tbsp; soil- 4oz	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	$\leq 6^oC$; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	$\leq 6^oC$	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; $\leq 6^oC$; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	$\leq 6^oC$; pH<2 1:1 HCl (optional)
Biomarkers		Solid	$\leq 10^oC$	1 Year/40 Days	$\leq 10^oC$
BOD/cBOD	SM5210B	Water	Plastic/Glass	$\leq 6^oC$	48 hours
Boiling Range Distribution of Petroleum Fractions	ASTM D2887-98	Product	10mL glass vials	$\leq 6^oC$	N/A
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	28 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	72 Hours
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloroacetic acid pH <3; $\leq 6^oC$	28 Days
Carbamates	8318	Water	Glass	Monochloroacetic acid pH 4-5; $\leq 6^oC$	7/40 Days
Carbamates	8318	Solid	Glass	$\leq 6^oC$	7/40 Days
Carbon Specific Isoptope Analysis (CSIA)	AM24	Water	40mL clear VOA vial with TLS	$\leq 6^oC$, trisodium phosphate or HCl	N/A

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Cations (Ferrous Iron, Ferric Iron, Divalent Manganese)	7199 modified	Water	40mL clear VOA vials with mylar septum	$\leq 6^oC$; HCl	48 Hours
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorinated Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil	$\leq 6^oC$	48 Hours to filtration
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq 6^oC$	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	24 Hours
Coliform, Fecal	SM9221E	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Coliform, Fecal	SM9221E	Solid	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	24 Hours
Coliform, Total	SM9222B	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Coliform, Total	SM9221B	Solid	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Coliform, Total, Fecal and E. coli	Colilert/ Quanti-tray	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	30 Hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	$\leq 6^oC$	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/ 9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; $\leq 6^oC$; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present-SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	$\leq 6^oC$	14/40 Days
Diesel Range Organics-Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; $\leq 6^oC$	14/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	$\leq -10^oC$	1 Year if frozen/40 Days
Diesel Range Organics- TPH DRO	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	$\leq 6^oC$ but above freezing	28 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; $\leq 6^oC$	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	$\leq 6^oC$	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	$\leq 6^oC$; pH <2 HCl	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	$\leq 6^oC$	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	1 year
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	$\leq 6^oC$	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	$\leq 6^oC$	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	$< -10^oC$	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	7/40 Days
Diquat/Paraquat	549.2	Water	Amber Plastic	$\leq 6^oC$; $Na_2S_2O_3$	7/21 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14 Days
Endothall	548.1	Water	Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$	7/14 Days
Enterococci	EPA 1600	Water	100mL Plastic	$\leq 10^oC$	8 Hours
Enterococci	Enterolert	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Explosives	8330/8332	Water	1L Amber Glass	$\leq 6^oC$	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Water	1L Amber Glass	pH < 2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	NJ EPH	Solid	4oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	8 Hours
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	Glass	None	Immediate
Flashpoint/ Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	≤ 6°C; pH <2 H_2SO_4 or HCl	7/40 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics (C3-C10)	8260B modified	Water	40mL vials	≤ 6°C; HCl	14 Days
Gasoline Range Organics (C3-C10)	8260B modified	Solid	4oz Glass Jar	≤ 6°C	14 Days
Gasoline Range Organics- Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics- Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6°C	7 Days unpreserved; 14 Days preserved

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Gasoline Range Organics- NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	$\leq 6^oC$; packed jars with no headspace	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; $\leq 6^oC$	14 Days
Gasoline Range Organics- Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	$\leq 6^oC$ in MeOH	21 Days
Glyphosate	547	Water	Glass	$\leq 6^oC$; $Na_2S_2O_3$	14 Days (18 Months frozen)
Grain Size	ASTM D422	Solid	Not specified	Ambient	N/A
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; $\leq 6^oC$	14/7 Days if extracts stored $\leq 6^oC$ or 14/14 Days if extracts stored at $\leq -10^oC$
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	$\leq 10^oC$; $Na_2S_2O_3$	8 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/ SM3500Cr-B, C	Water	Plastic/Glass	$\leq 6^oC$	24 Hours (see note 4)
Hexavalent Chromium	218.6/SM3500Cr-B, C	Water	Plastic/Glass	Ammonium Buffer to pH 9.3-9.7 Refer to local SOP	28 Days (see note 4)
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH >8	14 Days (see note 4)
Hexavalent Chromium	7196 (with 3060)	Solid	Glass	$\leq 6^oC$	30 Days from collection to extraction and 7 days from extraction to analysis

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Hydrocarbons in Vapor	AM4.02	Vapor	20cc vapor vial with flat septum	None	N/A
Hydrogen by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Light Hydrocarbons by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Light Hydrocarbons in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Lipids	Pace Lipids	Tissue	Plastic/Glass	$\leq$ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	$\leq$ 6°C	28 Days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	$\leq$ -10°C	180 Days if frozen

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175; PM01/AM20GAx	Water	20mL vials	HCl; or trisodium phosphate or benzalkonium chloride and $\leq 6^oC$	14 Days; 7 Days unpreserved
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	28 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
Methanol, Ethanol	8015 modified	Water	40mL vials	$\leq 6^oC$	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	$\leq 6^oC$	14 Days
Methyl Mercury	1630	Water	Teflon/ fluoropolymer	Fresh water- 4mL/L HCl; Saline water- 2mL/L H_2SO_4 (must be preserved within 48 hours of collection)	6 months
Methyl Mercury	1630	Tissue	2-4oz glass jar	$\leq 0^oC$	28 Days; ethylated distillate 48 hours
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq 6^oC$	28 Days
Nitrogen, Total Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	$\leq 6^oC$	28 Days
Nitrogen, Total Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq 6^oC$	28 Days
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	$\leq 6^oC$	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	$\leq 6^oC$	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq 6^oC$	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq 6^oC$	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	28 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	72 Hours
Odor	SM2150B	Water	Glass	$\leq 6^oC$	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; $\leq 6^oC$	28 Days
Oil and Grease/HEM	9071	Solid	Glass	$\leq 6^oC$	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Oil Range Organics	8015	Solid	Glass	$\leq 6^oC$	14/40 Days
Oil Range Organics	8015	Water	Glass	$\leq 6^oC$	7/40 Days
Organic Matter	ASA 29-3.5.2	Solid	Plastic/Glass	None; samples air-dried and processed prior to analysis	N/A
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Oxygenates on Product (GCMS SIM)	1625 modified	Product	10mL glass vial	$\leq 6^oC$	14 Days (7 Days from extraction)
PBDEs	1614	Water	1L Amber Glass	$\leq 6^oC$	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	$\leq 6^oC$	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	$\leq -10^oC$	1 Year/1 Year
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	Pest: 7/40 Days; PCB: 1 Year/1 Year
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3; pH<2 HCl; $\leq 6^oC$	14/30 Days
PCBs, total as Decachlorobiphenyl	508A	Water	1L Glass, TFE lined cap	$\leq 6^oC$	14/30 Days
Perchlorate	331	Water	Plastic/Glass	$\geq$0-6^oC, field filtered with headspace	28 Days
Permanent Gases (O2, N2, CO2)	RSK-175; PM01/AM20GAx	Water	40mL vials	benzalkonium chloride and $\leq 6^oC$	14 Days
Permanent Gases by Bubble Strip	SM9/AM20GAx	Water	20cc vapor vial with stopper septum	None	14 Days
Permanent Gases in Vapor	AM20GAx	Vapor	20cc vapor vial with flat septum	None	14 Days
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	$\leq$ -10°C	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	$\leq$ 6°C	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; $\leq$ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	$\leq$ 6°C; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	$\leq$ 6°C	1 Year/1 Year
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	$\leq$ -10°C	1 Year if frozen/1 Year
PCB Congeners	1668A	Water	1L Amber Glass	$\leq$ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	$\leq$ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	$\leq$ -10°C	1 Year/1 Year
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particle Size	ASA 15-5 modified	Solid	Plastic/Glass (100g sample)	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	28 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	5 Days
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	7 Days
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; $\leq$ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	$\leq$ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq$ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	$\leq$ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Polynuclear Aromatic Hydrocarbons (PAH)	TO-17	Air	Thermal desorption tubes via SKC Pocket Pumps or equivalent	$\leq 6^oC$ but above freezing	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	$\leq 6^oC$	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	$\leq 6^oC$; $Na_2S_2O_3$ if Cl present	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	$\leq -10^oC$	1 Year if frozen/40 Days
Purgeable Organic Halides (POX)	9021	Water	Glass; no headspace	$\leq 6^oC$	14 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid	Plastic/Glass		
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	$\leq 6^oC$	14/40 Days
Saturated Hydrocarbons		Water	$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	$\leq 6^oC$; pH<2 1:1 HCl (optional)
Silica, Dissolved	SM4500Si-D	Water	Plastic	$\leq 6^oC$	28 Days
Solids, Settleable	SM2540F	Water	Glass	$\leq 6^oC$	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	$\leq 6^oC$	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	$\leq 6^oC$	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	$\leq 6^oC$	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	$\leq 6^oC$	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	$\leq 6^oC$	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	$\leq 6^oC$	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	$\leq 6^oC$	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	$\leq 6^oC$	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/ 9038/375.2/ASTM D516	Water	Plastic/Glass	$\leq 6^oC$	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; $\leq 6^oC$	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid	Plastic/Glass	None	180 days
Total Inorganic Carbon (TIC)	PM01/AM20GAx	Water	40mL VOA vial with mylar septum	$\leq 6^oC$	14 Days
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; $\leq 6^oC$	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black/Lloyd Kahn	Solid	Glass	$\leq 6^oC$	14 Days
Total Organic Halogen (TOX)	SM5320/9020	Water	Glass; no headspace	$\leq 6^oC$	14 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Water	40mL vials	pH<2 HCl, no headspace, $\leq 6^oC$	7 Days
Total Petroleum Hydrocarbons (aliphatic and aromatic)	TPHCWG	Solid	Glass	$\leq 6^oC$	14 days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	$\leq 6^oC$	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
UCMR Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days
UCMR Hexavalent Chromium	218.7	Water	HDPE or propylene	$Na_2CO_3/NaHCO_3/(NH_4)_2SO_4$; pH>8	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
UCMR Chlorate	300.1	Water	Plastic or glass	EDA	28 Days
UCMR Perfluorinated Compounds	537	Water	Polypropylene	Trizma	14 Days
UCMR 1, 4 Dioxane	522	Water	Glass	Na_2SO_3, $NaHSO_4$; pH<4	28 Days
UV254	SM5910B	Water	Glass	$\leq 6^oC$	48 Hours
Vermiculite	EPA 600/R-93/116	Solid	Plastic/Glass	None (handling must be done in HEPA filtered fume hood; drying may be required)	N/A
Volatile Fatty Acids	AM21G	Water	40mL clear VOA vials	$\leq 6^oC$	21 Days
Volatile Fatty Acids (low level)	AM23G	Water	40mL clear VOA vials	$\leq 6^oC$ with benzalkonium chloride	14 Days
Volatiles	8260	Solid	5035 vial kit	See note 1 (analyze for acrolein and acrylonitrile per local requirements)	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	$\leq 6^oC$	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present (or unpreserved if run within 7 days of collection) (preserve and analyze for acrolein and acrylonitrile per local requirements)	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; $\leq 6^oC$; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4°C, **and** one vial methanol preserved at ≤6°C **and** one container of unpreserved sample stored at ≤6°C.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA Method Update Rule. Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate buffer to extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR program.

Attachment III – Shipping Exemption Regulation 49 CFR 173.4 "Small quantities for highway and rail"

(a) When transported domestically by highway or rail in conformance with this section, quantities of Division 2.2 (except aerosols with no subsidiary hazard), Class 3, Division 4.1, Division 4.2 (PG II and III), Division 4.3 (PG II and III), Division 5.1, Division 5.2, Division 6.1, Class 7, Class 8, and Class 9 materials are not subject to any other requirements when—

(1) The maximum quantity of material per inner receptacle or article is limited to—

(i) Thirty (30) mL (1 ounce) for authorized liquids, other than Division 6.1, Packing Group I, Hazard Zone A or B materials;

(ii) Thirty (30) g (1 ounce) for authorized solid materials;

(iii) One (1) g (0.04 ounce) for authorized materials meeting the definition of a Division 6.1, Packing Group I, Hazard Zone A or B material; and

(iv) An activity level not exceeding that specified in §§ 173.421, 173.424, 173.425 or 173.426, as appropriate, for a package containing a Class 7 (radioactive) material.

(v) Thirty (30) mL water capacity (1.8 cubic inches) for authorized Division 2.2 materials.

(2) With the exception of temperature sensing devices, each inner receptacle:

(i) Is not liquid-full at 55 °C (131 °F), and

(ii) Is constructed of plastic having a minimum thickness of no less than 0.2 mm (0.008 inch), or earthenware, glass, or metal;

(3) Each inner receptacle with a removable closure has its closure held securely in place with wire, tape, or other positive means;

(4) Unless equivalent cushioning and absorbent material surrounds the inside packaging, each inner receptacle is securely packed in an inside packaging with cushioning and absorbent material that:

(i) Will not react chemically with the material, and

(ii) Is capable of absorbing the entire contents (if a liquid) of the receptacle;

(5) The inside packaging is securely packed in a strong outer packaging;

(6) The completed package, as demonstrated by prototype testing, is capable of sustaining—

(i) Each of the following free drops made from a height of 1.8 m (5.9 feet) directly onto a solid unyielding surface without breakage or leakage from any inner receptacle and without a substantial reduction in the effectiveness of the package:

(A) One drop flat on bottom;

(B) One drop flat on top;

(C) One drop flat on the long side;

(D) One drop flat on the short side; and

(E) One drop on a corner at the junction of three intersecting edges; and

(ii) A compressive load as specified in § 178.606(c) of this subchapter.

*Note to paragraph (**a**)(6):Each of the tests in paragraph (a)(6) of this section may be performed on a different but identical package; i.e., all tests need not be performed on the same package.*

(7) Placement of the material in the package or packing different materials in the package does not result in a violation of § 173.21;

(8) The gross mass of the completed package does not exceed 29 kg (64 pounds);

(9) The package is not opened or otherwise altered until it is no longer in commerce; and

(10) The shipper certifies conformance with this section by marking the outside of the package with the statement "**This package conforms to 49 CFR 173.4 for domestic highway or rail transport only**."

(b) A package containing a Class 7 (radioactive) material also must conform to the requirements of § 173.421(a)(1) through (a)(5) or § 173.424(a) through (g), as appropriate.

(c) Packages which contain a Class 2 (other than those authorized in paragraph (a) of this section), Division 4.2 (PG I), or Division 4.3 (PG I) material conforming to paragraphs (a)(1) through (10) of this section may be offered for transportation or transported if approved by the Associate Administrator.

(d) Lithium batteries and cells are not eligible for the exceptions provided in this section.

Attachment IV – Shipping Exemption Regulation 49CFR 173.4a "Excepted quantities"

Title 49: Transportation
PART 173—SHIPPERS—GENERAL REQUIREMENTS FOR SHIPMENTS AND PACKAGINGS

II. § 173.4a Excepted quantities.
(a) Excepted quantities of materials other than articles transported in accordance with this section are not subject to any additional requirements of this subchapter except for:
(1) The shipper's responsibilities to properly class their material in accordance with §173.22 of this subchapter;
(2) Sections 171.15 and 171.16 of this subchapter pertaining to the reporting of incidents; and
(3) For a Class 7 (Radioactive) material the requirements for an excepted package.

(b) ***Authorized materials***. Only materials authorized for transport aboard passenger aircraft and appropriately classed within one of the following hazard classes or divisions may be transported in accordance with this section:
(1) Division 2.2 materials with no subsidiary hazard;
(2) Class 3 materials;
(3) Class 4 (PG II and III) materials except for self-reactive materials;
(4) Division 5.1 (PG II and III);
(5) Division 5.2 materials only when contained in a chemical kit or a first aid kit;
(6) Division 6.1, other than PG I, Hazard Zone A or B material;
(7) Class 7, Radioactive material in excepted packages
(8) Class 8 (PG II and III), except for UN2803 (Gallium) and UN2809 (Mercury); and
(9) Class 9, except for UN1845 (Carbon dioxide, solid or Dry ice), and lithium batteries and cells.

(c) ***Inner packaging limits***. The maximum quantity of hazardous materials in each inner packaging is limited to:
(1) 1 g (0.04 ounce) or 1mL (0.03 ounce) for solids or liquids of Division 6.1, Packing Group I or II or other materials that also meet the definition of a toxic material;
(2) 30 g (1 ounce) or 30mL (1 ounce) for solids or liquids other than those covered in paragraph (c)(1) of this section; and
(3) For gases a water capacity of 30mL (1.8 cubic inches) or less.

(d) ***Outer packaging aggregate quantity limits***. The maximum aggregate quantity of hazardous material contained in each outer packaging must not exceed the limits provided in the following paragraphs. For outer packagings containing more than one hazardous material, the aggregate quantity of hazardous material must not exceed the lowest permitted maximum aggregate quantity. The limits are as follows:
(1) For other than a Division 2.2 or Division 5.2 material:
(i) Packing Group I—300g (0.66 pounds) for solids or 300mL (0.08 gallons) for liquids;
(ii) Packing Group II—500g (1.1 pounds) for solids or 500mL (0.1 gallons) for liquids;
(iii) Packing Group III—1 kg (2.2 pounds) for solids or 1L (0.2 gallons) for liquids;
(2) For Division 2.2 material, 1L (61 cubic inches); or
(3) For Division 5.2 material, 500g (1.1 pounds) for solids or 250mL (0.05 gallons) for liquids.

(e) ***Packaging materials***. Packagings used for the transport of excepted quantities must meet the following:
(1) Each inner receptacle must be constructed of plastic, or of glass, porcelain, stoneware, earthenware or metal. When used for liquid hazardous materials, plastic inner packagings must have a thickness of not less than 0.2 mm (0.008 inch).

(2) Each inner packaging with a removable closure must have its closure held securely in place with wire, tape or other positive means. Each inner receptacle having a neck with molded screw threads must have a leak proof, threaded type cap. The closure must not react chemically with the material.
(3) Each inner packaging must be securely packed in an intermediate packaging with cushioning material in such a way that, under normal conditions of transport, it cannot break, be punctured or leak its contents. The intermediate packaging must completely contain the contents in case of breakage or leakage, regardless of package orientation. For liquid hazardous materials, the intermediate packaging must contain sufficient absorbent material that:
(i) Will absorb the entire contents of the inner packaging. In such cases, and
(ii) Will not react dangerously with the material or reduce the integrity or function of the packaging materials.
(iii) The absorbent material may be the cushioning material.
(4) The intermediate packaging must be securely packed in a strong, rigid outer packaging.
(5) Placement of the material in the package or packing different materials in the package must not result in a violation of §173.21.
(6) Each package must be of such a size that there is adequate space to apply all necessary markings.
(7) The package is not opened or otherwise altered until it is no longer in commerce.
(8) Overpacks may be used and may also contain packages of hazardous material or other materials not subject to the HMR subject to the requirements of §173.25.

(f) ***Package tests***. The completed package as prepared for transport, with inner packagings filled to not less than 95% of their capacity for solids or 98% for liquids, must be capable of withstanding, as demonstrated by testing which is appropriately documented, without breakage or leakage of any inner packaging and without significant reduction in effectiveness:
(1) Drops onto a solid unyielding surface from a height of 1.8 m (5.9 feet):
(i) Where the sample is in the shape of a box, it must be dropped in each of the following orientations:
(A) One drop flat on the bottom;
(B) One drop flat on the top;
(C) One drop flat on the longest side;
(D) One drop flat on the shortest side; and
(E) One drop on a corner at the junction of three intersecting edges.
(ii) Where the sample is in the shape of a drum, it must be dropped in each of the following orientations:
(A) One drop diagonally on the top chime, with the center of gravity directly above the point of impact;
(B) One drop diagonally on the base chime; and
(C) One drop flat on the side.

(2) A compressive load as specified in §178.606(c) of this subchapter. Each of the tests in this paragraph (f) of this section may be performed on a different but identical package; that is, all tests need not be performed on the same package.

(g) ***Marking***. Excepted quantities of hazardous materials packaged, marked, and otherwise offered and transported in accordance with this section must be durably and legibly marked with the following marking:

(1) The "*" must be replaced by the primary hazard class, or when assigned, the division of each of the hazardous materials contained in the package. The "**" must be replaced by the name of the shipper or consignee if not shown elsewhere on the package.
(2) The symbol shall be not less than 100 mm (3.9 inches) x 100 mm (3.9 inches), and must be durable and clearly visible.

(h) ***Documentation***.
(1) For transportation by highway or rail, no shipping paper is required.
(2) For transport by air, a shipping paper is not required, except that, if a document such as an air waybill accompanies a shipment, the document must include the statement "Dangerous Goods in Excepted Quantities" and indicate the number of packages.
(3) For transport by vessel, a shipping paper is required and must include the statement "Dangerous Goods in Excepted Quantities" and indicate the number of packages.

(i) ***Training***. Each person who offers or transports excepted quantities of hazardous materials must know about the requirements of this section.

(j) ***Restrictions***. Hazardous material packaged in accordance with this section may not be carried in checked or carry-on baggage.

ATTACHMENT C-6

THE DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD)
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD)
REFERENCE METHOD: EPA METHOD 410.4, REVISION 2.0

SOP NUMBER:	S-IN-I-012-rev.11
EFFECTIVE DATE:	August 28, 2017
SUPERSEDES:	S-IN-I-012-rev.10

APPROVAL

General Manager — August 17, 2017 Date

Quality Manager — August 17, 2017 Date

Department Manager — August 17, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining Chemical Oxygen Demand (COD) of aqueous samples while meeting the requirements specified in EPA Method 410.4, Revision 2.0.

2. Summary of Method

2.1. Samples, method blanks and standards are placed in sealed tubes and heated in a block digestor in the presence of dichromate at 150°C. After 2 hours, the tubes are removed, cooled, and measured spectrophotometrically at 420nm for low range samples or 600nm for high range samples.

2.2. Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the methods.

3. Scope and Application

3.1. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of COD analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to the measurement of COD in groundwater, surface waters and domestic and industrial wastewaters.

5. Limits of Detection and Quantitation

5.1. The default reporting limit is 10mg/L. Refer to the LIMS for method detection limit.

6. Interferences

6.1. Chlorides are quantitatively oxidized by dichromate and therefore represent a possible positive interference. Mercuric sulfate is added to the digestion tubes to complex with the chlorides.

6.2. Method interferences may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus. Method blanks are analyzed to check for these possible interferences.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	250mL in plastic or glass container	H_2SO_4 to pH<2	Cool to $\leq$6°C	Samples must be analyzed within 28 days of collection.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Description / Comments
COD Reactor	Capable of maintaining temp of 150°C
Spectrophotometer	Thermo AquaMate+ or equivalent, capable of reading 420nm and 600nm
Touch mixer	Vortex mixer or equivalent

9.2. General Supplies

Item	Description
Auto-Pipettes	Eppendorf or equivalent, various sizes
Volumetric flasks	Class A, various volumes

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Low range COD vials	Chemetrics/ catalog #K7355; 10-150mg/L, or equivalent COD vials
High range COD vials	Chemetrics/ catalog #K7365; 50-1500mg/L, or equivalent COD vials

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples. Table 9.3 describes the standards used.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains all target analytes and should be prepared from a different source than the calibration standards.	This solution is used for the LCS and MS.

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock High-Range COD Calibration Standard	Aqua Solutions COD Standard, catalog #TEN152; 10,000mg/L, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working High-Range COD Calibration Standards	Refer to Section 10.2.3.1	Must be prepared fresh daily.	Not applicable
Stock Low-Range COD Calibration Standard	Hach catalog #22539-29; 1000mg/L, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Low-Range COD Calibration Standards	Refer to Section 10.2.3.2	Must be prepared fresh daily.	Not applicable
Stock High-Range COD ICV/Spiking Standard	Environmental Express catalog #B1031; 10,000mg/L, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working High-Range COD ICV/LCS Standard	Refer to Section 10.2.3.3	Must be prepared fresh daily.	Not applicable
Stock Low-Range COD ICV/Spiking Standard	Ricca Spectro Pure catalog #SP069170500; 1000mg/L, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Low-Range COD ICV/LCS Standard	Refer to Section 10.2.3.4	Must be prepared fresh daily.	Not applicable

10.2.3. Standard Preparation Procedures

10.2.3.1. Working High-Range COD Calibration Standards

Prepare a series of standards covering the desired range by diluting appropriate volumes of the Stock High-Range COD Calibration Standard (10,000mg/L) in reagent water. Examples of calibration standards for high-range COD are as follows but may vary:

Standard ID	Amt. of Stock Calibration Std. Used	Final Volume	Final Concentration
Cal. Std. 1	0.125mL	25mL	50 mg/L
Cal. Std. 2	0.25mL	25mL	100 mg/L
Cal. Std. 3	0.30mL	10mL	300 mg/L
Cal. Std. 4 (CCV)	0.5mL	10mL	500 mg/L
Cal. Std. 5	1.0mL	10mL	1000 mg/L
Cal. Std. 6	1.5mL	10mL	1500 mg/L

10.2.3.2. Working Low-Range COD Calibration Standards

Prepare a series of standards covering the desired range by diluting appropriate volumes of the Stock Low-Range COD Calibration Standard (1000mg/L) in reagent water. Examples of calibration standards for low-range COD are as follows but may vary:

Standard ID	Amt. of Intermediate Calibration Std. Used	Final Volume	Final Concentration
Cal. Std. 1	0.25mL	25mL	10 mg/L
Cal. Std. 2	0.25mL	10mL	25 mg/L
Cal. Std. 3 (CCV)	0.50mL	10mL	50 mg/L
Cal. Std. 4	1.0mL	10mL	100 mg/L
Cal. Std. 5	1.5mL	10mL	150 mg/L

10.2.3.3. Working High-Range COD ICV/LCS Standard Preparation

Dilute 0.5mL of the Stock High-Range COD ICV/Spiking Standard (10,000mg/L) to 10mL with reagent water for a final concentration of 500mg/L.

10.2.3.4. Working Low-Range COD ICV/LCS Standard Preparation

Add 0.5mL of Stock Low-Range COD ICV/Spiking Standard (1000mg/L) to 10mL with reagent water for a final concentration of 50mg/L.

11. Calibration

11.1. **Initial Calibration:** A minimum of 5 calibration standards is required for each COD range. The lowest calibration standard for Low-Range COD must be at or below the reporting limit. New initial calibrations must be analyzed every 6 months at a minimum. Refer to the Quality Manual for more information regarding calibration curves.

11.2. **Linear Calibration:** For Low-Range analysis at 420nm, zero the spectrophotometer using reagent water. For High-Range analysis at 600nm, zero the spectrophotometer using an undigested reagent blank. Use the instrumentation software to prepare a standard curve by plotting absorbance versus concentration of each calibration blank and standard. The analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation.

11.3. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.995.

11.4. **Initial Calibration Corrective Action:** If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.5. **Initial Calibration Verification (ICV):** In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a

single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

11.6. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.7. Initial Calibration Blank (ICB): The ICB consists of reagent water for Low-Range analysis or an undigested reagent blank for High-Range analysis. An ICB must be analyzed after each ICV. If the ICB result is above the reporting limit, sample analysis cannot proceed. Samples associated with a failed ICB must be reanalyzed. Exception: If the ICB is >RL, associated samples determined to be <RL are reportable.

11.8. Continuing Calibration Verification (CCV): When an ICAL is not analyzed, the calibration must be verified by analyzing a CCV at the beginning of the analytical sequence. In all cases, a CCV must also be analyzed after every 10 samples and at the end of the analytical sequence to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.

11.9. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.10. Continuing Calibration Blank (CCB): The CCB consists of reagent water for Low-Range analysis or an undigested reagent blank for High-Range analysis. A CCB must be analyzed after each ICV or CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable.

12. Procedures

12.1. Turn on the COD reactor and heat to 150°C.

12.2. While holding a fresh COD vial at an angle, carefully pipette 2mL of blank, standard or sample into the vial. If any reagent is spilled, discard the tube and use another one. CAUTION: the reagents inside the COD tubes are hazardous – see Section 5.1. Avoid contact with skin. Waste vials must be disposed of properly – see Section 15.

12.3. Replace the cap and tighten securely. Mix on touch mixer for 10 seconds.

12.4. Prepare a method blank by pipetting 2mL of reagent water into a vial.

12.5. LCS Preparation:

12.5.1. Low-Range: Pipette 2mL of the Working Low-Range COD ICV/Spiking Standard into a low-range vials for an LCS concentration of 50mg/L.

12.5.2. High-Range: Pipette 2mL of the Working High-Range COD ICV/Spiking Standard into a high-range vial for an LCS concentration of 500mg/L.

12.6. MS/MSD Preparation:

12.6.1. Low-Range: Dilute 0.5mL of the Stock Low-Range COD ICV/Spiking Standard (1000mg/L) to 10mL with sample and mix well. Pipette 2mL of this spiked sample into a low-range vial for a spike concentration of 50mg/L.

12.6.2. High-Range: Dilute 0.5mL of the Stock High-Range COD ICV/Spiking Standard (10,000mg/L) to 10mL with sample and mix well. Pipette 2mL of this spiked sample into a high range vial for a spike concentration of 500mg/L.

12.7. Place the mixed sample vials in the preheated COD reactor. Check the vials after approximately 15 minutes. If any of the low-range COD vials has turned green, re-preparation of the sample in the high-range COD vial will be required. Heat the vials for a total of two hours at 150^{o}C. Turn off the reactor and allow the vials to cool for about 20 minutes to 120^{o}C.

12.8. While still warm, carefully invert the vial several times to mix the contents. Place the vials in a test tube rack to cool completely.

12.9. Turn on the spectrophotometer, adjust the wavelength to 420nm for Low-Range vials or to 600nm for High-Range vials and allow it to warm up. For Low-Range analysis at 420nm, zero the spectrophotometer using reagent water. For High-Range analysis at 600nm, zero the spectrophotometer using an undigested reagent blank.

12.10. Wipe all vials clean and read the absorbance of each. Record the absorbance once stabilized.

12.11. Compute sample concentration by comparing sample response with the standard curve. Multiply derived concentration by appropriate dilution factor.

12.12. Any sample that exceeds the concentration range of the Low-Range COD vials must be diluted by a maximum of 2x and reanalyzed using a Low-Range COD vial or reanalyzed using a High-Range COD vial. Any sample that exceeds the concentration range of the High-Range COD vials must be diluted and reanalyzed using a High-Range COD vial. Any sample having a concentration <50mg/L analyzed using the High-Range COD vials must be reanalyzed using the Low-Range COD vials.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples.	Target analyte must be less than the reporting limit.	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	90-110% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per batch plus an additional MS if >10 samples in the batch.	90-110% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (mg/L)} = \frac{(X_s)(V_f)}{(V_i)}$$

Where X_s = COD concentration from instrument in mg/L
V_f = Final volume of sample in Liters
V_i = Initial volume of sample in Liters

14.2. LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. Method Detection Limit (MDL) Study: An MDL study and/or LOD/LOQ verification must be conducted every 6 months for each COD range per instrument.

18.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Commercially-prepared COD vials pre-filled with digestion solution are used for consistency.

19.2. Method has been modified for manual spectrophotometric analysis of commercially-prepared COD vials for consistency and safety.

19.3. Method has been modified to include commercially-prepared low-range COD vials that are read at 420nm for improved accuracy at lower concentrations.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. After COD analysis is complete, return used COD vials to their original shipping container. When the original shipping container contains only used COD vials, move the shipping container to the Waste Disposal area for LabPack disposal.

23.3. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. EPA EMSL Method 410.4, Revision 2.0, 1993.

25.2. Standard Methods, 5220 Chemical Oxygen Demand (COD), 1997, editorial revisions 2011.

25.3. Pace Analytical Quality Manual; latest revision.

25.4. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-012-rev.09	1. Table of Contents: added new Section 14, Method Modifications 2. Section 3.2: reference to MDL added. 3. Table 9.3: updated standard information 4. Section 9.2.3: updated standard preparation information 5. Sections 11.5 and 11.7: updated standard references. 6. Section 11.8: added process for checking vials after 15 minutes in block digestor. 7. Section 11.10: added reference to Table 1 and Table 2. 8. Section 11.11: added procedure for handling over range samples. 9. Sections 11.12 and 11.13: removed previous tables and calculations and added reference to Table 1 and Table 2 for determining sample concentrations. 10. Table 12.1: updated Method Blank corrective action. 11. New Section 14, Method Modifications added 12. Section 15.1: updated SOP reference. 13. Section 17: added references to Table 1 and Table 2. 14. Table 1 and Table 2 added	06May2013
S-IN-I-012-rev.10	1. Cover: replaced Hach 8000 reference with 410.4. 2. Section 1: changed Hach 8000 reference to 410.4. 3. Section 2: changed 620nm wavelength to 600nm. 4. Table 8.1: updated spectrophotometer reference 5. Section 9: updated standard references and preparation 6. Section 10: updated calibration procedure for multi-point curves, removed CAL0, updated zeroing procedure for spectrophotometer and updated what is used for the ICB/CCB. 7. Section 11: updated procedure for spiking and for zeroing the spectrophotometer. 8. Section 15: added special disposal instructions for used COD vials. 9. Section 16: replaced Hach 8000 method reference with EPA 410.4 reference and added reference to SM5220. 10. Section 17: removed concentration tables.	10Aug2015
S-IN-I-012-rev.11	1. Converted to 27 section format. 2. Table 7.1: revised storage temperature format. 3. Table 10.3: updated details of ICV standard 4. Table 13.1: updated corrective actions for LCS and removed Duplicate. 5. Section 14.1: added calculation for final concentration. 6. Section 25.4: added years 2003 and 2009 to TNI reference.	06Aug2017

ATTACHMENT C-7

THE DETERMINATION OF AMMONIA NITROGEN
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF AMMONIA NITROGEN
REFERENCE METHODS: EPA METHOD 350.1 REVISION 2.0 AND STANDARD METHOD 4500-NH_3 G (1997/2011)

SOP NUMBER:	S-IN-I-043-rev.13
EFFECTIVE DATE:	July 5, 2017
SUPERSEDES:	S-IN-I-043-rev.12

APPROVAL

General Manager — June 21, 2017 Date

Quality Manager — June 20, 2017 Date

Department Manager — June 21, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining Ammonia Nitrogen in aqueous samples while meeting the requirements specified in EPA method 350.1, revision 2.0 and Standard Method 4500-NH_3 G, 1997, editorial revisions 2011.

2. Summary of Method

2.1. Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside and measured colorimetrically at 630nm.

3. Scope and Application

3.1. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ammonia nitrogen analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable for the measurement of ammonia nitrogen in potable water, ground water, surface and saline waters, domestic and industrial wastes and soils.

5. Limits of Detection and Quantitation

5.1. The default reporting limit for aqueous samples is 0.1mg/L (standard level) and 0.02mg/L (low level) and for soil samples is 5mg/kg. Refer to LIMS for method detection limits.

6. Interferences

6.1. Calcium and magnesium ions may precipitate if present in sufficient concentration. Tartrate or EDTA is added to the sample in-line in order to prevent this.

6.2. Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent at the pH of 9.5 at which distillation is carried out.

6.3. Residual chlorine must be removed by pretreatment of the sample prior to distillation/analysis.

6.4. Interferences due to turbidity may be filtered out of undistilled samples by means of 0.45 um filter prior to analysis.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	250mL in plastic or glass container	H_2SO_4 to pH<2	Cool to ≤6°C	Analysis must be completed within 28 days of collection.
Solid	50g in a glass jar	none	Cool to ≤6°C	Analysis must be completed within 28 days of collection.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Vendor	Description / Comments
Autoanalyzer	Lachat Quikchem 8500 or equivalent	Equipped with autosampler, heating unit, flow cell spectrophotometer for use at 630nm and data reduction system. Must use PVC pump tubing for this method.
Distillation apparatus	Lachat Micro-Dist Unit or equivalent	21 place micro-distillation unit, self contained, temperature controlled

9.2. General Supplies

Item	Description
Automatic-pipettors	Various sizes, Eppendorf or equivalent
Distillation tubes	Micro Dist Tubes, Lachat or equivalent
Distillation tube press	Wine bottle corker or equivalent to seal distillation tubes
Volumetric flasks	Class A, various sizes
Disposable Beakers	50mL or equivalent
Syringe filter	0.45um, Environmental Express or equivalent
Balance	Capable of weighing to 0.1g
Chlorine test strips	HF Scientific Micro Check low-range, or equivalent
pH test paper	Fisher narrow-range for pH 8-9.5, or equivalent
Autosampler tubes	Glass or plastic, for use with Lachat autosampler
Parafilm	Fisher or equivalent for capping stored distillates

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Simulated Soil Matrix	Ottawa sand or equivalent.
Phenol	Reagent grade crystals
Sodium Hydroxide	Reagent grade pellets
Sulfuric Acid	Concentrated, reagent grade
Sodium Phenolate solution	Dissolve 83g phenol crystals in 500mL of reagent water in small increments. Cautiously add, while stirring, 32g of sodium hydroxide pellets. Periodically cool flask under water faucet. When cooled completely, dilute to 1L with reagent water. Expires one week from date of preparation.
Sodium Hypochlorite solution	Dilute 250mLof commercial bleach solution (5.25%) to 500mL reagent water. Must be prepared fresh daily.
Disodium ethylenediamine tetraacetate (EDTA)	Reagent grade crystals
EDTA Buffer solution (5%)	Dissolve 50g of EDTA crystals and 5.5g sodium hydroxide pellets in 1L of reagent water. Expires one month from date of preparation.
Sodium Nitroprusside Dihydrate	Reagent grade crystals
Sodium Nitroprusside solution (0.35%)	Dissolve 3.5g of sodium nitroprusside crystals into 1L of reagent water. Expires two weeks from date of preparation.
1N Sodium Hydroxide solution	Dissolve 20g sodium hydroxide pellets in 500mL of reagent water.
Trapping solution (0.016M H_2SO_4)	Dilute 0.444mL concentrated sulfuric acid to 500mL with reagent water. Expires 6 months from date of preparation.
Borate Buffer	Ricca cat #1040-32 or equivalent Observe manufacturer's expiration date
Sodium Thiosulfate	Fisher cat #S445-500 or equivalent Observe manufacturer's expiration date
Sodium Thiosulfate 0.025N (Dechlorinating Reagent)	Dissolve 1.75g of Sodium Thiosulfate in 500mL of reagent water. Expires one week from date of preparation.
Antifoam	Silicon emulsion Fisher 02-002-333 or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This standard is used for spiking MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Ammonia Calibration Standard	Hach; catalog # 24065-49; 100mg/L Ammonia as N, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Ammonia Calibration Standards	Refer to Section 10.2.3.1	Must be prepared fresh daily.	Not Applicable
Stock Ammonia ICV Standard	SPEX; catalog # AS-NH3N9-2Y; 1000mg/L Ammonia as N, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Ammonia ICV/Spiking Standard	Refer to Section 10.2.3.2	Expires 1 month from date of preparation or on expiration date of stock standard, whichever comes first.	Same as stock standard.
Working Ammonia ICV Standard	Refer to Section 10.2.3.3	Must be prepared fresh daily.	Not Applicable

10.2.3. Preparation Procedures

10.2.3.1. Working Ammonia Calibration Standard Preparation

Working calibration standards must be prepared fresh daily by diluting the Stock Ammonia Calibration Standard (100mg/L) to 100mL with reagent water. Examples of possible calibration standards for the standard level analysis and low level analysis are as follows:

Standard Level (Water and Soil)

Standard ID	Amount of Stock Std.	Final Volume	Final Concentration
CAL0	0mL	100mL	0mg/L
CAL1	0.1ml	100mL	0.10mg/L
CAL2	0.25mL	100mL	0.25mg/L
CAL3	0.50mL	100mL	0.50mg/L
CAL4	1.0mL	100mL	1.0mg/L
CAL5	2.5mL	100mL	2.5mg/L
CAL6 (CCV)	5.0mL	100mL	5.0mg/L
CAL7	10mL	100mL	10mg/L

Low Level (Water only)

Standard ID	Amount of Stock Std.	Final Volume	Final Concentration
CAL0	0mL	100mL	0mg/L
CAL1	0.02ml	100mL	0.02mg/L
CAL2	0.05mL	100mL	0.05mg/L
CAL3	0.2mL	100mL	0.2mg/L
CAL4 (CCV)	0.8mL	100mL	0.8mg/L
CAL5	2.0mL	100mL	2.0mg/L

10.2.3.2. Intermediate Ammonia ICV/Spiking Standard Preparation

Dilute 5mL of the Stock Ammonia ICV Standard (1000mg/L) to 50mL with reagent water for a concentration of 100mg/L.

10.2.3.3. Working Ammonia ICV Standard Preparation

Standard Level ICV: Dilute 5mL of the Intermediate Ammonia ICV standard (100mg/L) to 100mL with reagent water for a concentration of 5mg/L. This standard must be prepared fresh daily.

Low Level ICV: Dilute 0.3mL of the Intermediate Ammonia ICV standard (100mg/L) to 100mL with reagent water for a concentration of 0.3mg/L. This standard must be prepared fresh daily.

11. Calibration

11.1. Initial Calibration: Initial calibration consists of a minimum of five standards and a calibration blank that are analyzed in decreasing order of concentration. The lowest calibration standard must be at or below the reporting limit. A new initial calibration curve is run on each working day. Refer to the Quality Manual for more information regarding calibration curves.

11.2. Linear Calibration: Using the Lachat software, prepare a standard curve by plotting peak area of standard versus the ammonia concentration. The analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

11.3. Back calculate the concentration of each calibration point. Acceptable recovery range for back-calculated calibration standards is 90-110%. Acceptable recovery for the lowest calibration standard is 50-150%.

11.4. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered.

11.5. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately after an initial calibration curve. The acceptable range for this standard is +/-10% Difference, which is equivalent to 90-110% Recovery.

$$\% \text{ Difference} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration})}{\text{Theoretical concentration}} \times 100$$

$$\% \text{ Recovery} = \frac{\text{Calculated concentration}}{\text{Theoretical concentration}} \times 100$$

11.6. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of

new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.7. Initial Calibration Blank (ICB): The ICB consists of reagent water. An ICB must be analyzed after each ICV. If the ICB result is above the reporting limit, sample analysis cannot proceed. Samples associated with a failed ICB must be reanalyzed. Exception: If the ICB is >RL, associated samples determined to be <RL are reportable.

11.8. Continuing Calibration Verification (CCV): A CCV must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable range for these standards is +/-10% Difference, which is equivalent to 90-110% Recovery.

11.9. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.10. Continuing Calibration Blank (CCB): The CCB consists of reagent water. A CCB must be analyzed after each CCV. If any CCB result is above the reporting limit, sample analysis must be stopped. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable.

12. Procedures

12.1. Sample Distillation (if applicable)

12.1.1. Water samples associated with an NPDES permit must be distilled prior to analysis, unless a comparative analysis is conducted for equivalency of results for undistilled samples, as described in 40CFR Part 136, Table IB, footnote 6. All soil samples must be distilled prior to analysis. Method Blank and LCS must also be distilled if associated samples are distilled.

12.1.2. Water Sample Distillation:

12.1.2.1. Sample preparation Step 1: Transfer approximately 50 mL aliquot of sample into a clean disposable beaker and check for residual chlorine using a Chlorine Test Strip. If chlorine is present, add 0.025N Sodium Thiosulfate dropwise until no chlorine is indicated by subsequent test strips.

12.1.2.2. Sample preparation Step 2: Using the same 50 mL aliquot of sample, add 1N NaOH dropwise to adjust pH to 9.5. Measure pH by means of narrow range pH paper or pH meter. Alternatively, sample may be placed on auto-titrator to adjust pH to 9.5 with 1N NaOH.

12.1.2.3. Set Distillation unit to 120° C. Allow heater to warm up to set temperature. This will take approximately 30 minutes.

12.1.2.4. Measure 6 mL of sample into a labeled sample tube.

12.1.2.5. Prepare a Method Blank by measuring 6 mL of reagent water into a labeled sample tube.

12.1.2.6. Prepare an LCS by diluting 0.10 mL of the Intermediate Ammonia ICV Standard (100mg/L)

to 6mL with reagent water for a concentration of 1.7 mg/L.

12.1.2.7. Prepare a Matrix Spike by diluting 0.10 mL of the Intermediate Ammonia ICV Standard (100mg/L) to 6mL with sample for a concentration of 1.7 mg/L.

12.1.2.8. Add 1.0 mL of Borate Buffer into each tube.

12.1.2.9. Dispense 1.0 mL of 0.016 M sulfuric acid trapping solution into 'M' end of distillation tube and seal the top with a membrane and distillation cap.

12.1.2.10. Immediately press the 'D' end of the distillation tube over the sample tube and place assembly into the press and seal the sample tube by applying downward pressure.

12.1.2.11. Put assembled distillation tube into the pre-heated hot block and set the timer for 30 minutes.

12.1.2.12. After 30 minutes, while wearing heat resistant gloves, immediately remove the sample tube from the distillation tube and roll tube to collect all sample condensate into the 'M' end of distillation tube.

12.1.2.13. Break the distillation tube at the constricted portion of the tube and fill to 6mL mark with reagent water. Analyze immediately or cap with parafilm until analysis can be performed.

12.1.3. Soil Sample Distillation:

12.1.3.1. Set Distillation unit to 120° C. Allow heater to warm up to set temperature. This will take approximately 30 minutes.

12.1.3.2. Add 0.5 g of sample into a labeled sample tube and add 5 mL of reagent water.

12.1.3.3. Prepare a Method Blank by adding 0.5g Ottawa Sand into a labeled sample tube and add 5 mL of reagent water.

12.1.3.4. Prepare an LCS by adding 0.1 mL of the Intermediate Ammonia ICV Standard (100mg/L) to a labeled sample tube with 0.5g Ottawa Sand and 5mL reagent water for a spike concentration of 20mg/Kg after distillation and volume adjustment to 10 mL.

12.1.3.5. Prepare a Matrix Spike by adding 0.1 mL of the Intermediate Ammonia ICV Standard (100mg/L) to a labeled sample tube with 0.5g sample and 5mL reagent water for a spike concentration of 20mg/Kg after distillation and volume adjustment to 10 mL.

12.1.3.6. Add 1.0 mL of Borate Buffer into each tube.

12.1.3.7. Dispense 1.0 mL of 0.016 M sulfuric acid trapping solution into 'M' end of distillation tube and seal the top with a membrane and distillation cap.

12.1.3.8. Immediately press the 'D' end of the distillation tube over the sample tube and place assembly into the press and seal the sample tube by applying downward pressure.

12.1.3.9. Put assembled distillation tube into the pre-heated hot block and set the timer for 30 minutes.

12.1.3.10. After 30 minutes, while wearing heat resistant gloves, immediately remove the sample tube from the distillation tube and roll tube to collect all sample condensate into the 'M' end of distillation tube.

12.1.3.11. Break the distillation tube at the constricted portion of the tube and fill to 10 mL mark with reagent water. Analyze immediately or cap with parafilm until analysis can be performed

12.2. Configure the instrument according to manufacturer's instructions. Allow the heating unit, colorimeter and recorder to warm up. Run a baseline with all reagents, using reagent water to flush the tubing. Whenever new tubing is used, allow ample time to flush residual compounds from the tubing.

12.3. Filter any samples that contain suspended solids using a 0.45um syringe filter. If any samples in a batch are filtered, the Method Blank and LCS must also be filtered.

12.4. Establish initial calibration as described in Sections 11.1 through 11.7.

12.5. Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration standards
ICV
ICB
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB

12.6. Sample concentrations exceeding the linear range must be diluted and reanalyzed, or result must be qualified as an estimated concentration. If sample was distilled, re-distillation at a dilution may be needed.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples.	Target analyte must be less than reporting limit.	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	90-110% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per batch plus an additional MS if >10 samples in the batch.	90-110% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

Undistilled Aqueous Sample (mg/L) = $(X_s)(D)$

Distilled Aqueous Sample (mg/L) = $\frac{(X_s)(V_f)(D)}{(V_i)}$

Solid Sample (mg/Kg) = $\frac{(X_s)(V_f)(D)}{(W_s)}$

Where: X_s = Ammonia concentration, mg/L
V_f = Final sample volume of distillate, L
D = Dilution factor
V_i = Initial sample volume distilled, L
W_s = Weight of solid sample distilled, Kg

Moisture corrected concentration = $\frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$

14.2. LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL study must be conducted every 6 months for each matrix per instrument.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Procedure modified for use of Lachat Micro-Dist system.

19.2. Samples which are distilled are distilled into a sulfuric acid solution per SM4500-NH3 B for use with the phenate method and not distilled into a boric acid solution.

19.3. Procedure modified for analysis of soils.

19.4. Calibration standards are not distilled.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

24. Pollution Prevention

24.1. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24.2. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. USEPA, Method 350.1, "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, Revision 2.0, August 1993.

25.2. Standard Methods for the Examination of Water and Wastewater; Method 4500-NH_3 G, 1997, editorial revisions 2011.

25.3. Lachat Method No. 10-107-06-1-K and J

25.4. Pace Analytical Quality Manual; latest revision.

25.5. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-043-rev.11	1. Cover page and header: added actual effective date. 2. Table of Contents: added Method Modifications 3. Section 3.1: added soils 4. Section 4: expanded interferences to include those in the method 5. Table 8.2: added a balance and KI test papers 6. Table 9.1: revised to include expiration dates and added sodium thiosulfate. 7. Section 10: added the requirement of five calibration standards for initial calibration and added the ICB. Removed regression equations. 8. Section 11.1.2: added dechlorination and pH adjustment steps and moved preparation instructions for MB, LCS and MS to this section from Section 12 9. Section 11.1.3: moved preparation instructions for MB, LCS and MS to this section from Section 12. 10. Section 11.5: added ICB to example run. 11. Section 11.6: added that over range samples can be qualified as estimated. 12. Table 12.1: revised corrective action for Method Blank. 13. Section 13.1: revised MDL frequency to every 6 months. 14. New Section 14, Method Modifications. 15. Section 16: added reference to Lachat method.	18Nov2013
S-IN-I-043-rev.12	1. Cover page: Added SM4500NH3G method reference and updated document control format. 2. Section 1.0: Added SM4500NH3G method reference. 3. Section 2: Added potable water matrix, low-level RL and reference to LIMS for MDLs. 4. Section 4.3: clarified that chlorine is removed prior to distillation/analysis. 5. Table 8.1: updated Lachat model number and added heating unit. Specific that PVC tubing must be used. 6. Table 8.2: updated chlorine test strips and added narrow-range pH paper. 7. Table 9.1: updated expiration of phenolate, buffer and nitroprusside solutions. 8. Table 9.3: updated expiration of intermediate ICV/Spiking standard. 9. Section 9.2.3: Added low level curve and ICV. 10. Section 10.1: Added calibration blank. 11. Section 10.2: Changed absorbance to peak height. 12. Section 14: added modification for use with Lachat Micro-Dist system and use of sulfuric acid solution instead of a boric acid solution for the distillation. 13. Section 16: Added SM4500NH3G and Lachat method references.	30Sep2015
S-IN-I-043-rev.13	1. Converted to 27-section format. 2. Cover page: added 2011 Standard Method reference. 3. Section 1.1: added 2011 Standard Method reference. 4. Table 7.1: revised storage conditions format. 5. Section 9.2: added tube press and parafilm. 6. Section 10.1: added conc. H2SO4, antifoam and sodium thiosulfate dry reagent. 7. Section 11: added requirement for back-calculation of curve points. 8. Section 12.6: added option to re-distill sample at a dilution. 9. Section 14.1: updated units so that equations are in like terms and added separate equations for distilled and undistilled aqueous samples. 10. Section 25: added 2011 Standard Method reference and added years 2003 and 2009 to TNI reference.	20Jun2017

ATTACHMENT C-8

THE DETERMINATION OF SULFIDE
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SULFIDE
COLORIMETRIC; METHYLENE BLUE METHOD
REFERENCE METHOD: STANDARD METHOD 4500-S^2-D (2000)

SOP NUMBER:	S-IN-I-076-rev.08
EFFECTIVE DATE:	July 5, 2017
SUPERSEDES:	S-IN-I-076-rev.07

APPROVAL

General Manager — June 21, 2017 Date

Quality Manager — June 20, 2017 Date

Department Manager — June 21, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining sulfide in aqueous samples while meeting the requirements specified in Standard Method 4500-S^2-D (2000).

2. Summary of Method

2.1. Sulfide reacts with dimethyl-p-phenylenediamine to produce methylene blue. The intensity of the blue color is proportional to the sulfide concentration. This compound is measured at a wavelength maximum of 665nm.

3. Scope and Application

3.1. Acid insoluble sulfides are not measured by this method. Copper sulfide is the only common sulfide in this class.

3.2. Reporting limits, control limits, volumes used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of sulfide analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable for the measurement of total and dissolved sulfides in ground water, surface water, and saline waters and domestic and industrial wastes

5. Limits of Detection and Quantitation

5.1. The default reporting limit is 0.1mg/L. Refer to the LIMS for the method detection limit.

6. Interferences

6.1. Samples should be taken with minimum aeration and container should be filled with minimal headspace. Sulfide can be volatilized by aeration or converted to a form that is not measurable.

6.2. Color or turbidity may interfere with photometric readings.

6.3. Strong reducing substances such as sulfite, thiosulfate, and hydrosulfite may reduce the blue color or prevent it from developing.

7. Sample Collection, Preservation and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	250mL in plastic container. Fill container completely without overflowing.	pH>9 with 1mL of 1:1 NaOH plus 0.5mL of 1N zinc acetate per 250mL sample.	Cool to ≤6°C	**Preserved**: Analysis must be completed within 7 days of collection. **Unpreserved**: Analysis must be completed within 24 hours of collection.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Model / Version	Description / Comments
Spectrophotometer	Spectronic 20D+ or equivalent	Capable of measuring at 665nm with a light path of 1cm

9.2. General Supplies

Item	Vendor	Description
Test tubes	Fisher or equivalent	Borosilicate glass, 16x100mm
Beakers	Fisher or equivalent	Disposable, 10mL
Erlenmeyer flasks	Fisher or equivalent	500mL
Mechanical Pipettors	Eppendorf or equivalent	Various capacities
Syringe filters	Environmental Express or equivalent	0.45um

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Sulfide 1 Reagent	Hach catalog #1816-49
Sulfide 2 Reagent	Hach catalog #1817-49

10.2. Analytical Standards

10.2.1. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Sulfide Calibration Standard	Absolute catalog #54139, 1000mg/L in single-use ampules, or equivalent	Manufacturer's recommended expiration date. After opening ampule, remainder can be stored in a 20mL vial for up to one week.	Refrigerate
Intermediate Sulfide Calibration Standards	Refer to Section 10.2.2.1	Must be prepared fresh daily, immediately prior to use.	Not applicable
Working Sulfide Calibration Standards	Refer to Section 10.2.2.2	Must be prepared fresh daily, immediately prior to use.	Not applicable
Stock Sulfide ICV Standard	Aqua Solutions catalog #8975, 1000mg/L or equivalent	Manufacturer's recommended expiration date.	Refrigerate
Intermediate Sulfide ICV Standard	Refer to Section 10.2.2.3	Must be prepared fresh daily, immediately prior to use.	Not applicable
Working Sulfide ICV Standard	Refer to Section 10.2.2.4	Must be prepared fresh daily, immediately prior to use.	Not applicable

10.2.2. Standard Preparation

10.2.2.1. Intermediate Sulfide Calibration Standard Preparation

Bring Stock Sulfide Calibration Standard in ampule to room temperature prior to opening. Dilute 1.0mL of the Stock Sulfide Calibration Standard (1000mg/L) to 100mL with reagent water for a concentration of 10mg/L. This standard must be prepared fresh daily, immediately prior to use.

10.2.2.2. Working Sulfide Calibration Standard Preparation

Working calibration standards are prepared using the Intermediate Sulfide Calibration Standard (10mg/L) and must be prepared fresh daily in reagent water immediately prior to use. Examples of possible calibration standards are as follows:

Standard	Int. Std. Volume	Final Volume	Final Conc.
CAL1	0.05mL	5mL	0.1 mg/L
CAL2	0.125mL	5mL	0.25 mg/L
CAL3 (CCV)	0.25mL	5mL	0.5 mg/L
CAL4	0.375mL	5mL	0.75 mg/L
CAL5	0.5mL	5mL	1.0 mg/L

10.2.2.3. Intermediate Sulfide ICV Standard Preparation

Bring Stock Sulfide ICV Standard to room temperature prior to opening. Dilute 1.0mL of the Stock Sulfide ICV Standard (1000mg/L) to 100mL with reagent water for a concentration of 10mg/L. This standard must be prepared fresh daily, immediately prior to use.

10.2.2.4. Working Sulfide ICV Standard Preparation

Dilute 0.25mL of the Intermediate Sulfide ICV Standard (10mg/L) to 5mL with reagent water for a concentration of 0.5mg/L. This standard must be prepared fresh daily, immediately prior to use.

11. Calibration and Standardization

11.1. **Initial Calibration:** A minimum of 5 calibration standards is required. The lowest calibration standard must be at or below the reporting limit. A new initial calibration must be analyzed every 6 months at a minimum. Refer to the Quality Manual for more information regarding calibration curves.

11.2. **Linear Calibration:** After zeroing the spectrophotometer with a reagent blank, prepare a standard curve by plotting absorbance versus sulfide concentration of each calibration standard. The analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.995.

11.3. Back calculate the concentration of each calibration point. Acceptable recovery range for back-calculated calibration standards is 90-110%. Acceptable recovery for the lowest calibration standard is 50-150%.

11.4. **Initial Calibration Corrective Action:** If the calibration does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second calibration attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.5. **Initial Calibration Verification (ICV):** In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy. A single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

11.6. **ICV Corrective Action:** If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.7. **Initial Calibration Blank (ICB):** The ICB consists of reagent water. An ICB must be analyzed after each ICV. If the ICB result is above the reporting limit, sample analysis cannot proceed. Samples associated with a failed ICB must be reanalyzed. Exception: If the ICB is >RL, associated samples determined to be <RL are reportable

11.8. **Continuing Calibration Verification (CCV):** When an ICAL is not analyzed, the calibration must be verified by analyzing a CCV at the beginning of the analytical sequence. In all cases, a CCV must also be analyzed after every 10 samples and at the end of the analytical sequence to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.

11.9. **CCV Corrective Action:** If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed.

Exception: If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.10. Continuing Calibration Blank (CCB): A CCB consists of reagent water. A CCB must be analyzed after each CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable.

12. Procedures

12.1. Sample Pretreatment for Interferences

12.1.1. If necessary or required by program or client, eliminate interferences due to sulfite, thiosulfate, iodide and other soluble substances by allowing the ZnS precipitate to settle for 30 minutes. Mark the sample volume level on the sample container using a permanent marker. Decant as much supernatant as possible without loss of the precipitate. Refill the bottle to the mark with reagent water, shake to re-suspend the precipitate, and quickly withdraw a sample aliquot for analysis. **NOTE: This step is required for all Marathon samples.**

12.2. Sample Analysis

12.2.1. Transfer 5mL of **well mixed** sample into a labeled disposable beaker.

12.2.2. Prepare a Method Blank by transferring 5mL reagent water to a labeled disposable beaker.

12.2.3. Prepare an LCS by diluting 0.25mL of the Intermediate Sulfide ICV Standard (10mg/L) to 5mL with reagent water in a labeled disposable beaker for a spike concentration of 0.5mg/L.

12.2.4. Prepare a Matrix Spike by diluting 0.25mL of the Intermediate Sulfide ICV Standard (10mg/L) to 5mL with sample in a labeled disposable beaker for a spike concentration of 0.5mg/L.

12.2.5. Add 0.25mL of Hach Sulfide 1 reagent and 0.25mL of Hach Sulfide 2 reagent to each disposable beaker. Shake or mix gently. Wait five minutes for color development.

12.2.6. Pour sample into a test tube and wipe the test tube clean prior to determining absorbance.

12.2.7. Adjust the wavelength control of the spectrophotometer to 665nm. Zero the spectrophotometer using the reagent blank.

12.2.8. Measure the absorbance of the standards, blanks and samples. A typical run sequence may be as follows:

ICAL Standards
ICV
(If ICAL not run, CCV would replace the ICAL and the ICV in the sequence)
ICB/CCB
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB

12.2.9. If the sample has a significant amount of color, make a dilution or prepare a background correction sample by preparing a second 5mL aliquot of the sample at the same dilution, adding only Sulfide 1 reagent.

12.2.10. From the absorbance or corrected absorbance, determine the concentration of sulfide present using the calculation in Section 14.1. Any sample with a sulfide concentration that exceeds the linear range of the calibration curve must be diluted and reanalyzed, or over range results must be qualified as estimated.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples.	Target analyte must be less than reporting limits	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	90-110% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per batch plus an additional MS if >10 samples in the batch.	90-110% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

Aqueous Sample (mg/L) = $(X_s)(D)$

Where: X_s = Sample concentration
D = Dilution factor (Final sample volume/Initial sample volume)

14.2. LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL and/or LOD/LOQ verification study must be conducted every 12 months for each matrix per instrument.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Sample containers are purchased pre-preserved with 1mL 1:1 NaOH plus 0.5mL 1N Zinc Acetate per 250mL.

19.2. A spectrophotometric wavelength of 665 nm is used.

19.3. Certified standards are purchased and have a short shelf-life. Standard solutions are not standardized prior to use.

19.4. Hach Sulfide 1 and Sulfide 2 reagents are used for analysis.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. **The Hach Sulfide 2 reagent contains potassium dichromate. The final solution will contain hexavalent chromium at a high level. The excess solution should be disposed of properly.**

22.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Standard Methods for the Examination of Water and Wastewater; Sulfide Method 4500 S^{2-} D, 2000.

25.2. Hach Water Analysis Handbook; 4th edition; Method 8131; Methylene Blue method

25.3. Pace Analytical Quality Manual; latest revision.

25.4. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-076-rev.06	1. Section 3.2: added reference to MDL 2. Table 7.1: revised preservation to match purchased pre-preserved containers 3. Section 9.2.2.1: added reference to standardization procedure. 4. Section 11: added procedure for pre-treatment for interferences. 5. Section 11.2: revised reagent volume used from 0.4mL to 0.5mL. 6. Section 11.2.6: added requirement to qualify over range results and revised calculation for concentration from curve. 7. Table 12.1: revised corrective action for method blank contamination. 8. Inserted new Method Modifications section.	24Sep2012
S-IN-I-076-rev.07	1. Cover page: changed phone number, added year reference to method, changed effective date format and revised document control format. 2. Section 1.1: added method year to reference. 3. Section 8.1: updated instrument information. 4. Section 9.3: updated standard information to reflect purchased certified standards. 5. Section 9.2.2: updated standard preparation information. 6. Section 10: removed standardization procedure for standards due to purchasing certified standards with short shelf life. Added ICB. 7. Section 11.1: added sample volume determination procedure. 8. Section 11.2: changed sample volume to 5mL and changed volume of reagents added. 9. Section 11.3: removed D from calculation. 10. Table 12.1: updated MS/MSD frequency. 11. Section 14: added modification that standards are purchased as certified, short-life solutions and are not standardized. 12. Section 16.1: added method year to reference.	10Nov2015

S-IN-I-076-rev.08	1. Converted to 27-section format. 2. Section 6.1: added that sample container should be filled with minimum headspace. 3. Table 7.1: added that sample container should be filled with minimum headspace, revised storage temperature format. 4. Section 9.1: updated spectrophotometer details. 5. Section 9.2: added syringe filters. 6. Section 10.1: removed all reagents except reagent water, Sulfide 1 and Sulfide 2. 7. Table 10.3: added storage procedure for opened ampule of stock calibration standard. 8. Section 10.2.2: added detail to bring standards to room temperature prior to use. 9. Section 11: added requirement to back-calculate curve standards and acceptance criteria. 10. Table 13.1: revised LCS corrective action. 11. Section 14: updated equation to remove Vi and Vf. 12. Section 25.4: added years 2003 and 2009 to TNI reference.	03Jun2017

ATTACHMENT C-9

THE MEASUREMENT OF SOLIDS IN WASTEWATER AND WATER

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STANDARD OPERATING PROCEDURE

THE MEASUREMENT OF SOLIDS IN WASTEWATER AND WATER
REFERENCE METHODS: STANDARD METHODS 2540 B, C, D, E AND F (1997/2011)

SOP NUMBER:	S-IN-I-084-rev.06
EFFECTIVE DATE:	May 15, 2017
SUPERSEDES:	S-IN-I-084-rev.05

APPROVAL

General Manager — May 1, 2017 Date

Quality Manager — April 25, 2017 Date

Department Manager — April 21, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining Total Solids (TS), also known as Total Residue, Total Dissolved Solids (TDS), also known as Filterable Residue, Total Suspended Solids (TSS), also known as Non-Filterable Residue, Fixed and Volatile Solids Ignited at 550°C, and Settleable Solids, while meeting the requirements specified in Standard Methods 2540 B, C, D, E and F, 1997, editorial revisions 2011.

2. Summary of Method

2.1. For TS, a well-mixed sample is placed into a beaker and evaporated to dryness in an oven.

2.2. For TSS, a well-mixed sample is filtered through a glass fiber filter and the residue remaining on the filter is dried in an oven.

2.3. The filtrate from the TSS analysis can be used for the TDS analysis by evaporating to dryness in an oven.

2.4. For Fixed and Volatile Solids, the residue from Method B is ignited to constant weight at 550°C.

2.5. For Settleable Solids, a well mixed sample aliquot is poured into a graduated Imhoff cone and allowed to settle.

3. Scope and Application

3.1. This method is applicable for the measurement of TDS, TSS, TS, Settleable Solids and Volatile Solids.

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of solids analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to drinking water, surface and saline waters and domestic and industrial wastes.

5. Limits of Detection and Quantitation

5.1. The reporting limits for the various forms of solids are shown in the table below. Refer to LIMS for method detection limits.

Parameter	Reporting Limit	Method
Total Solids (TS)	10 mg/L	2540 B
Total Dissolved Solids (TDS)	10 mg/L	2540 C
Total Suspended Solids (TSS)	5 mg/L	2540 D
Total Volatile Solids (TVS)	10 mg/L	2540 E
Settleable Solids	0.1 mL/L/Hr	2540 F

6. Interferences

6.1. Water samples containing significant amounts of calcium, magnesium, chloride or sulfate may require prolonged drying and desiccating time and may require rapid weighing. Samples with high bicarbonate concentration may require prolonged drying time to convert the bicarbonate into carbonate.

6.2. Samples high in TDS, such as saline waters, may be subject to positive interference. The appropriate filtering apparatus and filters should be chosen to minimize this possible interference.

6.3. Floating oil and grease, if present, should be dispersed by mixing and included with the sample.

6.4. Negative errors in the volatiles solids may be produced by loss of volatile matter during drying. Determination of low concentrations of volatile solids in the presence of high fixed solids may be subject to considerable error.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous samples for TS, TDS, TS, or TVS	250mL minimum in plastic container	None required	Cool to ≤6°C	Samples must be analyzed within 7 days of collection date.
Aqueous samples for Settleable Solids	1000mL minimum in plastic container	None required	Cool to ≤6°C	Settleable Solids must be determined within 48 hours of collection.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Constant Weight – the process of repeated cycles of drying/igniting, cooling, desiccating and weighing until the weight change is less than 4% of the previous weight or <0.5mg, whichever is less.

8.2. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment

Equipment	Vendor	Description / Comments
Drying Oven	Precision Scientific or equivalent	For TDS, capable of holding temperature at 180°C +/-2°C. For TSS and TS, capable of holding temperature at 103°C to 105°C.
Filter support	Fisher or equivalent	For use with 47mm filters
Suction Flask	Fisher or equivalent	Side-arm flask, 1L capacity or equivalent
Analytical Balance	Mettler, OHaus or equivalent	Capable of weighing to 0.1mg.
Desiccator	Fisher or equivalent	
Vacuum pump		
Muffle furnace		Capable of maintaining 550°C

9.2. General Supplies

Item	Vendor	Description
Filter discs	Whatman 934-AH, Environmental Express F93447MM or equivalent	47mm glass-fiber
Graduated cylinder	Fisher or equivalent	Class A, 100mL capacity
Glass beakers	Fisher or equivalent	100mL capacity
Volumetric flask	Fisher or equivalent	Class A, 1000mL for standard preparation
Tongs or forceps	Fisher or equivalent	For handling of glass fiber filers
Crucibles/Dishes		Porcelain for determining volatile solids, various sizes
Imhoff cones		With stand for determining settleable solids

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for preparing the LCS, as applicable.

Table 10.2 Standard Definitions and Vendors

Standard	Description
Spiking Standard (LCS)	This solution contains all target analytes.

10.2.2. Storage Conditions

Table 10.3 – Analytical Standards and Storage Conditions

Standard Type	Description	Expiration	Storage
Stock TSS Reference Standard	Celite, Fisher catalog #C212-500 or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions.
Working TSS Reference Standard	Refer to Section 10.2.3.1	Standard expires 6 months from date of preparation.	Refrigerate
Stock TS/TDS Reference Standard	Fisher Sodium Chloride; catalog #S271-3 or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions.
Working TS/TDS Reference Standard	Refer to Section 10.2.3.2	Standard expires 6 months from date of preparation.	Ambient

10.2.3. Preparation Procedures

10.2.3.1. Working TSS Reference Standard Preparation

Place 100mg of the Stock TSS Reference Standard into a 1L volumetric flask and dilute to volume with reagent water for a final concentration of 100mg/L. Place prepared solution on a stir plate and stir for a minimum of 24 hours in order to saturate solids with water.

10.2.3.2. Working TS/TDS Reference Standard Preparation

Place 0.3000g of the Stock TS/TDS Reference Standard into a 1L volumetric flask and dilute to volume with reagent water for a final concentration of 300mg/L.

11. Calibration

11.1. Analytical balance must be checked each day of use with Class 1 weights. The balance should be vendor-calibrated and serviced annually, at a minimum.

12. Procedures

12.1. Total Solids (TS) – Method 2540B

12.1.1. Preparation of glass beakers or evaporating dishes: Heat the clean beakers to 103-105^{o}C for one hour. Cool in desiccator and weigh immediately before use. If volatile solids are to be measured, ignite clean evaporating dish at 550°C for 1 hour in a muffle furnace. Cool in desiccator and weigh immediately before use.

12.1.2. Prepare a Method Blank by transferring 100mL of reagent water into a pre-weighed beaker.

12.1.3. Prepare a Laboratory Control Sample (LCS) by quantitatively transferring 100mL of the Working TS Reference Standard into a pre-weighed beaker.

12.1.4. Allow samples to come to room temperature. Quantitatively transfer 100mL of well mixed sample to a pre-weighed glass beaker.

12.1.5. Evaporate the samples to dryness in a drying oven. The oven temperature may need to be lowered initially to prevent splattering. Sample volume may vary and should result in 2.5 – 200mg of residue.

12.1.6. Continue drying the evaporated sample for at least one hour at 103°-105^{o}C.

12.1.7. Cool the beaker completely in a desiccator and weigh. Repeat the drying and desiccating cycle until a constant weight is achieved.

12.2. Total Dissolved Solids (TDS) – Method 2540C

12.2.1. Preparation of glass beakers or evaporating dishes: Heat the clean beakers to 180^{o}C +/- 2^{o}C for one hour. Cool in desiccator and weigh immediately before use. . If volatile solids are to be measured, ignite clean evaporating dish at 550°C for 1 hour in a muffle furnace. Cool in desiccator and weigh immediately before use.

12.2.2. Preparation of glass fiber filter disc: Place the glass fiber filter on the filter apparatus. While a vacuum is applied, rinse the disc with three successive 20mL volumes of reagent water. Continue to apply the vacuum until all water has passed through. Discard the rinsate.

12.2.3. Prepare a Method Blank by transferring 100mL of reagent water to the filter using a graduated cylinder. Remove all traces of water by continuing to apply the vacuum after the sample has passed through it.

12.2.4. Prepare a Laboratory Control Sample (LCS) by quantitatively transferring 100mL of the Working TDS Reference Standard to the filter using a graduated cylinder. Remove all traces of water by continuing to apply the vacuum after the sample has passed through it.

12.2.5. Allow samples to come to room temperature. Quantitatively transfer 100mL of well mixed sample to the filter using a graduated cylinder. Remove all traces of water by continuing to apply the vacuum after the sample has passed through it. Sample volume may vary and should result in 2.5 – 200mg of residue.

12.2.6. With the vacuum still on, rinse the graduated cylinder, filter, and filter holder with three 10mL portions of reagent water, allowing complete drainage in between washings. Continue suction for about 3 minutes.

12.2.7. Transfer the filtrate to a weighed evaporating dish or beaker and evaporate to dryness in the oven at 180^{o}C.

12.2.8. Cool the beaker completely in a desiccator and weigh. Repeat the drying and desiccating cycle until a constant weight is achieved.

12.3. Total Suspended Solids (TSS) – Method 2540D

12.3.1. Preparation of glass fiber filter disc: Place the glass fiber filter on the filter apparatus. While a vacuum is applied, rinse the disc with three successive 20mL volumes of reagent water. Continue to apply the vacuum until all water has passed through. Remove the filter from the apparatus and dry in an oven at 103°-105^{o}C for one hour. Remove the filter from the oven and place in desiccator. Repeat the drying procedure until a constant weight is achieved. Weigh the filter immediately before use. Alternatively, commercially prepared pre-washed and pre-weighed filters may be used.

12.3.2. Assemble the filtering apparatus and begin vacuum suction. Wet the filter with a small amount of reagent water to seat it against the fritted support.

12.3.3. Prepare a Method Blank by transferring 100mL of reagent water to the filter using a graduated cylinder. Remove all traces of water by continuing to apply the vacuum after the sample has passed through it.

12.3.4. Prepare a Laboratory Control Sample (LCS) by quantitatively transferring 100mL of the Working TSS Reference Standard to the filter using a graduated cylinder. Remove all traces of water by continuing to apply the vacuum after the sample has passed through it

12.3.5. Allow samples to come to room temperature. Shake the sample vigorously and quantitatively transfer 100mL to the filter using a graduated cylinder. Remove all traces of water by continuing to apply the vacuum suction after the sample has passed through it. Sample volume may vary and should result in 2.5 – 200mg of residue.

12.3.6. With the suction still on, wash the graduated cylinder, filter, and filter holder with three 10mL portions of reagent water, allowing complete drainage in between washings. Continue suction for about 3 minutes.

12.3.7. Carefully remove the filter from the filter apparatus. Dry for a minimum of one hour at 103°-105°C.

12.3.8. Cool the filter completely in a desiccator and weigh. If sample is being prepared and reported in the same day, repeat the drying cycle until a constant weight is achieved. Constant weight is not required if sample is dried in the oven over night.

12.4. Fixed and Volatile Solids Ignited at 550°C – Method 2540E

12.4.1. Preheat muffle furnace to 550°C and ignite residue produced by Method 2540B to constant weight. Usually 15-20 minutes ignition is required for 200mg residue.

12.4.2. Let dish cool partially in air until most of the heat is dissipated. Transfer to a desiccator for final cooling in a dry atmosphere. Do not overload desiccator.

12.4.3. Weigh dish as soon as it has cooled to balance temperature. Repeat cycle of igniting, cooling, desiccating and weighing until a constant weight is obtained.

12.5. Settleable Solids – Method 2540F

12.5.1. Fill an Imhoff cone to the 1-L mark with a well-mixed sample.

12.5.2. Settle for 45 minutes, gently agitate sample near the sides of the cone with a rod or by spinning.

12.5.3. Settle 15 minutes longer.

12.5.4. Record volume of settleable solids in the cone as milliliters per liter.

12.5.5. If the settled matter contains pockets of liquid between large settled particles, estimate volume of these and subtract from volume of settle solids.

12.5.6. Where a separation of settleable and floating materials occurs, do not estimate the floating material as settleable matter.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples.	Target analyte must be less than reporting limits.	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	80-120% Recovery	Reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Sample Duplicate (DUP)	Sample	One sample duplicate for every 10 or fewer samples analyzed.	≤10% RPD	No corrective action necessary. Qualify data as appropriate. ***Exception:*** 1. Duplicate sample values <5x RL are not evaluated because values at or near the RL provide statistically insignificant RPD results.

14. Data Analysis and Calculations

14.1. Calculate **TS** as follows:

$$\text{Total residue (TS), mg/L} = \frac{(A - B) \times 1000}{C}$$

Where A = weight of sample + dish, in mg
B = weight of dish, in mg
C = mL of sample used

14.2. Calculate **TDS** as follows:

$$\text{Filterable residue (TDS), mg/L} = \frac{(A - B) \times 1000}{C}$$

Where A = weight of dried residue + dish, in mg
B = weight of dish, in mg
C = mL of sample used

14.3. Calculate **TSS** as follows:

$$\text{Non-Filterable residue (TSS), mg/L} = \frac{(A - B) \times 1000}{C}$$

Where A = weight of filter + residue, in mg
B = weight of filter, in mg
C = mL of sample used

14.4. Calculate **volatile solids** as follows:

$$\text{Volatile Solids, mg/L} = \frac{(A - B) \times 1000}{\text{Sample Volume, mL}}$$

$$\text{Fixed Solids, mg/L} = \frac{(B - C) \times 1000}{\text{Sample Volume, mL}}$$

Where A = weight of residue + dish before ignition, mg
B = weight of residue + dish or filter after ignition, mg
C = weight of dish or filter, mg

14.5. LCS equation

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.6. RPD calculations (for duplicates):

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Section 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Section 13.

18. Method Performance

18.1. Demonstration of Capability (DOC): Every analyst who performs these methods must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Samples are measure using Class A graduated cylinders, not pipets.

19.2. Samples are shaken, not stirred.

19.3. Constant weight is not determined on samples for TSS dried over night.

19.4. Lab uses ≤10% RPD to evaluate duplicates which is roughly equivalent to method recommendation of duplicate sample agreement within 5% of their average weight.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. Equipment: Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Pollution Prevention

23.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

24. Waste Management

24.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

24.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

25. References

25.1. Standard Methods for the Examination of Wastewater and Water; Methods 2540B, C, D, E and F 1997, editorial revisions 2011.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003, 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-084-rev.05	1. Cover: added method references for 2540E and F. 2. Section 1: added method references for 2540E and F. 3. Section 2: added reference to Settleable Solids and Volatile Solids. 4. Section 3: added reference and RLs for Settleable Solids and Volatile Solids 5. Section 6: definition for constant weight added. 6. Table 7.1: added separate detail for Settleable Solids. 7. Section 8: added materials needed for Settleable Solids and Volatile Solids 8. Section 9: updated reference standards used and updated minimum stirring time for TSS standard prep. 9. Sections 11.1, 11.2: added dish prep instructions if residue will be used to determine volatile solids. 10. Section 11.1.2: specified that volume of sample should yield 2.5-200mg residue. 11. Section 11.2.3, 11.3.3: specified that volume of sample should yield 2.5-200mg residue. 12. Section 11.2.4, 11.3.4: specified that suction should continue for 3 minutes. 13. Section 11.4, 11.5: added procedures for Volatile Solids and Settleable Solids. 14. Table 12.1: revised RPD requirement and duplicate frequency. 15. Section 16: updated references to include 2540E and F.	02Nov2015
S-IN-I-084-rev.06	1. Converted to Corporate 27-section format. 2. Cover page: added 2011 to method reference. 3. Section 1.1: added 2011 to method reference. 4. Table 7.1: updated storage temperature format. 5. Section 10: removed reference to standard for MS/MSD. 6. Sections 12.1, 12.2, 12.3: added instructions for preparation of method blank and LCS. Moved calculations to Section 14. 7. Table 13.1: changed RPD to ≤10%, which is roughly equivalent to +/-5% of the average weight as stated in the method. 8. Section 19: added modification for use of ≤10% RPD for duplicates. 9. Section 25: added 2011 to method reference and added 2003 and 2009 to TNI reference.	1May2017

ATTACHMENT C-10

THE DETERMINATION OF TOTAL ORGANIC CARBON (TOC) AND DISSOLVED ORGANIC CARBON (DOC)
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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF TOTAL ORGANIC CARBON (TOC) AND DISSOLVED ORGANIC CARBON (DOC)

REFERENCE METHOD: STANDARD METHOD 5310C (2011)

SOP NUMBER:	S-IN-I-169-rev.02
EFFECTIVE DATE:	May 11, 2018
SUPERSEDES:	S-IN-I-169-rev.01

APPROVAL

General Manager — May 9, 2018 Date

Quality Manager — May 9, 2018 Date

Department Manager — May 8, 2018 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining Total Organic Carbon (TOC) and Dissolved Organic Carbon (DOC) in aqueous samples while meeting the requirements specified in Standard Method 5310C.

2. Summary of Method

2.1. Organic carbon is oxidized to carbon dioxide by persulfate in the presence of heat or ultraviolet light. The carbon dioxide produced may be purged from the sample, dried and transferred with a carrier gas to a nondispersive infrared (NDIR) analyzer or be coulometrically titrated.

2.2. DOC is the fraction of TOC that passes through a 0.45 um pore diameter filter.

3. Scope and Application

3.1. Reporting limits, control limits, volumes used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of TOC and DOC analysis equipment and reagents.

4. Applicable Matrices

4.1. This method is applicable to the measurement of TOC or DOC in aqueous samples.

5. Limits of Detection and Quantitation

5.1. The laboratory reporting limit is 1 mg/L. Refer to the LIMS for method detection limit.

6. Interferences

6.1. Insufficient acidification of samples may result in incomplete release of carbon dioxide.

6.2. The intensity of the ultraviolet light reaching the sample may be reduced by highly turbid samples or with aging of the ultraviolet light source, resulting in sluggish or incomplete oxidation.

6.3. Large organic particles or very large or complex organic molecules may be oxidized slowly because persulfate oxidation is rate-limited.

6.4. Persulfate oxidation of organic molecules is slowed in samples containing significant concentrations of chloride.

6.5. With any organic carbon measurement, contamination during sample handling and treatment is a likely source of interference.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous TOC	250 mL amber glass bottle	pH<2 with H_2SO_4 or H_3PO_4	Cool to ≤6°C	Samples must be analyzed within 28 days of collection.
Aqueous DOC	250 mL amber glass bottle	Filtered 0.45um, pH<2 with H_2SO_4 or H_3PO_4	Cool to ≤6°C	Samples must be analyzed within 28 days of collection.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions

9. Equipment and Supplies

9.1. Equipment and Instrumentation

Equipment	Description / Comments
TOC Analyzer	Shimadzu TOC-Vwp, Teledyne Tekmar Phoenix 8000, or equivalent, including autosampler and data system.

9.2. General Supplies

Item	Description
Autopipettes	Various volume ranges, calibration checked
Volumetric flasks	Class A, various sizes
Graduated cylinders	Class A, various sizes
Vials	40mL glass with screw cap
Filtration equipment	0.45um pore diameter filters and filtration apparatus for Dissolved Organic Carbon analysis

10. Reagents and Standards

10.1. Reagents

Reagent	Description
Reagent water	ASTM Type II water
Phosphoric acid	Extra pure, 85% solution in water. Acros Organics 29570, or equivalent
Acidified Water	Place about 500mL reagent water into a 1L volumetric flask. Add 8mL Phosphoric acid and bring to 1L with reagent water. Store in amber glass at ambient temperature. Solution expires one month from date of preparation.
Phosphoric acid solution for Shimadzu TOC-Vwp	Place about 500mL reagent water into a 1L volumetric flask. Slowly add 200mL Phosphoric acid while stirring. Allow solution to cool and bring to 1L with reagent water. Store in amber glass at ambient temperature. Solution expires one month from date of preparation.
Phosphoric acid solution for Tekmar Phoenix 8000	Place about 500mL reagent water into a 1L volumetric flask. Slowly add 250mL Phosphoric acid while stirring. Allow solution to cool and bring to 1L with reagent water. Store in amber glass at ambient temperature. Solution expires one month from date of preparation.
Sodium Persulfate	Crystalline, reagent grade. Acros Organics 20202, or equivalent.

Reagent	Description
Sodium Persulfate solution for Shimadzu TOC-Vwp	Place about 500mL reagent water into a 1L volumetric flask. Add 120g Sodium Persulfate and 30mL Phosphoric acid and stir to mix well. Bring to 1L with reagent water. It is recommended that this solution be allowed to stand in a cool dark location for 24 hours before use. Store in amber glass at ambient temperature. Solution expires one month from date of preparation.
Sodium Persulfate solution for Tekmar Phoenix 8000	Place about 500mL reagent water into a 1L volumetric flask. Add 117g Sodium Persulfate and 42mL Phosphoric acid and stir to mix well. Bring to 1L with reagent water. Allow this solution to equilibrate for 12 hours before use. Store in amber glass at ambient temperature. Solution expires one week from date of preparation.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analyte and is used to spike MS/MSD sets.	Same solution can be used for both the LCS and MS/MSD.

10.2.2. Storage Conditions

Table 10.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock TOC Calibration Standard	Ricca: catalog #1847; 1000mg/L potassium hydrogen phthalate, or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working TOC Calibration Standards	Refer to Section 10.2.3.1	Expires one month from preparation.	Same as stock standard.
Stock TOC ICV Standard	AlfaAesar; catalog #42562; 1000mg/L potassium hydrogen phthalate, or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working TOC ICV Standard	Refer to Section 10.2.3.2	Prepare fresh daily.	Same as stock standard.
Stock Inorganic Carbon Standard	Ricca; catalog #1845-16; 1000mg/L, or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions

10.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for additional instructions regarding preparation of standards for TOC analysis.

10.2.3.1. Working TOC Calibration Standard Preparation

Working calibration standards must be prepared fresh each day in reagent water. Prepare standards in 100mL volumetric flasks and add 2-3 drops of 85% Phosphoric acid to each flask prior to mixing. Pour standards into labeled 40mL vials for analysis. Examples of possible calibration standards are as follows but may vary:

Standard	Stock TOC Cal. Std. Volume	Final Volume	TOC Final Conc.
Cal Std 0	0 mL	100mL	0 mg/L
Cal Std 1	0.1 mL	100mL	1 mg/L
Cal Std 2	0.5 mL	100mL	5 mg/L
Cal Std 3 (CCV)	1.0 mL	100mL	10 mg/L
Cal Std 4	1.5 mL	100mL	15 mg/L
Cal Std 5	2.0 mL	100mL	20 mg/L

10.2.3.2. Working TOC ICV Standard Preparation

Dilute 1.0 mL of the Stock TOC ICV Standard (1000mg/L) and 2-3 drops of 85% Phosphoric acid to 100mL with reagent water for a final spike concentration of 10 mg/L.

11. Calibration

11.1. **Initial Calibration:** Initial calibration standards are analyzed in increasing order of concentration. The lowest calibration standard must be at or below the reporting limit. A new initial calibration curve is analyzed every six months, at a minimum, or as needed. Refer to the Quality Manual for more information regarding calibration curves.

11.2. **Linear Calibration:** Using the instrument software, construct a standard curve by plotting instrument response versus TOC concentration. The regression calculation will generate a correlation coefficient that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$0.995.

11.3. **Initial Calibration Corrective Action**: If the calibration curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered.

11.4. **Initial Calibration Verification (ICV**): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy of the calibration, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately after an initial calibration curve. The acceptable range for the ICV is 90-110% Recovery.

11.5. **ICV Corrective Action:** If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be

reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.6. **Initial Calibration Blank (ICB):** The ICB consists of acidified reagent water. An ICB must be analyzed after each ICV. If the ICB result is >2x MDL, sample analysis cannot proceed. Samples associated with a failed ICB must be reanalyzed. Exception: If the ICB is >2x MDL, associated samples determined to be <RL are reportable.

11.7. **Continuing Calibration Verification (CCV):** When an ICAL is not analyzed, the calibration must be verified by analyzing a CCV at the beginning of the analytical sequence. In all cases, a CCV must also be analyzed after every 10 samples and at the end of the analytical sequence to verify the system is still calibrated. The CCV should be from the same material as the calibration standards. The acceptable recovery range for the CCV is 90-110%.

11.8. **CCV Corrective Action:** If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.9. **Continuing Calibration Blank (CCB):** A CCB consists of acidified reagent water. A CCB must be analyzed after each CCV. If the CCB result is >2x MDL, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >2x MDL, associated samples determined to be <RL are reportable.

12. Procedures

12.1. Sample Pretreatment

12.1.1. Allow samples to come to room temperature.

12.1.2. If dissolved organic carbon is to be determined and samples were not filtered in the field, filter sample, Method Blank and LCS through 0.45um filter, taking care to avoid contamination.

12.1.3. Pour well-mixed sample into a labeled 40mL vial.

12.1.4. If the sample is high in solids, invert to mix and allow solids to settle. Decant or pipet the supernatant into a labeled 40mL vial for analysis.

12.1.5. When sample dilution is needed, use acidifed water as the diluent. Dilution should be considered when sample is turbid, high in solids, or has an organic odor.

12.2. Sample Analysis

12.2.1. Set up and calibrate instrument per manufacturer's instructions.

12.2.2. Prepare a Method Blank by filling a labeled 40mL vial with acidified water.

12.2.3. Prepare an LCS by diluting 0.4 mL of the Stock TOC ICV Standard (1000mg/L) to 40mL with acidified water for a final spike concentration of 10 mg/L.

12.2.4. Prepare a Matrix Spike by diluting 0.4 mL of the Stock TOC ICV Standard (1000mg/L) to 40mL with sample for a final spike concentration of 10 mg/L.

12.2.5. Prepare an Inorganic Carbon Check by diluting 0.4 mL of the Stock Inorganic Carbon Standard (1000mg/L) to 40mL with sample for a final spike concentration of 10 mg/L.

12.2.6. Analyze samples per instrument manufacturer's instructions. A typical analytical sequence may be as follows:

ICAL Standards
ICV (If ICAL not analyzed, CCV would replace the ICAL and ICV in the sequence)
ICB/CCB
Method Blank
LCS
Client samples
Inorganic Carbon Check
MS/MSD
CCV
CCB
Client samples
MS
CCV
CCB

12.2.7. Use instrument manufacturer's data system for the determination of sample concentrations. Calculate the final concentration in the sample as follows:

TOC or DOC (mg/L) = $(X_s)(D)$

Where: X_s = Concentration of the analyte in the sample from the curve in mg/L
D = Dilution factor (Final volume/Initial volume)

12.2.8. Replicate measurements should be reproducible to within ±10% RPD. Repeat analysis if replicate measurements are outside of the ±10% RPD criteria.

12.2.9. Samples that exceed the linear range must be reanalyzed at a dilution or over range concentrations must be qualified as estimated.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Acidified water	One per preparation batch of up to 20 samples.	Target analyte must be <2x MDL	Reanalyze method blank. If target compound is still >2x MDL in method blank, reanalyze all associated samples that are >RL. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	**TOC:** 90-110% Recovery **DOC:** 90-110% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per batch plus an additional MS if >10 samples in the batch.	80-120% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Inorganic Carbon Check	Inorganic Carbon spike	One Inorganic Carbon Check per analytical run.	Spiked result should equal the unspiked result within ≤20% RPD.	Instrument maintenance is required.

14. Data Analysis and Calculations

14.1. Use instrument manufacturer's data system for the determination of sample concentrations. Calculate the final concentration in the sample as follows:

$$\text{TOC or DOC (mg/L)} = (X_s)(D)$$

Where: X_s = Concentration of TOC or DOC in the sample from the curve in mg/L
D = Dilution factor (Final volume/Initial volume)

14.2. LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. MDLs must be conducted per EPA *Definition and Procedure for the Determination of the Method Detection Limit, Revision 2*; December 2016, 40 CFR Part 136 Appendix B, effective August 28, 2017.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Stock standards are purchased as certified solutions and are not prepared in the lab from dry chemicals.

19.2. Reagents are prepared per instrument manufacturer's instructions and may differ from those listed in the method.

19.3. Calibration check standard analyzed after every tenth analysis is not made from a source material other than the calibration standards.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling or other relevant SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Standard Methods for the Examination of Water and Wastewater, Method 5310C, 2000. Editorial revision 2011.

25.2. Shimadzu TOC-Vwp User's Manual, 2004.

25.3. Teledyne Tekmar Phoenix 8000 User's Manual, 14-7045-074 Rev. E, 2003.

25.4. Pace Analytical Quality Manual; latest revision.

25.5. NELAC/TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-169-rev.00	1. Converted to Pace SOP format. 2. Added reagent detail for Shimadzu TOC-Vwp instrument. 3. Removed 9060 reference.	23Sep2015
S-IN-I-169-rev.01	1. Converted to 27 section format. 2. Table 7.1: revised storage temperature format and removed holding time for unpreserved samples. 3. Section 9.2: added graduated cylinders. 4. Section 10.1: added Acidified Water and updated storage of other reagents. 5. Table 10.2: added Stock Inorganic Carbon standard. 6. Section 12.2: updated diluents to acidified water, updated preparation of LCS and MS and added preparation of Inorganic Carbon Check. 7. Table 13.1: updated corrective action for method blank and LCS and added Inorganic Carbon check. 8. Section 25.1: corrected method reference. 9. Section 25.5: added years 2003 and 2009 to TNI reference.	11Oct2017
S-IN-I-169-rev.01	1. Table 7.1: removed “preserved” from hold time language. 2. Section 11.6: updated ICB acceptance criteria. 3. Section 11.9: updated CCB acceptance criteria. 4. Section 12.1: added detail for sample pre-treatment when samples are high in solids or may contain interferences. 5. Table 13.1: updated components and acceptance criteria for Method Blank. 6. Section 18.1: updated reference for MDL procedure. 7. Section 19: added a modification for CCV analyzed after every tenth analysis, not LCS. 8. Section 25.5: added NELAC to reference.	8May2018

ATTACHMENT C-11

THE DETERMINATION OF TOTAL PHOSPHORUS
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF TOTAL PHOSPHORUS
REFERENCE METHOD: EPA METHOD 365.1, REVISION 2.0

SOP NUMBER:	S-IN-I-174-rev.01
EFFECTIVE DATE:	October 23, 2017
SUPERSEDES:	S-IN-I-174-rev.00

APPROVAL

General Manager — October 13, 2017 Date

Quality Manager — October 13, 2017 Date

Department Manager — October 13, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining total phosphorus in aqueous samples while meeting the requirements specified in EPA Method 365.1, Revision 2.0.

2. Summary of Method

2.1. Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This is reduced to an intensely blue-colored complex by adding ascorbic acid. The color is measured with an automated spectrometer and is proportional to the phosphorus concentration.

3. Scope and Application

3.1. Reporting limits, control limits, volumes used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of phosphorus analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable for the measurement of Total Phosphorus in groundwater, drinking, surface, and saline waters.

5. Limits of Detection and Quantitation

5.1. The reporting limit is 0.05mg/L. Refer to LIMS for method detection limits.

6. Interferences

6.1. Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1 mg As/L interfere with the Phosphorous determination.

6.2. Hexavalent chromium and nitrite interfere to give results about 3% low at concentrations of 1 mg/L and 10% to 15% low at 10 mg/L.

6.3. High iron and calcium concentration can cause precipitation of and therefore loss of phosphorus.

6.4. Sample color that absorbs in the photometric range used for analysis may also interfere.

6.5. Many commercially available detergents contain phosphorus and should never be used to clean glassware used for this analysis. Glassware must be rinsed with 1:1 HCl and reagent water prior to use for this method. Preferably dedicated glassware would be used for this method.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous samples for Total Phosphorus	125mL in plastic or glass container	H_2SO_4 to pH <2	Cool to ≤6°C	Analysis must be completed within 28 days of collection

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation/Equipment

Equipment	Model / ID	Description / Comments
Automated Spectrophotometer	SmartChem 200 Discrete Analyzer, or equivalent	Equipped with an autosampler and data system. Capable of measuring at 880nm or 650nm with a light path of 1cm or greater
Analytical Balance	OHaus AV412 or equivalent	Capable of weighing to the nearest 0.01g
Block Digester or Hot Plate	Fisher or equivalent	For sample digestion

9.2. General Supplies

Item	Description
Volumetric flasks	Class A, various sizes
Graduated cylinders	Class A, various sizes
Mechanical pipettors	Various sizes
Digestion Tubes	Glass 100mL capacity or equivalent
Erlenmeyer Flasks	125mL capacity
Boiling Chips	Chemware Ultra Pure PTFE chips or equivalent
Filters	Environmental Express syringe filters or equivalent
Sample tubes	Environmental Express 50mL screw top plastic tubes with lids or equivalent
Autosampler cups	Fisher 02-544-4 or equivalent, 4mL capacity

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Sulfuric acid	Concentrated, reagent grade or equivalent
Sulfuric acid solution (11N)	Aqua Solutions #ERL192, or equivalent.
Ammonium peroxydisulfate	Fisher #A682, or equivalent
Sulfuric acid solution (5N)	Aqua Solutions #9109-4LC, or equivalent
Antimony potassium tartrate	Fisher #A867, or equivalent
Antimony potassium tartrate solution	Dissolve 0.3g of antimony potassium tartrate in reagent water in a 100mL volumetric flask. Dilute to volume with reagent water and mix well. Store in a dark glass bottle and refrigerate when not in use. This solution expires 6 months from date of preparation.
Ammonium Molybdate	Fisher #A674, or equivalent
Ammonium Molybdate solution	Dissolve 4g of ammonium molybdate in reagent water in a 100mL volumetric flask. Dilute to volume with reagent water and mix well. Store in a plastic bottle and refrigerate when not in use. This solution expires 6 months from date of preparation.
Sodium Dodecyl Sulfate (SDS)	Acros Organics #23042, or equivalent
Sodium Dodecyl Sulfate Solution, 15%	Dissolve 15g of Sodium Dodecyl Sulfate in 85mL of reagent water. This solution may require gentle stirring and heat to fully dissolve. This solution expires 6 months from date of preparation.
Ascorbic acid	Fisher #A62, or equivalent
Ascorbic acid solution (Reagent 3)	Dissolve 0.88g of ascorbic acid in reagent water in a 50mL volumetric flask. Add 0.5mL 15% SDS solution and dilute to volume with reagent water. Mix gently to minimize foaming. This solution must be prepared fresh daily. Do not refrigerate.
Color Reagent (Reagent 2)	Mix together in order, 17.8mL of 5N sulfuric acid, 15mL of ammonium molybdate solution, 5mL of antimony potassium tartrate solution, 10mL of 15% sodium dodecyl sulfate solution and 52.2mL of reagent water. Mix solution after addition of each ingredient. Store the solution at room temperature and prepare fresh weekly. When this Color Reagent is prepared as described, acid digested samples can be analyzed without pH adjustment of digestate.
Cuvette Cleaning Solution Concentrate	Westco part number 3AS-RN00-20 or equivalent. Dilute 50mL of this concentrated solution to 1L with reagent water and invert five times to mix, for use as the Cuvette Wash Solution. Store at room temperature.
Probe Rinse Solution Concentrate	Westco part number 3AS-RN00-21 or equivalent. Dilute 0.5mL of this concentrated solution to 1L with reagent water and invert five times to mix, for use as the Probe Rinse Solution. Store at room temperature.
Diluent (Reagent 1)	Reagent water which has been digested per the procedure in Section 11.1.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains all target analytes and should be prepared from a different source than the calibration standards.	This solution is used for the LCS and MS.

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Phosphorous Calibration Standard	Ricca catalog # 5839-4; 326mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Phosphorous Calibration Standard	Refer to Section 10.2.3.1	Solution good for 6 months from date of preparation	Same as stock standard
Working Phosphorous Calibration Standard	Refer to Section 10.2.3.2 and 10.2.3.3	Must be prepared fresh each day of use	Not applicable
Stock Phosphorous ICV Standard	HACH catalog #2321142; 1000ppm or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Phosphorous ICV Standard	Refer to Section 10.2.3.4	Solution good for 6 months from date of preparation	Same as stock standard
Working Phosphorous ICV Standard	Refer to Section 10.2.3.5	Must be prepared fresh each day of use	Not applicable

10.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for additional instructions regarding preparation of standards.

10.2.3.1. Intermediate Phosphorous Calibration Standard Preparation

Dilute 15.33mL of the Stock Phosphorous Calibration Standard (326mg/L) to 100mL with reagent water for a final concentration of 50mg/L.

10.2.3.2. Working Phosphorous Manual Calibration Standard Preparation

Working calibration standards are prepared using the Intermediate Phosphorous Calibration Standard (50mg/L) and must be prepared fresh daily in diluent. Examples of possible calibration standards are as follows:

Standard ID	Amt. of Intermediate Calibration Std. Used	Final Volume	Final Concentration
Calibration Blank	0mL	50mL	0mg/L
Cal. Std. 1	0.05mL	50mL	0.05mg/L
Cal. Std. 2	0.1mL	50mL	0.10mg/L
Cal. Std. 3	0.25mL	50mL	0.25mg/L
Cal. Std. 4 (CCV)	0.50mL	50mL	0.50mg/L
Cal. Std. 5	0.75mL	50mL	0.75mg/L
Cal. Std. 6	1.0mL	50mL	1.0mg/L

10.2.3.3. Working Phosphorus Auto-dilution Calibration Standard Preparation

Dilute 0.2mL of the Intermediate Phosphorus Calibration Standard (50mg/L) to 10mL in diluent for a final concentration of 1.0mg/L. This standard must be prepared fresh daily and will be auto-diluted by the SmartChem autosampler to prepare the other calibration curve standards as detailed below:

Standard ID	Percentage of 1.0mg/L Calibration Std. Used	Final Concentration
Calibration Blank	0%	0mg/L
Cal. Std. 1	5%	0.05mg/L
Cal. Std. 2	10%	0.10mg/L
Cal. Std. 3	25%	0.25mg/L
Cal. Std. 4 (CCV)	50%	0.50mg/L
Cal. Std. 5	75%	0.75mg/L
Cal. Std. 6	100%	1.0mg/L

10.2.3.4. Intermediate Phosphorous ICV Standard Preparation

Dilute 5mL of the Stock Phosphorous ICV Standard (1000mg/L) to 100mL with reagent water for a final concentration of 50mg/L. This standard is also used for the LCS and MS/MSD spiking solution.

10.2.3.5. Working Phosphorous ICV Standard Preparation

Dilute 0.1mL of the Intermediate Phosphorous ICV Standard (50mg/L) to 10mL with diluent for a final concentration of 0.5mg/L.

11. Calibration

11.1. Initial Calibration: The instrument is calibrated each day that phosphorus analysis is performed. A minimum of 5 calibration standards and a calibration blank is required. The lowest calibration standard must be at or below the reporting limit. The instrument automatically dilutes a prepared standard to create the individual calibration points. Calibration points are analyzed in order of increasing concentration. Refer to the Quality Manual for more information regarding calibration curves.

11.2. Linear Calibration: The instrumentation software constructs a standard curve by plotting optical density versus concentration of each calibration standard. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

11.3. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.4. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

11.5. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.6. Initial Calibration Blank (ICB): The ICB consists of reagent water. An ICB must be analyzed after each ICV. If the ICB result is above the reporting limit, sample analysis cannot proceed. Samples associated with a failed ICB must be reanalyzed. Exception: If the ICB is >RL, associated samples determined to be <RL are reportable.

11.7. Continuing Calibration Verification (CCV): A CCV must be analyzed after every 10 samples and at the end of the analytical sequence to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.

11.8. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.9. Continuing Calibration Blank (CCB): A CCB consists of reagent water. A CCB must be analyzed after each ICV or CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable.

12. Procedures

12.1. Digestion of Aqueous Samples for Total Phosphorus

12.1.1. Place 50mL of well mixed aqueous sample into a labeled 125mL flask or block digester tube. A smaller volume may be used if sample is high in solids content or historically above the linear range of the curve.

12.1.2. Prepare a Method Blank by placing 50mL of reagent water into a labeled 125mL flask or block digester tube.

12.1.3. Prepare an LCS by placing 50mL of reagent water and 0.5mL of the Working Phosphorus ICV Standard (50mg/L) into a labeled 125mL flask or block digester tube for a spike concentration of 0.5mg/L.

12.1.4. Prepare a Matrix Spike by placing 50mL of sample and 0.5mL of the Working Phosphorus ICV Standard (50mg/L) into a labeled 125mL flask or block digester tube for a spike concentration of 0.5mg/L

12.1.5. Add 1mL of 11N sulfuric acid solution, 0.4g of ammonium peroxydisulfate and 3 or 4 boiling chips to each 125mL flask or block digester tube.

12.1.6. Boil gently on a pre-heated hot plate or in a block digester for approximately 90 minutes or until a final volume of about 10mL is reached. Do not allow sample to boil dry.

12.1.7. Allow samples to cool. Quantitatively transfer each sample to a 50mL sample tube and dilute to 50mL with reagent water.

12.1.8. If sample digestate is not clear, it may be filtered. Method Blank and LCS must also be filtered if any samples in the batch are filtered.

12.2. Digestion of Aqueous Samples for Acid Hydrolyzable Phosphorus

12.2.1. Place 50mL of well mixed aqueous sample into a labeled 125mL flask or block digester tube. A smaller volume may be used if sample is high in solids content or historically above the linear range of the curve.

12.2.2. Add 1mL of 11N sulfuric acid solution and 3 or 4 boiling chips to each 125mL flask or block digester tube.

12.2.3. Boil gently on a pre-heated hot plate or in a block digester for approximately 90 minutes or until a final volume of about 10mL is reached. Do not allow sample to boil dry.

12.2.4. Allow samples to cool. Quantitatively transfer each sample to a 50mL sample tube and dilute to 50mL with reagent water.

12.2.5. If sample digestate is not clear, it may be filtered.

12.3. Phosphorus Determination

12.3.1. Configure the instrument per manufacturer's instructions.

12.3.2. Fill disposable sample cups with samples and load them into the autosampler in the desired order. Fill clean reagent bottles with the appropriate reagents for this method as noted in Section 9.1.

12.3.3. Select the appropriate method in the software with the following parameters. The method as described here is equivalent to the EPA Method 365.1:

Type	End Point
Direction	Up
Decimals	3
Model	Linear
Filter 1	880 or 660 nm
Sample Blanking	No after Reagent 1
Calibration Code	OP1W

Method Code: WP1W **Range: 0.01 to 1.0 mg/L P**	**Volume uL**	**Delay Time sec.**	**Read Time sec.**	**Rinse uL**	**Code**
Sample Volume	290				
Reagent 1: Digested Blank Diluent	9	36	0	0	DIL1
Reagent 2: Color Reagent	65	0	0	0	MOL1
Reagent 3: Ascorbic Acid	28	0	342	0	ASC1

12.3.4. A typical run sequence may be as follows:

ICAL Standards
CCV
CCB
ICV
ICB
CCV
CCB
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB

12.3.5. Any sample with a concentration that exceeds the linear range of the calibration curve must be diluted and reanalyzed or qualified as an estimated concentration.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples.	Target analyte must be less than the reporting limit.	Reanalyze method blank. If target compound is still >RL in method blank and associated samples, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	90-110% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per batch plus an additional MS if >10 samples in the batch.	90-110% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (mg/L as P)} = \frac{(X)(V_f)(D)}{(V_i)}$$

Where: X = Phosphorus concentration, mg/L
V_f = Final sample volume, L
D = Dilution factor
V_i = Initial sample volume, L

14.2. Phosphate = Total Phosphorus x 3.064

14.3. Phosphonate Phosphorus = Total Phosphorus – Acid Hydrolyzable Phosphorus

14.4. Total Hydrolyzable Phosphorus = Acid Hydrolyzable Phos. – Ortho Phosphate

14.5. Total Organic Phosphorus = Total Phosphorus – (Acid Hydrolyzable Phos. + Orthophosphate)

(Orthophosphate determined separately)

14.6. LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.7. MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.8. RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL study and/or LOD/LOQ verification must be conducted every 6 months for each matrix per instrument.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

14.1 Method modified for use with a block digester or hot plate for the digestion step.

14.2 Method adapted for use with the SmartChem 200 per SmartChem Method 410-3651.

14.3 Method Blank is evaluated to the reporting limit, not the MDL as indicated in Method 365.1.

14.4 Sample pH adjustment prior to analysis is not performed because it is not required per the SmartChem Method.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. EPA EMSL Method 365.1, Revision 2.0, August 1993.

25.2. SmartChem 200 Method 410-3651, Rev. A-03-1206

25.3. Standard Methods for the Examination of Waste and Wastewater; method 4500-P B, E, Phosphorus, 1999 with editorial revisions 2011.

25.4. Pace Analytical Quality Manual; latest revision.

25.5. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-174-rev.00	1. Converted to Pace SOP format. 2. Section 11: added procedure for Acid Hydrolyzable Phosphorus and added calculation for multiple species of P.	23Sep2015
S-IN-I-174-rev.01	1. Converted to 27 section format. 2. Table 7.1: revised storage temperature format. 3. Section 9.1: revised balance specifications. 4. Section 9.2: updated digestion tubes specifications. 5. Table 10.3: updated Stock ICV. 6. Section 12.1.6: updated digestion time. 7. Section 12.2.3: updated digestion time. 8. Section 12.3.3: updated table for no sample blanking. 9. Section 12.3.4: updated example sequence. 10. Table 13.1: updated corrective action for MB and LCS. 11. Section 14.4: revised modification for clarity. 12. Section 25.5: added years 2003 and 2009 to TNI reference.	11Oct2017

ATTACHMENT C-12

THE DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP)
PACE, INDIANAPOLIS

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Indianapolis, IN 46268
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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP)
REFERENCE METHOD: EPA SW-846 METHOD 6010B

SOP NUMBER:	S-IN-M-019-rev.12
EFFECTIVE DATE:	January 8, 2018
SUPERSEDES:	S-IN-M-019-rev.11

APPROVAL

General Manager — January 3, 2018 Date

Quality Manager — December 28, 2017 Date

Department Manager — January 2, 2018 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of metals in aqueous and solid environmental samples while meeting the requirements specified in SW-846 method 6010B.

2. Summary of Method

2.1. Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2. This method describes multielement determinations by Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES). The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.

2.3. Background correction may be required to compensate for spectral interferences. Background is measured adjacent to analyte lines at a wavelength selected to be free of spectral interference and which reflects the same change in background intensity as occurs at the wavelength measured. Background correction is not required in cases of line broadening where a correction would actually degrade the analytical result.

3. Scope and Application

3.1. This method is applicable to the determination of most trace elements, including metals, in solution.

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ICP systems and interpretation of ICP data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to groundwater, surface water, wastewater, extract, leachate, soil, sediment, sludge and other solid samples.

5. Limits of Detection and Quantitation

5.1. Refer to Table 1 for the list of metals and reporting limits. Refer to the LIMS for method detection limits.

6. Interferences

6.1. **Spectral interferences:** Overlap of emission lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light can contribute to spectral interferences. These interferences can typically be minimized by careful selection of quantitation wavelengths, inter-element corrections, and background correction.

6.2. **Physical interferences:** Changes in sample viscosity, surface tension, or other effects associated with sample transport and nebulization can produce significant inaccuracies, especially in samples containing high concentrations of dissolved solids and acids. Dissolved solids may build up on the nebulizer tip, altering the sample flow rate and causing instrument drift. These effects can be minimized by sample

dilution or use of a specially designed high-solids nebulizer.

6.3. High Salt Concentrations: high salt concentrations in sample digestates can cause signal suppression and confuse interference tests.

6.4. Chemical interferences: Molecular compound formation, ionization effects, and solute vaporization effects are typically not significant with ICP determinations. If observed, they can be minimized by careful selection of plasma and spectrometer operating parameters.

6.5. Memory interferences: Sample deposition on the nebulizer tubing, spray chamber, and plasma torch can cause apparent sample carryover. Memory interferences can be minimized by flushing the system with rinse blanks between samples. If memory interference is suspected for a sample, the sample must be re-analyzed after a sufficient rinse period.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	250mL in plastic container	- HNO_3 to pH of <2 - Samples received at pH>2 must be preserved to pH<2 with HNO_3 and equilibrate for 24 hours before being prepared for analysis. Record date/time of preservation in preservation logbook.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Aqueous - Dissolved	250mL in plastic container	- Filter; HNO_3 to pH<2	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Solid	50 grams in glass or plastic container	- No chemical preservation	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
ICP-AES	Thermo-Fisher iCAP6500 or equivalent	Equipped with and autosampler and data system

9.2. General Supplies

Item	Vendor	Description
Volumetric Flasks	Class A	Various capacities
Volumetric Pipettors	Eppendorf or equivalent	Various sizes
Autosampler Vials	Environmental Express or equivalent	
Analytical Balance	Ohaus or eqivalent	Capable of weighing to 0.01g
Graduated Cylinders	Class A	Various capacities
pH strips	Fisher or equivalent	Full range

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Argon	High purity, liquefied
Nitric acid	Concentrated, trace metal analyzed or equivalent
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analytes and is used to spike MS/MSD sets.	Same solution can be used for the LCS and MS/MSD
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Yttrium
Interference Check Standards	Prepared to contain a known amount of interfering elements that will provide an accurate test of the interelement correction factors. If the ICP will display overcorrection as a negative number, the additional spiking with interfered elements is not necessary.	ICSA (ICSAB for BP Samples only)

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Calibration Standards	SPEX; catalog #'s MIXSTD1-100; MIXSTD2-100; MIXSTD3-100; MIXSTD4-100; MIXSTD5-100; PLS19-2Y; CLSN2-2Y; CLTI9-2Y; PLLI2-2Y; PLP9-3Y; CLAG2-2Y; PLSR2-2Y or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Calibration Standards	Refer to Section 10.2.3.2	Must be prepared fresh weekly	Same as stock standards
Stock ICV Standard	Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working ICV Standard	Refer to Section 10.2.3.4	Must be prepared fresh weekly	Same as stock standard
Working Second Source Spiking Solution	Refer to Section 10.2.3.5	Expires 6 months from date of preparation.	Same as stock standard

Standard Type	Description	Expiration	Storage
Stock Interference Check Standard A	SPEX; catalog # INT-A1 or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Interference Check Standard A (ICSA)	Refer to Section 10.2.3.6	Must be prepared fresh weekly	Same as stock standards
Stock Interference Check Standard AB	SPEX; catalog #INT-A1, XFSMN-26-250A (mix 1B), XFSMN-27-250A (mix 2B), or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Interference Check Standard AB (ICSAB)	Refer to Section 10.2.3.8	Must be prepared fresh weekly	Same as stock standards
Stock CRDL standards	SPEX individual standards for each element; 1000 or 10,000 mg/L, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate CRDL Standard	Refer to Section 10.2.3.11	Expires 6 months from date of preparation.	Same as stock standards
Working CRDL standard	Refer to Section 10.2.3.12	Must be prepared fresh weekly	Same as stock standards
Stock Internal Standard	SPEX; catalog # PLY2-2X; 1000mg/L yttrium or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Internal Standard	Refer to Section 10.2.3.13	Must be prepared fresh weekly	Same as stock standards

10.2.3. Standard Preparation Procedures

10.2.3.1. Stock Calibration Standard Details

The following table shows the seven stock standard mixes that may be used to prepare the initial calibration and calibration check standards:

Analyte	Concentration (mg/L)
Catalog # MIXSTD1-100	
Lead	500
Selenium	200
Cadmium	150
Zinc	150
Manganese	100
Beryllium	50
Catalog # MIXSTD2-100 + strontium PLSR2-2Y	
Iron	10,000
Barium	100
Cobalt	100
Copper	100
Vanadium	100
Strontium	100
Catalog # MIXSTD3-100 + silicon PLSI9-2Y	
Arsenic	500
Molybdenum	100
Silicon	100
Catalog #MIXSTD4-100	
Calcium	1000
Potassium	400

Catalog #MIXSTD4-100 Cont'd	
Aluminum	200
Sodium	200
Chromium	20
Nickel	20
Catalog #MIXSTD5-100	
Magnesium	1000
Antimony	200
Thallium	200
Boron	100
Silver	50
Mix #6(combines: CLSN2-2Y; CLTI9-2Y, PLLI2-2Y, PLP9-3Y)	
Lithium	1000
Phosphorus	10,000
Tin	1000
Titanium	1000
Mix #7- Catalog #CLAG2-2Y	
Silver	1000

10.2.3.2. Working Calibration Standards Preparation

Prepared fresh weekly and diluted from the stock standard mixes listed above, using a reagent water mixture that is 5% nitric acid and 2% hydrochloric acid unless otherwise noted.

Working Std. ID	Stock Standard	Vol. of Stock Std.	Final Volume
Calibration Std. Mix 1	MIXSTD1-100	2mL	100mL
Calibration Std. Mix 2	MIXSTD2-100 Strontium PLSR2-2Y	1mL 0.1mL	100mL
Calibration Std. Mix 3	MIXSTD3-100 Silicon PLSI9-2Y	2mL 0.8mL	100mL
Calibration Std. Mix 4	MIXSTD4-100	5mL	100mL
Calibration Std. Mix 5	MIXSTD5-100	2mL	100mL
Calibration Std. Mix 6	Lithium PLLI-2Y Phosphorus PLP9-3Y Tin CLSN2-2Y Titanium PLTI9-2Y	1mL 0.1mL 1mL 1mL	100mL
Calibration Std. Mix 7	Silver CLAG2-2Y	0.2mL	100mL in 10% HCl solution

10.2.3.3. Stock ICV Standard Details

The following table shows the concentrations of the stock standards purchased from Inorganic Ventures as three mixes:

Analyte	Concentration (mg/L)
Inorganic Ventures PA-STD1B / SPEX XFSMN-26-250A	
Arsenic	200/100
Barium	200/100
Beryllium	200/100
Cadmium	200/100
Cobalt	200/100
Chromium	200/100
Copper	200/100
Manganese	200/100

Inorganic Ventures PA-STD1B Cont'd	
Nickel	200/100
Phosphorus	200/100
Lead	200/100
Selenium	200/100
Thallium	200/100
Lithium	200/100
Strontium	200/100
Vanadium	200/100
Zinc	200/100
Inorganic Ventures PA-STD2B / SPEX XFSMN-27-250A	
Silicon	1000/500
Boron	200/100
Molybdenum	200/100
Antimony	200/100
Tin	200/100
Titanium	200/100
Zirconium	200/100
Silver	100/50
Inorganic Ventures PA-STD3B / SPEX XFSMN-28-250A	
Aluminum	2000/1000
Calcium	2000/1000
Iron	2000/1000
Potassium	2000/1000
Magnesium	2000/1000
Sodium	2000/1000

10.2.3.4. Working ICV Standard Preparation

Add 0.5mL of each Stock ICV Standard mix to a 100mL volumetric flask and dilute to volume with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid. If using SPEX stock standards, add 1.0 mL of each mix.

10.2.3.5. Working Second Source Spiking Solution

Add 25.0 mL of each Stock ICV Standard mix to a 100 mL volumetric flask and dilute to volume with reagent water solution that is 2% nitric acid.

10.2.3.6. Stock Interference Check Standard A (ICSA) Details

SPEX Interference Check Standard A (ICSA), mg/L	
Aluminum	5000
Calcium	5000
Magnesium	5000
Iron	2000

10.2.3.7. Working Interference Check Standard A (ICSA) Preparation

Dilute 10mL of the Stock ICSA Standard to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

10.2.3.8. Stock Interference Check Standard AB (ICSAB) Details

SPEX INT-A1	
Al, Ca, Mg	5000
Fe	2000
SPEX XFSMN-26-250A (Mix 1B)	
As, Ba, Be, B, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl, V, Zn, Li, P	100
SPEX XFSMN-27-250A (Mix 2B)	
Mo, Sb, Sn, Ti, B	100
Ag	50
Si	500

10.2.3.9. Working Interference Check Standard AB (ICSAB) Preparation

Dilute 10mL of the stock INT-A1 standard and 0.5mLof the stock Mix 1B and Mix 2B to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

10.2.3.10. Stock CRDL Standards Detail

When specified by client or program requirements, a low-level check standard, also known as a CRDL standard, must be analyzed prior to sample analysis and at the end of each analytical batch to bracket the client samples. Acceptance limits for all target elements is 50-150% recovery. The Stock CRDL standards are as follows:

Element	Conc. (ug/mL)	SPEX Catalog #	Element	Conc. (ug/mL)	SPEX Catalog #
Aluminum	1000	CLAL2-2Y	Manganese	1000	CLMN2-2Y
Antimony	1000	CLSB7-2Y	Molybdenum	1000	CLMO9-2Y
Arsenic	1000	CLAS2-2Y	Nickel	1000	CLNI2-2Y
Barium	1000	CLBA2-2Y	Phosphorus	10,000	PLP9-3Y
Beryllium	1000	CLBE2-2Y	Potassium	10,000	PLK2-3Y
Boron	1000	PLB9-2Y	Selenium	1000	CLSE2-2Y
Cadmium	1000	CLCD2-2Y	Silicon	1000	PLSI9-2Y
Calcium	10,000	PLCA2-3Y	Silver	1000	CLAG2-2Y
Chromium	1000	CLCR2-2Y	Sodium	10,000	PLNA2-3Y
Cobalt	1000	PLCO2-2Y	Strontium	1000	PLSR2-2Y
Copper	1000	CLCU2-2Y	Thallium	1000	CLTL2-2Y
Iron	10,000	PLFE2-3Y	Tin	1000	CLSN2-2Y
Lead	1000	CLPB2-2Y	Titanium	1000	CLTI9-2Y
Lithium	1000	PLLI2-2Y	Vanadium	1000	CLV2-2Y
Magnesium	10,000	PLMG2-3Y	Zinc	1000	CLZN2-2Y

10.2.3.11. Intermediate CRDL Standard Preparation

Dilute the following volumes of the stock CRDL standards to 50mL with a reagent water solution that is 2% nitric acid:

Element	Volume (mL)	Final Conc. (mg/L)	Element	Volume (mL)	Final Conc. (mg/L)
Aluminum	2.5	50	Manganese	0.125	2.5
Antimony	0.075	1.5	Molybdenum	0.125	2.5
Arsenic	0.125	2.5	Nickel	0.125	2.5
Barium	0.125	2.5	Phosphorus	1.25	250
Beryllium	0.05	1	Potassium	1.25	250
Boron	1.25	25	Selenium	0.125	2.5
Cadmium	0.025	0.5	Silicon	2.5	50
Calcium	1.25	250	Silver	0.125	2.5
Chromium	0.125	2.5	Sodium	1.25	250
Cobalt	0.125	2.5	Strontium	0.125	2.5
Copper	0.125	2.5	Thallium	0.125	2.5
Iron	0.125	25	Tin	0.125	2.5
Lead	0.125	2.5	Titanium	0.125	2.5
Lithium	0.25	5	Vanadium	0.125	2.5
Magnesium	1.25	250	Zinc	0.25	5

10.2.3.12. Working CRDL Standard Preparation

Dilute 1mL of the Intermediate CRDL Standard to 250mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid. Final concentrations are shown below.

Element	Final Conc. (ug/L)	Element	Final Conc. (ug/L)
Aluminum	200	Manganese	10
Antimony	6	Molybdenum	10
Arsenic	10	Nickel	10
Barium	10	Phosphorus	1000
Beryllium	4	Potassium	1000
Boron	100	Selenium	10
Cadmium	2	Silicon	200
Calcium	1000	Silver	10
Chromium	10	Sodium	1000
Cobalt	10	Strontium	10
Copper	10	Thallium	10
Iron	100	Tin	10
Lead	10	Titanium	10
Lithium	20	Vanadium	10
Magnesium	1000	Zinc	20

10.2.3.13. Working Internal Standard Preparation

Dilute 5mL of yttrium stock standard (1000mg/L) to 1L with a reagent water solution that is 2% nitric acid for a final concentration of 5mg/L.

11. Calibration

11.1. Initial Calibration: Calibrate the ICP each working day according to the instrument manufacturer's recommended procedures. Flush the system with the Calibration Blank solution prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. The calibration curve must consist of a minimum of a calibration blank and a standard.

11.2. Linear Calibration: Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The analyst may employ a regression equation that does not pass through the origin. If a multi-point calibration is performed, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

11.3. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.4. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110% and the RSD of replicate readings must be <5%.

11.5. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.6. Initial Calibration Blank (ICB): The ICB consists of a reagent water solution that is 5% HNO_3 and 2% HCl. An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, another ICB may be analyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable. **If required by client or program, the ICB must be evaluated as follows: If the ICB result exceeds ½ the RL, the ICB is considered to be unacceptable. Only samples determined to be <RL are reportable. If the absolute value of a negative concentration exceeds twice the established MDL, the ICB is considered to be unacceptable. Samples associated with a failed ICB must be re-analyzed unless the concentration of the target analyte is greater than 10 times the absolute value of the ICB result.**

11.7. Continuing Calibration Verification (CCV): A CCV must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The acceptable recovery range for the CCV is 90-110% and the RSD of replicate readings must be <5%.

11.8. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.9. Contract Required Detection Limit (CRDL) Standard: A CRDL standard must be analyzed with each analytical run, at a minimum, after calibration. Acceptance limits for all target elements is 50-150%

recovery. If required by client or program, another CRDL standard must be analyzed after samples – not to exceed 8 hours between CRDL analyses.

11.10. CRDL Corrective Action: Samples associated with a failed CRDL must be re-analyzed unless the concentration of the target has failed high, then the associated samples determined to be <RL are reportable.

11.11. Interference Check Standard A (ICSA): An ICSA must be analyzed at the beginning of each analytical run. ICSA must be 80-120% of the true value for the elements in the mix. Non-ICSA elements must be within +/-2x the reporting limit.

11.12. ICSA Corrective Action: If the ICSA fails the acceptance criteria, another ICSA may be analyzed. If the second ICSA fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSA must be reanalyzed. **Exception:** If the ICSA is >120% for any element in the mix or if any non-ICSA element is >2x the reporting limit, indicating high bias, associated samples determined to be <RL are reportable.

11.13. Interference Check Standard AB (ICSAB): If required by client or program an ICSAB must be analyzed at the beginning of each analytical run. ICSAB must be 80-120% of the true value for the elements in the mix.

11.14. ICSAB Corrective Action: If an ICSAB is required by client or program and the ICSAB fails the acceptance criteria, another ICSAB may be analyzed. If the second ICSAB fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSAB must be reanalyzed. **Exception:** If the ICSAB is >120% for any element in the mix, indicating high bias, associated samples determined to be <RL are reportable.

11.15. Continuing Calibration Blank (CCB): The CCB consists of a reagent water solution that is 5% HNO_3 and 2% HCl. A CCB must be analyzed after every 10 samples following the CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable. **If required by client or program, the CCB must be evaluated as follows: If the CCB result exceeds ½ the RL, the CCB is considered to be unacceptable. Only samples determined to be <RL are reportable. If the absolute value of a negative concentration exceeds twice the established MDL, the CCB is considered to be unacceptable. Samples associated with a failed CCB must be re-analyzed unless the concentration of the target analyte is greater than 10 times the absolute value of the CCB result.**

12. Procedure

12.1. Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data document the selection criteria of background correction points; analytical dynamic ranges; the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis.

12.2. Configure the ICP per manufacturer's instructions and allow it to become thermally stable.

12.3. Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.

12.4. Establish initial calibration as described in Section 11.

12.5. Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration blank
Mix 1
Mix 2
Mix 3
Mix 4
Mix 5
Mix 6
Mix 7
ICV
ICB
CRDL
ICSA
ICSAB (if required)
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB
CRDL (if required)
ICSA (if required)
ICSAB (if required)

12.6. The instrument performs two replicate readings for each analysis and the average of the two readings is used to derive the concentration. For samples, the difference between the two readings must be $\leq$20% RSD for values that are >4x the reporting limit. If the RSD is >20% for values that are >4x the reporting limit, the sample must be reanalyzed.

12.7. Samples with analyte concentrations above the upper linear range must be diluted and reanalyzed or the over range results must be qualified as estimated.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or boiling chips	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be less than reporting limits	Reanalyze method blank. Re-digest and reanalyze if target compound is still >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. 3) If sample concentration is >10x blank level, sample and method blank may be reported, but sample must be qualified. (Not for VAP)
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified. 3) If the batch's associated MS or MSD recovery falls within LCS acceptance limits, associated samples may be reported. The LCS data must be qualified. (Not applicable to OH VAP projects)
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Internal Standard	Yttrium	Automatically added to each sample, blank, and standard as part of the analysis.	No acceptance criteria – used to monitor interferences. 60-140%	No corrective action required. Sample may be analyzed at a dilution if interference is indicated.

14. Data Analysis and Calculations

14.1. Calculate sample concentrations using the following equation:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_f)(D)}{(V_i)} \qquad \text{Solid Sample (ug/kg)} = \frac{(X_s)(V_f)(D)}{(W_i)}$$

Where: X_s = Element concentration, ug/L
V_f = Final volume of digestate, L
D = Dilution factor
V_i = Initial volume of aqueous sample digested, L
W_i = Initial weight of solid sample digested, kg

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

14.2. LCS equation:

$R = (C/S) * 100$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. **Method Detection Limit (MDL) Study**: An MDL study must be conducted every 12 months for each matrix per instrument.

18.2. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

18.3. **Linear Dynamic Range Study**: A linear dynamic range study must be conducted for each element by analyzing increasing concentrations of at least three, preferably five different concentration standards across the range. One of these should be near the upper limit of the range. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Samples determined to be above the upper range limit must be diluted and reanalyzed. New dynamic ranges should be determined whenever there is a significant change in instrument response. For those analytes that periodically approach the upper limit, the range should be checked every six months. Refer to Section 7.2.5.4 of Method 6010B for more information.

18.4. **Interelement Correction Factors** must be verified and updated every 6 months or when an instrumentation change occurs. Refer to Section 3.1 of Method 6010B for more information.

18.5. **Post-Digestion Spike Addition:** An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

18.6. **Dilution test:** If the analyte concentration is sufficiently high, minimally, a factor of 10 above the instrument detection limit after dilution, an analysis of a 1:5 dilution should agree within +/-10% of the original determination. If not, a chemical or physical interference effect should be suspected.

19. Method Modifications

19.1. Mixed standard solutions are purchased as certified standards.

19.2. Instrument conditions may vary from those stated in the method.

19.3. Calibration blanks are evaluated to the reporting limit and not to three times the IDL.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and must be handled with extreme

care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

22.2. **Samples**: Take precautions when handling samples. Samples must always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment**: Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

24. Pollution Prevention

24.1. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24.2. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Method 6010B.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Target Metals and Default Reporting Limits

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-019-rev.10	1. Section 3.1: added reference to MDLs. 2. Table 9.2: removed reference to Profiling Standard 3. Table 9.3: removed reference to Profiling Standard 4. Section 9.2.3: removed reference to Profiling Standard 5. Section 9.2.3.2: changed to a tabular format 6. Section 10: removed reference to Profiling Standard. 7. Section 11.9: added language that over range results can be reported if qualified as estimated. 8. Table 12.1: revised method blank corrective action. 9. Inserted new Method Modifications section.	19Sep2012
S-IN-M-019-rev.11	1. Converted SOP to Corporate 27-section format. 2. Cover page: changed phone number, changed effective date format and revised document control format. 3. Table 7.1: added requirement to record date/time of preservation. 4. Table 10.3: updated standard sources and added Li and P. 5. Section 10.2.3: updated standard preparation. 6. Section 11: removed linear regression equation, made CRDL a requirement for each analytical batch, and removed BP requirements and replaced with "If required by client or program." 7. Table 1: updated RLs and added Li, Sr, and P.	18Dec2015
S-IN-M-019-rev.12	1. Table 7.1: updated storage temperature format. 2. Table 10.3: update standard IDs. 3. Section 10.2.3: updated standard preparation to match current procedures. 4. Section 11.7: removed "immediately following ICB" language. 5. Section 12.5: updated example sequence order. 6. Table 13.1: updated corrective action for method blank and LCS. 7. Section 14.1: updated equation to be in like terms with instrument output. 8. Section 18: moved PDS and SD from Section 13 to Section 18. 9. Section 25.3: added years 2003 and 2009 to TNI reference. 10. Section 26.1: updated title of Table 1. 11. Table 1: added "Default" to the title and added "subject to change" footnote.	27Dec2017

Table 1: Target Metals and Default Reporting Limits[1]

Metals	Aqueous (µg/L)	Solid (mg/kg)
Aluminum - Al	200	50
Antimony - Sb	6	1
Arsenic - As	10	1
Barium - Ba	10	1
Beryllium - Be	4	0.5
Boron - B	100	5
Cadmium - Cd	2	0.5
Calcium - Ca	1000	50
Chromium - Cr	10	1
Cobalt - Co	10	1
Copper - Cu	10	1
Iron – Fe	100	50
Lead – Pb	10	1
Lithium – Li	20	5
Magnesium – Mg	1000	50
Manganese – Mn	10	1
Molybdenum - Mo	10	1
Nickel – Ni	10	1
Phosphorus – P	N/A	50
Potassium - K	1000	50
Selenium - Se	10	1
Silver – Ag	10	0.5
Sodium – Na	1000	50
Strontium – Sr	10	1
Thallium - Tl	10	1
Tin – Sn	10	5
Titanium - Ti	10	1
Vanadium - V	10	1
Zinc – Zn	20	1

[1]Subject to change

ATTACHMENT C-13

THE DETERMINATION OF TOTAL HARDNESS
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF TOTAL HARDNESS
REFERENCE METHOD: STANDARD METHOD 2340 B (2011)

SOP NUMBER:	S-IN-M-032-rev.12
EFFECTIVE DATE:	October 23, 2017
SUPERSEDES:	S-IN-I-032-rev.11

APPROVAL

General Manager — October 9, 2017 Date

Quality Manager — October 9, 2017 Date

Department Manager — October 9, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining total hardness in aqueous samples while meeting the requirements specified in Standard Method 2340B, editorial revisions 2011.

2. Summary of Method

2.1. Hardness is computed from the results of separate determinations of calcium and magnesium by ICP analysis.

3. Scope and Application

3.1. This method is applicable for all concentration ranges of hardness.

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the determination of total hardness. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable for the measurement of total hardness in drinking, surface and saline waters and domestic and industrial wastes.

5. Limits of Detection and Quantitation

5.1. The default reporting limit for hardness is 1mg/L. Refer to the LIMS for calcium and magnesium method detection limits.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	250mL in plastic container	- HNO_3 to pH of <2 - Samples received at pH>2 must be preserved to pH<2 with HNO_3 and allowed to equilibrate for 24 hours before being prepared for analysis.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Vendor	Description / Comments
ICP-AES	Thermo-Fisher iCAP6500 or equivalent	Equipped with and autosampler and data system

9.2. General Supplies

Item	Vendor	Description
Refer to SOP for ICP analysis		

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Refer to SOP for ICP analysis	

10.2. Analytical Standards

10.2.1. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Calcium and Magnesium Calibration Standard	Refer to SOP for ICP analysis	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions

11. Calibration

11.1. ICP must be calibrated per applicable SOP.

12. Procedures

12.1. Analyze samples for Calcium and Magnesium per applicable metals digestion and ICP analysis SOPs.

12.2. Hardness is computed from the results of separate determinations of calcium and magnesium:

Calcium hardness as mg $CaCO_3$/L = 2.497[Ca, mg/L]

Magnesium hardness as mg $CaCO_3$/L = 4.118[Mg, mg/L]

Total hardness as mg $CaCO_3$/L = Calcium hardness + Magnesium hardness

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per analytical batch of up to 20 samples	Target analyte must be less than reporting limit.	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Sample Duplicate	Target analytes	One duplicate per 10 samples analyzed.	≤20% RPD	No corrective action necessary. Qualify data as appropriate

14. Data Analysis and Calculations

14.1. Refer to Section 12.2.

14.2. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Section 13.

16. Corrective Action for Out-of-Control Data

16.1. Refer to Section 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Section 13.

18. Method Performance

18.1. The analyst must read and understand this procedure with written documentation maintained in his/her training file.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. Equipment: Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling and Management or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Standard Methods for the Examination of Water and Wastewater"; method 2340B, 1997, Editorial Revisions 2011.

25.2. Pace SOPs S-IN-I-019 ICP Metals by 6010 and S-IN-I-131 ICP Metals 200.7, or their replacements.

25.3. Pace Analytical Quality Manual; latest revision.

25.4. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-032-rev.10	1. Table of Contents: added new Section 14, Method Modifications 2. Section 3.2: added reference to MDLs for Ca and Mg. 3. Table 7.1: copied table from ICP SOPs – includes contingency for underpreserved samples. 4. Table 8.1: replaced with table from ICP SOPs – more specific information regarding ICP equipment. 5. Tables 8.2 and 9.1: added reference to ICP SOPs 6. Table 12.1: updated corrective action for method blank and revised LCS control limit from 90-110% to 80-120% to match ICP SOPs. 7. Added new Section 14, Method Modifications 8. Section 16: added reference to ICP SOPs.	13May2013
S-IN-M-032-rev.11	1. Cover page: updated method reference to include date, changed SOP number to indicate "M" for metals department, updated document control format and changed phone number. 2. Section 11.2: updated calculations to reflect those in Method 2340B 1997, Editorial Revisions 2011 indicating that both Ca and Mg are expressed in mg/L $CaCO_3$. 3. Table 12.1: removed LCS. 4. Section 12: removed LCS equation. 5. Section 15.1: removed specific SOP reference. 6. Section 16.1: updated method reference to include date. 7. Section 16.2: added "or their replacements" regarding specified SOPs.	20Sep2015
S-IN-M-032-rev.12	1. Converted to 27 section format. 2. Section 1.1: added reference to 2011 editorial revisions of method. 3. Table 7.1: revised storage temperature format. 4. Section 25.4: added years 2003 and 2009 to TNI reference.	08Oct2017

ATTACHMENT C-14

THE DETERMINATION OF MERCURY BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY
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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF MERCURY BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

REFERENCE METHODS: EPA SW-846 METHODS 7470A AND 7471A

SOP NUMBER:	S-IN-M-040-rev.15
EFFECTIVE DATE:	July 5, 2017
SUPERSEDES:	S-IN-M-040-rev.14

APPROVAL

General Manager — June 22, 2017 Date

Quality Manager — June 22, 2017 Date

Department Manager — June 22, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining total mercury concentration while meeting the requirements specified in EPA method 7470A for aqueous samples and method 7471A for solid samples.

2. Summary of Method

2.1. Prior to analysis, all samples are digested by heating with appropriate acids and oxidizing agents to dissolve and oxidize mercury contents.

2.2. This cold-vapor method is based on the absorption of radiation at 253.7nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

3. Scope and Application

3.1. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of mercury analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable for the measurement of mercury in groundwater, surface and saline waters, domestic and industrial wastes, TCLP extracts, soil, sediment, bottom deposits and sludge-type materials.

5. Limits of Detection and Quantitation

5.1. The default reporting limits for mercury are 0.002 mg/L for aqueous samples and 0.20 mg/kg for solid samples. Refer to the LIMS for method detection limits.

6. Interferences

6.1. High concentrations of sulfide may interfere in some water or solid samples. Potassium permanganate is added during digestion to eliminate sulfide interference. Concentrations as high as 20mg/L in water or 20mg/kg in soils have been demonstrated to cause no interference in spiked samples.

6.2. High concentrations of copper have been reported to interfere with mercury determinations. Concentrations as high as 10mg/L in water or 10mg/kg in soil have been demonstrated to cause no interference in spiked samples.

6.3. High concentrations of chloride, present in samples require additional potassium permanganate. The free chlorine produced during digestion should be removed with excess hydroxylamine hydrochloride solution.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	500 mL in plastic container.	HNO_3 to pH<2 Samples received at pH>2 must be preserved to pH<2 with HNO_3 and be allowed to equilibrate for 24 hours before being prepared for analysis. Record date/time of preservation in preservation logbook.	Ambient	Analysis must be completed within 28 days of collection date.
Aqueous - Dissolved	500 mL in plastic container	Filter; HNO_3 to pH<2	Ambient	Analysis must be completed within 28 days of collection date.
Solid	100g in a 4oz glass container	None	Cool to ≤6°C	Analysis must be completed within 28 days of collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Automated Mercury Analyzer	CETAC/Teledyne Leeman	M-6100, M-7600 or equivalent	To include an atomic absorption spectrophotometer, mercury lamp, absorption cell, air pump, flow meter, drying tube, autosampler and data system.
Hot Block	Environmental Express	56-well or equivalent	Adjustable and capable of maintaining a temperature of 90°C to 95°C.
Balance	OHaus	GT400 or equivalent	Readability to 0.01g

9.2. General Supplies

Item	Vendor	Description
Auto-pipettes	Eppendorf or equivalent	Various sizes
Volumetric flasks	Class A	100mL
Graduated cylinder	Class A	25mL
Digestion cups	Environmental Express or equivalent	50mL capacity, volumetrically certified
Autosampler tubes	Moldpro, Inc or equivalent	17x100 mm
Plunger filters	Environmental Express or equivalent	For use with digestion cups

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Hydrochloric acid	Concentrated, trace metal grade or equivalent
Hydrochloric acid (3%)	Dilute 30 mLs of concentrated HCl to 1L with reagent water. Used for dilution preparation.
Nitric acid	Concentrated, trace metal grade or equivalent
Sulfuric acid	Concentrated, trace metal grade or equivalent
Aqua Regia	Carefully add one volume of nitric acid to three volumes of hydrochloric acid. Must be prepared in a hood and must be prepared immediately before use each day.
Stannous Chloride	Crystals, reagent grade
Stannous Chloride solution	Add 100g stannous chloride and 70 mL conc. HCl in reagent water and dilute to 1L. This solution is good for 3 days. Refrigerate when not in use.
Sodium Chloride	Crystals, reagent grade
Hydroxylamine hydrochloride	Crystals, reagent grade
Sodium Chloride/ Hydroxylamine Hydrochloride solution	Dissolve 120g sodium chloride and 120g hydroxylamine hydrochloride in reagent water and dilute to 1L. This solution is good for 6 months from preparation (hydroxylamine sulfate may be substituted for hydroxylamine hydrochloride).
Potassium permanganate solution (5%)	Commercially purchased Mercury-free, 5% solution (w/v)
Potassium persulfate	Crystals, reagent grade
Potassium persulfate solution	Dissolve 50g Potassium persulfate in reagent water and dilute to 1L. Expires 6 months from the date of preparation and can be stored at room temperature.
Rinse/Probe Wash Solution	Add 25mL HNO3 and 10mL HCl to 500mL reagent water and dilute to 1L.
Boiling chips	Or equivalent to be used as a simulated soil matrix.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Contract Required Detection Limit Standard	A standard prepared at a concentration equivalent to the reporting limit for verification at that level.	CRDL only if required by program or client
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analyte and is used to spike MS/MSD sets.	Same solution can be used for both the LCS and MS/MSD.

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Mercury Calibration standard	Ricca; catalog # AHG1KN; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Mercury Calibration standard	Refer to Section 10.2.3.1	Solution expires 6 months from date of preparation.	Same as for stock standard.
Daily Spike Mercury Calibration standard	Refer to Section 10.2.3.2	Must be prepared fresh daily.	Not Applicable
Working Mercury Calibration standards	Refer to Section 10.2.3.3	One-time use standards.	Not Applicable
Stock Mercury ICV/Spiking standard	SPEX; catalog # PLHG4-2Y; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Mercury ICV/Spiking standard	Refer to Section 10.2.3.4	Solution expires 6 months from date of preparation.	Same as for stock standard.
Daily Spike Mercury ICV/Spiking standard	Refer to Section 10.2.3.5	Must be prepared fresh daily.	Not Applicable
Working Mercury ICV standard	Refer to Section 10.2.3.6	One-time use standard.	Not Applicable
Working CRDL standard	Refer to Section 10.2.3.3	One-time use standard.	Not Applicable

10.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for specific instructions regarding preparation of standards for Mercury analysis

10.2.3.1 Intermediate Mercury Calibration Standard Preparation

Dilute 1mL of the Stock Mercury Calibration Standard (1000mg/L) to 100mLs with 2% HNO_3 for a final concentration of 10mg/L. This standard is good for 6 months from the date of preparation.

10.2.3.2 Daily Spike Mercury Calibration Standard Preparation

Dilute 1mL of the Intermediate Mercury Calibration Standard (10mg/L) to 100mLs with 2% HNO_3 for a final concentration of 100ug/L. This standard must be prepared fresh daily.

10.2.3.3 Working Mercury Calibration Standards Preparation

Working calibration standards are one-time use and are prepared by diluting the Daily Spike Mercury Calibration Standard (100ug/L) with reagent water. Examples of possible calibration standards are as follows:

Aqueous:

Standard ID	Amount of Daily Spike	Final Volume in reagent water	Final Concentration
Standard 1 (CRDL)	0.06mL	30mL	0.2ug/L
Standard 2	0.3mL	30mL	1.0ug/L
Standard 3	0.6mL	30mL	2.0ug/L
Standard 4 (CCV)	1.5mL	30mL	5.0ug/L
Standard 5	2.25mL	30mL	7.5ug/L
Standard 6	3.0mL	30mL	10.0ug/L

Solid:

Standard ID	Amount of Daily Spike	Final Volume in reagent water	Final Concentration
Standard 1 (CRDL)	0.1mL	50mL	0.2ug/L
Standard 2	0.5mL	50mL	1.0ug/L
Standard 3	1.0mL	50mL	2.0ug/L
Standard 4 (CCV)	2.5mL	50mL	5.0ug/L
Standard 5	3.75mL	50mL	7.5ug/L
Standard 6	5.0mL	50mL	10.0ug/L

10.2.3.4 Intermediate Mercury ICV/Spiking Standard Preparation

Intermediate Mercury ICV Standard: Dilute 1mL of the Stock Mercury ICV/Spiking Standard (1000mg/L) to 100mLs with 2% HNO_3 for a final concentration of 10mg/L. This standard is good for 6 months from the date of preparation.

10.2.3.5 Daily Spike Mercury ICV/Spiking Standard Preparation

Dilute 1mL of the Intermediate Mercury ICV/Spiking Standard (10mg/L) to 100mLs with 2% HNO_3 for a final concentration of 100ug/L. This standard must be prepared fresh daily and is also used to prepare the LCS and MS/MSD.

10.2.3.6 Working Mercury ICV Standard Preparation

Aqueous: Dilute 1.5mL of the Daily Spike Mercury ICV Standard (100ug/L) to 30mL with reagent water for a standard concentration of 5.0ug/L. This standard is a one-time use standard.

Solid: Dilute 2.5mL of the Daily Spike Mercury ICV Standard (100ug/L) to 50mL with reagent water for a standard concentration of 5.0ug/L. This standard is a one-time use standard.

11. Calibration

11.1. Initial Calibration: A minimum of a calibration blank and five calibration standards is required. The lowest calibration standard must be at or below the reporting limit. A new initial calibration curve with freshly prepared standard is analyzed on each working day. Refer to the Quality Manual for more information regarding calibration curves.

11.2. Linear Calibration: Using the instrumentation software, prepare a standard curve by plotting absorbance versus mercury concentration of each calibration standard. The analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

11.3. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be digested analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed. Refer to Section 11.12 for additional information.

11.4. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

11.5. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported. Refer to Section 11.12 for additional information.

11.6. Initial Calibration Blank (ICB): The ICB consists of reagent water that is prepared per Section 11. An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, the ICB may be reanalyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable. Refer to Section 11.12 for additional information.

11.7. Continuing Calibration Verification (CCV): A CCV must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.

11.8. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable. Refer to Section 11.12 for additional information.

11.9. Continuing Calibration Blank (CCB): The CCB consists of reagent water that is prepared per Section 11. A CCB must be analyzed after each CCV. If the CCB result is above the reporting limit, the CCB may be reanalyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable. Refer to Section 11.12 for additional information.

11.10. Contract Required Detection Limit Standard (CRDL): The CRDL is an optional check standard at or below the concentration of the reporting limit that is only analyzed if required by program or client. If required by client or program, the CRDL must be analyzed at the beginning of an analytical run, after every 20 samples, and at the end of the analytical run. The acceptable recovery range for the CRDL is 50-150%.

11.11. CRDL Corrective Action: If the CRDL is required by client or program and fails the acceptance criteria, another CRDL may be analyzed. If the second CRDL fails, associated samples must be qualified. **Exception:** If the CRDL is required and is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable without qualification.

11.12. Failure of the initial calibration, ICV, CCV, ICB or CCB that is due to improper or inadequate preparation requires the re-digestion and reanalysis of the associated preparation batch(es). Failure of the initial calibration, ICV, CCV, ICB or CCB due to instrument malfunction requires the instrument to be restored to proper working order and the reanalysis of samples associated with the failed QC.

12. Procedures

12.1. Aqueous Sample Preparation

12.1.1 Transfer a 30mL aliquot of well-mixed sample to a labeled 50mL graduated digestion cup.

12.1.2 Prepare a Method Blank by adding 30mL of reagent water to a labeled digestion cup.

12.1.3 Prepare an LCS by adding 1.5mL of the Daily Spike Mercury ICV/Spiking Standard (100ug/L) to a labeled digestion cup and diluting to 30mL with reagent water for a spike concentration of 5.0ug/L.

12.1.4 Prepare an MS and MSD set by transferring 30mL aliquots of well-mixed sample to separate labeled digestion cups and adding 1.5mL of the Daily Spike Mercury ICV/Spiking Standard (100ug/L) for a spike concentration of 5.0ug/L

12.1.5 Add 0.75mL concentrated HNO_3 to each digestion cup then add 1.5mL concentrated H_2SO_4 to each digestion cup, mixing after each addition.

12.1.6 Add 5mL of 5% potassium permanganate solution to each digestion cup. Ensure that equal amounts of permanganate solution are added to Method Blank and LCS. Swirl to mix. If the purple color does not persist after 15 minutes, then start over at Section 12.1.1 using a diluted aliquot of sample.

12.1.7 Add 2.5mL potassium persulfate solution to each digestion cup, cap loosely and heat samples for 2 hours in the Hot Block at 95°C.

12.1.8 Cool samples and add 1.8mL of sodium chloride/hydroxylamine hydrochloride solution to each sample to reduce the excess potassium permanganate. **CAUTION**: perform this addition in a fume hood, as chlorine gas could be produced. Proceed to Section 12.4.

12.2. Solid Sample Preparation

12.2.1 Weigh 0.3g of sample into a labeled 50mL digestion cup. To ensure the sample is representative of the entire container, the analyst should weigh out three 0.1g aliquots from different parts of the same container.

12.2.2 Prepare a Method Blank by placing several boiling chips into a labeled digestion cup.

12.2.3 Prepare an LCS by placing several boiling chips into a labeled digestion cup and adding 1.5mL of the Daily Spike Mercury ICV/Spiking Standard (100ug/L) for a final concentration of 0.5mg/Kg.

12.2.4 Prepare an MS and MSD by weighing 0.3g portions of a sample into separate labeled digestion cups and adding 1.5mL of the Daily Spike Mercury ICV/Spiking Standard (100ug/L) for a spike concentration of 0.5mg/Kg.

12.2.5 Add 5mL of reagent water to each digestion cup.

12.2.6 Add 2.5mL of aqua regia to each digestion cup.

12.2.7 Heat samples for 2 minutes in the Hot Block at 95°C.

12.2.8 Cool samples and add 25mL reagent water then add 7.5mL of 5% potassium permanganate

solution. Loosely cap each digestion cup.

12.2.9 Return the samples to the Hot Block and heat for 30 minutes at 95°C.

12.2.10 Cool samples again and add 3mL of the sodium chloride/hydroxylamine hydrochloride solution to each sample to reduce the excess potassium permanganate. **CAUTION**: perform this addition in a fume hood, as chlorine gas could be produced.

12.2.11 Adjust the digestate volumes to 50mL with reagent water and mix. Proceed to Section 12.4.

12.2.12 If needed, a plunger filter may be used to filter the digestate. The Method Blank and LCS must also be filtered if any client samples are filtered in the batch.

12.3. Calibration Standard Preparation

12.3.1 Prepare calibration standards in labeled 50mL digestion cups per the instructions in Section 10.2.3.3.

12.3.2 Follow steps in Section 12.1 to prepare calibration standards for aqueous matrix.

12.3.3 Follow steps in Section 12.2 to prepare calibration standards for solid matrix.

12.4. All sample volumes, reagent volumes, spiking standard volumes, standard/reagent ID numbers, hot block ID numbers, hot block temperature, thermometer ID number, and preparation date and time must be recorded in the electronic prep log.

12.5. Determination of Mercury

12.5.1 Configure the mercury analyzer according to manufacturer's instructions. Allow the colorimeter and recorder to warm up. Run a baseline with all reagents, using reagent water to flush the tubing. Whenever new tubing is used, allow ample time to flush the tubing.

12.5.2 Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.

12.5.3 Establish initial calibration as described in Sections 11.1 through 11.6.

12.5.4 Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration standards
ICV
ICB
CRDL (only if required)
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB
CRDL (only if required)

12.6. Any sample digestate with a mercury concentration that exceeds the linear range of the calibration curve must be diluted with 3% HCl solution and re-analyzed or over range results must be qualified as estimated. Alternatively, the sample may be re-digested at a dilution and re-analyzed.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or boiling chips	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be less than reporting limits	Re-digest and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	If original LCS is outside acceptance limits, re-analyze the LCS. If LCS is still outside acceptance limits, re-digest and re-analyze associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculations are performed directly by the instrument software. If dilutions were performed, the appropriate factors must be applied.

14.2. The instrument software calculates the amount of Mercury in the sample aliquot as follows:

$$X_s = (y - b)/a$$

Where: X_s = Concentration of the analyte
y = Total area or response of the analyte
a = slope of the line (the coefficient of x)
b = intercept of the line

14.3. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_f)(D)}{(V_i)} \qquad \text{Solid Sample (mg/kg)} = \frac{(X_s)(V_f)(D)}{(W_s)} \times 1000$$

Where: X_s = Mercury concentration, ug/L
V_f = Final sample volume of digestate, L
D = Dilution factor of the sample digestate
V_i = Initial sample volume digested, L
W_s = Weight of solid sample digested, mg

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

14.4. LCS equation:

$R = (C/S) * 100$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.5. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.6. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. Method Detection Limit (MDL) Study: An MDL study must be conducted annually for each matrix per instrument.

18.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Digestion procedure modified to use digestion cups in a hot block instead of BOD bottles in a water bath.

19.2. Standards and some reagents purchased as certified solutions.

19.3. Stannous Chloride solution not stirred continually because it is a solution and not a suspension.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. Equipment: Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 7470A and 7471A .

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not Applicable

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-040-rev.12	1. Section 3.2: added reference to MDLs and revised RL for solids. 2. Section 9.1: added potassium persulfate, wash solution and boiling chips. Revised or expanded other reagents. 3. Table 9.2: added RLVS 4. Table 9.3: added RLVS 5. Section 9.2.3.2: added RLVS 6. Section 10: added RLVS 7. Section 11.1.5: added that digestion cups be capped loosely for digestion. 8. Section 11.4.4: added RLVS 9. Section 11: added that calculations are performed by instrument software. 10. Section 11.8: added that over range results must be qualified. 11. Table 12.1: revised method blank corrective action. 12. Inserted new Method Modifications section.	27Sep2012
S-IN-I-040-rev.13	1. Table 9.1: revised details of Stannous Chloride reagent use and handling. 2. Section 14: added a modification for no continuous stirring of Stannous Chloride.	29Oct2012
S-IN-M-040-rev.14	1. Converted SOP to Corporate 27-section format. 2. Cover page: changed SOP name to reflect "M" for metals department, changed phone number, changed effective date format and changed document control format. 3. Section 9.2: added plunger filters. 4. Section 10.1: revised wash solution recipe. 5. Section 10: updated standard information and changed Intermediate #2 to Daily Spike. 6. Section 11: removed linear regression equation, made RLVS optional unless required by client or program and updated RLVS corrective action. 7. Section 12: changed Intermediate #2 to Daily Spike, updated procedure when permanganate color does not persist for 15 minutes, added optional use of plunger filters, and added requirement to document all information in the prep log.	15Dec2015
S-IN-M-040-rev.15	1. Section 5.1: updated default RL for solids. 2. Table 7.1: revised storage conditions for solids. 3. Section 9.1: updated instrument information. 4. Section 10.1: updated reagent information. 5. Tables 10.2 and 10.3: changed RLVS to CRDL. 6. Section 10.2.3.3: changed RLVS to CRDL. 7. Section 11.1: added requirement for calibration blank to be analyzed. 8. Sections 11.10 and 11.11: changed RLVS to CRDL. 9. Section 12.5.4: changed RLVS to CRDL. 10. Section 12.6: clarified that digestate can be diluted or sample can be re-digested at a dilution. 11. Table 13.1: updated LCS corrective action. 12. Section 14.3: updated units in equations and added x1000 to equation for solids. 13. Section 25: added years 2003 and 2009 to TNI reference.	21Jun2017

ATTACHMENT C-15

THE DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS)
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY (ICP-MS)

REFERENCE METHOD: EPA SW-846 METHOD 6020

SOP NUMBER:	S-IN-M-180-rev.01
EFFECTIVE DATE:	October 23, 2017
SUPERSEDES:	S-IN-M-180-rev.00

APPROVAL

General Manager — October 10, 2017 Date

Quality Manager — October 10, 2017 Date

Department Manager — October 10, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of metals in aqueous and solid environmental samples while meeting the requirements specified in SW-846 method 6020.

2. Summary of Method

2.1. Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples.

2.2. This method describes multi-element determinations by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS). The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced into a mass spectrometer. The ions are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier.

2.3. Interferences must be assessed and valid corrections applied or the data flagged. Interference correction must include compensation for background ions contributed by the plasma gas, reagents and constituents of the sample matrix.

3. Scope and Application

3.1. An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant native amounts of the recommended internal standards.

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ICP-MS systems and interpretation of ICP-MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to the determination of low concentrations of a large number of elements in water samples, solid samples, and in waste extracts or digestates. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

5. Limits of Detection and Quantitation

5.1. Refer to Table 1 for the list of target elements and reporting limits. Refer to the LIMS for method detection limits.

6. Interferences

6.1. **Spectral interferences:** Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. Isobaric molecular and doubly-charged ion interferences in ICP-MS

are caused by ions of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the applicable literature. Refer to Method 6020, Section 3.0 for more information regarding spectral interferences.

6.2. **Physical interferences:** Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers, reducing orifice size and instrument performance. Total solids levels below 0.2% are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

6.3. **Memory interferences:** When there are large concentration differences between samples or standards analyzed sequentially, memory interferences, or carryover can occur. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences. The rinse period between samples must be long enough to eliminate significant memory interference.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	250mL in plastic container	- HNO_3 to pH <2 - Samples received at pH>2 must be preserved to pH<2 with HNO_3 and allowed to equilibrate for 24 hours prior to digestion.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Aqueous - Dissolved	250mL in plastic container	- Filter; HNO_3 to pH <2 - Samples filtered in the lab are preserved to pH<2 with HNO_3 and allowed to equilibrate for 24 hours prior to digestion.	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.
Solid	50 grams in glass or plastic container	- No chemical preservation	Ambient or Cool to ≤6°C	Must be analyzed within 6 months of the collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Description / Comments
ICP-MS	Agilent 7700, or equivalent, equipped with autosampler and data system

9.2. General Supplies

Item	Description
Volumetric Flasks	Class A, various capacities
Volumetric Pipettors	Various sizes
Autosampler Vials	Environmental Express or equivalent
Analytical Balance	Ohaus or equivalent, capable of weighing to 0.01g
Graduated Cylinders	Class A, various capacities
pH strips	Full range

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Argon	High purity, liquefied
Nitric acid	Concentrated, trace metal analyzed or equivalent
Nitric acid, 1%	Place approximately 700mL reagent water into a 1L volumetric flask and add10mL concentrated nitric acid. Bring to volume with reagent water and mix well.
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent
Gold solution	Inorganic Ventures CGAUN-1, 1000ug/mL or equivalent for rinse water
Rinse Water	Dilute 200mL concentrated nitric acid, 100mL concentrated hydrochloric acid and 3mL Gold solution (1000ug/mL) to 10L with reagent water. This solution expires 3 months from the date of preparation.
Diluent (matrix match to digestates)	Dilute 10mL concentrated nitric acid and 5mL concentrated hydrochloric acid to 1L with reagent water. Concentration of acids may vary but must match digestate acid concentration.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Tuning Standard	A solution containing elements representing all of the mass regions of interest used to verify that the instrument resolution and mass calibration are within specifications and that the instrument has reached thermal stability.	
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analytes and is used for the Post Digestion Spike.	PDS
Internal Standards	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. These standards are used to adjust response ratios to account for instrument drift.	
Interference Check Standards	Prepared to contain a known amount of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections being used.	ICSA and ICSAB
CRDL Standard (Optional)	A standard prepared at or below the reporting limit for each element to verify recovery at that level. This standard is not required by Method 6020 but is optional.	CRDL

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Calibration Standards	Inorganic Ventures; catalog #'s HERT-CAL-5, PACE-49, and 2008CAL-1, and SPEX catalog #PLB9-2Y, or equivalent. See Section 10.2.3.1	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Boron Calibration Standard	Refer to Section 10.2.3.2	Standard is good for 3 months from date of preparation.	Same as stock standard
Working Calibration Standards	Refer to Section 10.2.3.3	Must be prepared fresh weekly	Same as stock standards
Stock ICV Standards	Inorganic Ventures; catalog #s HESIN-ICV-1 and HESIN-ICV-2, or equivalent. See Section 10.2.3.4	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate ICV Standard	Refer to Section 10.2.3.5	Standard is good for 1 month from date of preparation.	Same as stock standards
Working ICV Standard	Refer to Section 10.2.3.6	Must be prepared fresh weekly	Same as stock standard
Stock Interference Check Standard A (ICSA)	SPEX; catalog # CL-INT-A2, or equivalent. See Section 10.2.3.7	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate ICSA Standard	Refer to Section 10.2.3.8	Standard is good for 3 months from date of preparation.	Same as stock standard
Working ICSA Standard	Refer to Section 10.2.3.9	Must be prepared fresh weekly	Same as stock standards
Stock Interference Check Standards AB (ICSAB)	SPEX; catalog # CL-INT-A2 and Inorganic Ventures; catalog # HERT-CAL-2A, HERT-CAL-2B, or equivalent. See Section 10.2.3.10	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate ICSAB Standard	Refer to Section 10.2.3.11	Standard is good for 3 months from date of preparation.	Same as stock standard
Working ICSAB Standard	Refer to Section 10.2.3.12	Must be prepared fresh weekly	Same as stock standards
Stock CRDL Standard (Optional)	Inorganic Ventures; catalog #PACE-55-REV1, or equivalent. See Section 10.2.3.13	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working CRDL Standard (Optional)	Refer to Section 10.2.3.14	Must be prepared fresh weekly	Same as stock standards
Stock Internal Standards	Inorganic Ventures; catalog #HERT-IS-1, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Internal Standard	Refer to Section 10.2.3.16	Must be prepared fresh weekly	Same as stock standards
Stock Tune Standard	Inorganic Ventures; catalog #HERTVAR-TS-MS-REV1, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Tune Standard	Refer to Section 10.2.3.18	Must be prepared fresh weekly	Same as stock standards
Stock Spiking Standard #1	Inorganic Ventures; catalog #HERT-CAL-2A or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Stock Spiking Standard #2	Inorganic Ventures; catalog #HERT-CAL-2B or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions

10.2.3. Standard Preparation Procedures

10.2.3.1. Stock Calibration Standard Details

The following table shows the stock standard mixes that may be used to prepare the initial calibration and calibration check standards:

Analyte	Concentration (ug/mL)
SPEX Catalog # PLB9-2Y	
B	1000
Inorganic Ventures Catalog # 2008CAL-1	
Mo, Sb	20
Inorganic Ventures Catalog #PACE-49	
Al, Se	100
As, Be, Ba, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl, Th, U, V, Zn	20
Inorganic Ventures Catalog # HERT-CAL-5	
B, Sr, Ti, Sn	20

10.2.3.2. Intermediate Boron Calibration Standard Preparation

Dilute 1mL of the PLB9-2Y Stock Boron Standard (1000ug/mL) to 100mL with diluent for a final concentration of 10mg/L.

10.2.3.3. Working Calibration Standards Preparation

Prepared fresh weekly and diluted from the stock standard mixes in diluent, unless otherwise noted. Below are examples of calibration standards, actual standards may vary:

Working Calibration Std. ID	Volume Int. Boron Standard	Volume 2008CAL-1 Standard	Volume PACE-49 Standard	Volume HERT-CAL-5 Standard	Final Volume	Final Nominal Conc.
Calibration Blank	0mL	0mL	0mL	0mL	1000mL	0ug/L
Calibration Std. 1	0.3mL	0.1mL	0.1mL	0.1mL	1000mL	2ug/L
Calibration Std. 2	0mL	1mL	1mL	1mL	1000mL	20ug/L
Calibration Std. 3	0mL	10mL	10mL	10mL	1000mL	200ug/L

10.2.3.4. Stock ICV Standard Details

The following table shows the stock standard mixes that may be used to prepare the initial calibration verification standard:

Analyte	Concentration (ug/mL)
Inorganic Ventures Catalog #HESIN-ICV-1	
Al, As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Sr, Tl, Th, U, V, Zn	100
Inorganic Ventures Catalog #HESIN-ICV-2	
Sb, Mo, Sn, Ti	100

10.2.3.5. Intermediate ICV Standard Preparation

Dilute 0.5mL of the ICV-1 Stock Standard (100ug/mL) and 0.5mL of the ICV-2 Stock Standard (100ug/mL) to 100mL with diluent for a final concentration of 500ug/L.

10.2.3.6. Working ICV Standard Preparation

Dilute 1mL of the Intermediate ICV Standard (500ug/L) to 10mL with diluent for a final concentration of 50ug/L.

10.2.3.7. Stock Interference Check Standard A (ICSA) Details:

***SPEX CL-INT-A2* (ug/mL)**	
Al, Ca, Fe, Mg, P, K, Na, S	1000
C	2000
Chloride	10000
Mo, Ti	20

10.2.3.8. Intermediate ICSA Standard Preparation

Dilute 1mL of the Stock ICSA Standard (1000ug/mL nominal) to 10mL with diluent for a final nominal concentration of 100ug/mL.

10.2.3.9. Working ICSA Standard Preparation

Dilute 2mL of the Intermediate ICSA Standard (100ug/mL nominal) to 10mL with 1% nitric acid solution for a final nominal concentration of 20ug/mL.

10.2.3.10. Stock Interference Check Standard AB (ICSAB) Details:

***SPEX CL-INT-A2* (ug/mL)**	
Al, Ca, Fe, Mg, P, K, Na, S	1000
C	2000
Chloride	10000
Mo, Ti	20
***Inorganic Ventures HERT-CAL-2A* (ug/mL)**	
Si	20
Mo, Sb, Sn, Ti, Zr	2
***Inorganic Ventures HERT-CAL-2B* (ug/mL)**	
Al, Ca, Fe, K, Mg, Na	20
Ag, As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Sr, Th, Tl, U, V, Zn	2

10.2.3.11. Intermediate ICSAB Standard Preparation

Dilute 1mL of the CL-INT-A2 standard, 0.2mL of the HERT-CAL-2A standard and 0.2mL of the HERT-CAL-2B standard to10mL with diluent for a final nominal concentration of 40ug/L.

10.2.3.12. Working ICSAB Standard Preparation

Dilute 2mL of the Intermediate ICSAB Standard (40ug/L nominal) to 10mL with 1% nitric acid solution for a final nominal concentration of 8ug/L.

10.2.3.13. Stock CRDL Standard Details (Optional):

Inorganic Ventures PACE-55-REV1 ug/L	
Al	1000
B	500
Zn	300
Cr	200
As, Ba, Co, Cu, Mn, Mo, Pb, Sb, Se, Sn, Sr, Th, Ti, Tl, V	100
Ag, Ni	50
Be, Cd, U	20

10.2.3.14. Working CRDL Standard Preparation (Optional)

Dilute 1mL of the Stock CRDL Standard (20-1000ug/L) to 100mL with a 1% nitric acid solution for a final concentration as shown below:

Working CRDL Standard Concentration, ug/L	
Al	10
B	5
Zn	3
Cr	2
As, Ba, Co, Cu, Mn, Mo, Pb, Sb, Se, Sn, Sr, Th, Ti, Tl, V	1
Ag, Ni	0.5
Be, Cd, U	0.2

10.2.3.15. Stock Internal Standard Details:

Stock Internal Standard Concentration, ug/mL	
Bismuth	1000 (0.1%)
Indium	1000 (0.1%)
6-Lithium	1000 (0.1%)
Scandium	1000 (0.1%)
Terbium	1000 (0.1%)
Yttrium	1000 (0.1%)

10.2.3.16. Working Internal Standard Preparation

Dilute 1mL of Stock Internal Standard (1000ug/mL) to 1L with a 2% nitric acid solution for a final concentration of 1ug/mL (0.0001%).

10.2.3.17. Stock Tune Standard Details:

Stock Tune Standard Concentration, ug/mL	
Ba, Be, Ce, Co, In, Li, Mg, Pb, Th, Tl, U, Y	10

10.2.3.18. Working Tune Standard Preparation

Dilute 0.1mL of the Stock Tune Standard (10ug/mL) to 1L with a 1% nitric acid solution for a final concentration of 1ug/L.

11. Calibration and Standardization

11.1. Follow the instrument manufacturer's instructions for setup, tuning, calibration and operation of the ICP-MS. Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples. Rinse between samples using rinse water.

11.2. **Tuning:** The ICP-MS tuning standard must be analyzed to verify that the instrument has reached thermal stability and that resolution and mass calibration are within the required specifications. The tuning standard is analyzed at least five times and the relative standard deviation (RSD) must be $\leq$5% for all analytes contained in the tuning standard. Conduct mass calibration and resolution checks in the mass regions of interest. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be <0.9 amu full width at 5% peak height.

11.3. **Initial Calibration:** Calibrate the ICP-MS each working day according to the instrument manufacturer's recommended procedures. Flush the system with the Calibration Blank solution prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. The calibration curve must consist of a minimum of a calibration blank and three non-zero standards. Use the average of at least three integrations for both calibration and sample analyses.

11.4. **Linear Calibration:** Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The analyst may employ a regression equation that does not pass through the origin. When a multi-point calibration is performed, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.995.

11.5. **Initial Calibration Corrective Action:** If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.6. **Initial Calibration Verification (ICV):** In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

11.7. **ICV Corrective Action:** If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.8. **Initial Calibration Blank (ICB):** An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, another ICB may be analyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable.

11.9. **Continuing Calibration Verification (CCV):** A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The acceptable recovery range for the CCV is 90-110%.

11.10. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.11. Continuing Calibration Blank (CCB): A CCB must be analyzed immediately following each CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. Exception: If the CCB is >RL, associated samples determined to be <RL are reportable.

11.12. Interference Check Standard A (ICSA): An ICSA must be analyzed at the beginning of each analytical run or once every 12 hours. The ICSA must be 80-120% of the true value for the elements in the mix. All other analytes not included in the ICSA standard must be within +/-2x the reporting limit.

11.13. ICSA Corrective Action: If the ICSA fails the acceptance criteria, another ICSA may be analyzed. If the second ICSA fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSA must be reanalyzed. **Exception:** If the ICSA is >120% for any element in the mix or if any non-ICSA element is >2x the reporting limit, indicating high bias, associated samples determined to be <RL are reportable.

11.14. Interference Check Standard AB (ICSAB): An ICSAB must be analyzed at the beginning of each analytical run or once every 12 hours. The ICSAB must be 80-120% of the true value for the elements in the mix.

11.15. ICSAB Corrective Action: If the ICSAB fails the acceptance criteria, another ICSAB may be analyzed. If the second ICSAB fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSAB must be reanalyzed. **Exception:** If the ICSAB is >120% for any element in the mix, indicating high bias, associated samples determined to be <RL are reportable.

11.16. CRDL Standard (Optional): A CRDL standard may be analyzed prior to sample analysis and also at the end of each analytical batch to bracket the associated samples. Advisory range for all target elements is 50-150% recovery. No corrective action is required if the CRDL recovery is outside the advisory range.

12. Procedure

12.1. Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data document the selection criteria of background correction points; analytical dynamic ranges; the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis.

12.2. Configure the ICP-MS per manufacturer's instructions and allow it to become thermally stable. Tune and calibrate the instrument per the manufacturer's instructions and the requirements outlined in Section 10.

12.3. Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.

12.4. Once tuning initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. Rinse between samples using rinse water. An example sequence may be as follows:

Tuning Standard x 5
Calibration Blank (CAL0)
CAL1
CAL2
CAL3
Blank
ICV
ICB
CRDL (Optional)
ICSA
ICSAB
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB

12.5. The instrument performs three replicate readings for each analysis and the average of the three readings is used to derive the concentration. For samples, the difference between the three readings must be ≤20% RSD for values that are >4x the reporting limit. If the RSD is >20% for values that are >4x the reporting limit, the sample must be reanalyzed.

12.6. If dilutions were performed, the appropriate factors must be applied to sample values. Samples with analyte concentrations above the upper linear range must be diluted and reanalyzed or the over range results must be qualified as estimated.

12.7. Calculations must include appropriate interference corrections, internal-standard normalization, and the summation of signals at 206, 207, and 208 m/z for Lead.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or boiling chips	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be below the reporting limit.	Re-digest and reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) A matrix spike that passes LCS criteria may be used in place of a failed LCS for batch acceptance. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 3) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	If the MS/MSD fails, perform a Post Digestion Spike on the same sample used for the MS/MSD, as described in Section 12.5.
Internal Standards	Lithium, Scandium, Yttrium, Indium, Terbium, Bismuth	Automatically added to each sample, blank, and standard as part of the analysis.	**ICB/CCB/ICS:** 80-120% of CAL0 response. **All Others:** 30-120% of CAL0 response	When the internal standard response of the ICB, CCB, ICSA or ICSAB fails, recalibration and reanalysis of affected samples is required. When the internal standard response of other samples fails, the sample must be diluted fivefold or greater and reanalyzed.

13.2. Post-Digestion Spike Addition: If the MS/MSD recoveries are unacceptable, the sample from which the MS/MSD was performed should also be spike with a post digestion spike. Otherwise, another sample from the same preparation batch should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 75-125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the reporting limit. If this spike fails, then the dilution test should be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed. Post digestion spike is prepared by diluting 0.1mL of HERT-CAL-2A (2ug/mL) and 0.1mL of HERT-CAL-2B (2-20ug/mL) to 10mL with sample for a final concentration of 2ug/L (20ug/L for Al).

13.3. Dilution test: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the reporting limit after dilution), an analysis of a 1:5 dilution should agree within +/-10% of the original determination. If not, then a chemical or physical interference effect should be suspected.

14. Data Analysis and Calculations

14.1. Calculate the sample concentration using the following equations:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_f)(D)}{(V_i)} \qquad \text{Solid Sample (ug/kg)} = \frac{(X_s)(V_f)(D)}{(W_s)}$$

Where: X_s = Element concentration, ug/L
V_f = Final volume of digestate, L
D = Dilution factor
V_i = Initial sample volume digested, L
W_s = Weight of solid sample digested, kg

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

14.2. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

18.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Samples are analyzed for Boron, Molybdenum, Strontium, Thorium, Tin, Titanium, and Uranium in addition to the elements listed in Method 6020.

19.2. Tuning criteria observed is more stringent than required by the method so that the same criteria can be used for both methods 6020 and 200.8.

19.3. Rinse water contains a small amount of gold as recommended by instrument manufacturer to prevent plating of some elements in the sample introduction system.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. Equipment: Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Method 6020.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Target Elements and Reporting Limits

26.2. Table 2: Characteristic Masses and Internal Standard Assignments

27. Revisions

Document Number	Reason for Change	Date
S-IN-M-180-rev.00	1. Converted to Pace SOP format. 2. Added detailed tune criteria. 3. Separated Method 200.8 into its own SOP.	23Sep2015
S-IN-M-180-rev.01	1. Converted to 27-section format. 2. Table 7.1: revised storage temperature format. 3. Table 10.2: added "optional" to CRDL. 4. Table 10.3: revised stock ICSA, stock CRDL and stock tune information. 5. Section 10.2.3: revised stock ICSA, stock CRDL and stock tune information. 6. Section 11: reworded CCV and CCB sections for clarity. 7. Table 13.1: updated LCS corrective action and Internal Standard Acceptance Criteria and Corrective Action. 8. Section 13.2: changed acceptance criteria to 75-125% recovery. 9. Section 14.1: updated calculations to be in like terms with instrument output. 10. Section 25.3: added years 2003 and 2009 to TNI reference. 11. Table 1: updated barium RLs and added subject to change.	09Oct2017

Table 1: Target Elements and Reporting Limits[1]

Element	Aqueous RL (µg/L)	Solid RL (mg/kg)
Aluminum - Al	10	1
Antimony - Sb	1	0.1
Arsenic - As	1	0.1
Barium - Ba	1	0.5
Beryllium - Be	0.2	0.05
Boron - B	5	0.5
Cadmium - Cd	0.2	0.05
Chromium - Cr	2	0.2
Cobalt - Co	1	0.1
Copper - Cu	1	0.05
Lead – Pb	1	0.1
Manganese – Mn	1	0.1
Molybdenum - Mo	1	0.1
Nickel – Ni	0.5	0.05
Selenium - Se	1	0.1
Silver – Ag	0.5	0.05
Strontium – Sr	1	0.1
Thallium - Tl	1	0.1
Thorium – Th	1	0.1
Tin – Sn	1	0.1
Titanium - Ti	1	0.1
Uranium – U	1	0.1
Vanadium - V	1	0.05
Zinc – Zn	3	0.5

[1]Reporting limits are subject to change.

Table 2: Characteristic Masses and Internal Standard Assignments

Element	Characteristic Mass(es)	Internal Standard
Aluminum - Al	27	^{45}Sc
Antimony - Sb	121	^{115}In
Arsenic - As	75	^{89}Y
Barium - Ba	137	^{159}Tb
Beryllium - Be	9	^{6}Li
Boron - B	11	^{6}Li
Cadmium - Cd	114	^{115}In
Chromium - Cr	52	^{45}Sc
Cobalt - Co	59	^{45}Sc
Copper - Cu	65	^{45}Sc
Lead – Pb	206,207,208	^{209}Bi
Manganese – Mn	55	^{45}Sc
Molybdenum - Mo	98	^{89}Y
Nickel – Ni	60	^{45}Sc
Selenium - Se	78	^{89}Y
Silver – Ag	107	^{115}In
Strontium – Sr	88	^{89}Y
Thallium - Tl	205	^{209}Bi
Thorium – Th	232	^{209}Bi
Tin – Sn	118	^{115}In
Titanium - Ti	48	^{45}Sc
Uranium – U	238	^{209}Bi
Vanadium - V	51	^{45}Sc
Zinc – Zn	66	^{45}Sc

ATTACHMENT C-16

THE DETERMINATION OF VOLATILE ORGANICS BY GC/MS
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF VOLATILE ORGANICS BY GC/MS
REFERENCE METHOD: EPA SW-846 METHODS 8260C, 5030A, 5030B AND 5035A

LOCAL SOP NUMBER:	S-IN-O-029-rev.19
EFFECTIVE DATE:	December 19, 2016
SUPERSEDES:	S-IN-O-029-rev.18

LOCAL APPROVAL

General Manager — December 8, 2016 Date

Quality Manager — December 2, 2016 Date

Department Manager — December 2, 2016 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. This Standard Operating Procedure (SOP) documents the procedures used by Pace Analytical Services – Indianapolis to determine the concentration of Volatile Organic Compounds (VOCs) in environmental samples. The laboratory utilizes purge-and-trap GC/MS and bases these documented procedures on those listed in SW-846 Method 8260C, 5030A, 5030B, and 5035A.

2. Summary of Method

2.1. Volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot or extract using an inert gas. The purged analytes are collected on an absorbent trap. At the completion of the purge time, the trap is rapidly heated and back flushed to drive out the trapped analytes. The analytes are transferred into the inlet of a capillary gas chromatography column. The carrier gas flow through the column is controlled and the temperature is increased according to a set program to achieve optimum separation of purged analytes. The mass spectrometer is operated in a repetitive scan mode. Analytes are identified by the GC/MS retention times and by a comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected primary ion relative to an internal standard against a calibration curve.

3. Scope and Application

3.1. This method is applicable to most organic compounds that have boiling points below 200 ºC and are insoluble or slightly soluble in water. Volatile water-soluble compounds may also be determined although quantitation limits are typically higher due to their hydrophilic properties (e.g. ketones, oxygenates).

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable to most water and solid samples, regardless of moisture content. Matrices are groundwater, surface water, soil, sediment and waste. Procedures may need to be adapted to address limitations in the method or equipment that might hinder or interfere with sample analysis.

5. Limits of Detection and Quantitation

5.1. The list of target compounds and reporting limits is found in Table 1. Refer to the LIMS for method detection limits.

6. Interferences

6.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the absorbent trap. Many common solvents, most notably acetone and methylene chloride, are frequently found in laboratory air at low levels. The use of polytetrafluoroethylene (PTFE, Teflon) as thread sealants, tubing, or in flow controllers is highly recommended since other materials can be sources of contamination which may concentrate in the trap during the purging.

6.2. A common source of interfering contamination is carryover. This may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive action to this condition is rinsing the purging apparatus and sample syringes with organic free water between samples. Analyze one or more blanks to check for contamination prior to sample analysis. If the sample immediately following the high concentration sample does not contain the compounds present in the high level sample, freedom from carryover contamination has been established.

6.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

6.4. Since methylene chloride and acetone are common laboratory solvents, special precautions must be taken. The volatiles analysis and sample storage area must be located as far as possible from areas where these solvents are used or stored. Where possible, the volatiles analysis and sample storage area should be served by a separate HVAC system and maintained under positive pressure to prevent intrusion of contaminants. Laboratory clothing previously exposed to methylene chloride fumes during extraction procedures can contribute to sample contamination.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
5030B Aqueous	Minimum (3) VOA vials Additional sample is required if MS/MSD is requested	Acidified w/ 1:1 HCl to pH<2, no headspace	Cool to ≤6°C	pH>2: Analysis must be completed within 7 days of collection date. pH <2: Analysis must be completed within 14 days of collection date. (pH determined post analysis)
5035A Solid Terra Core Kits (Preferred)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) 5-g portions in vials with magnetic stir bar and 5.0mL reagent water plus one (1) 5 g portion in a vial with 5.0mL methanol. Additional sample is required if MS/MSD is requested.	Either no preservative or Methanol as a preservative.	Cool to ≤6°C for no more than 48 hours from collection then freeze at -7°C to -20°C. Methanol vials may be stored at 0° to 6°C.	Analysis must be completed within 48 hours if samples are not frozen or preserved with methanol prior to the expiration of the 48 hour period. The holding time may be extended to 14 days if the sample is frozen or preserved with methanol prior to the expiration of the 48 hour period.
5035A Solid Coring Devices (Alternate)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) EnCore, TerraCore or similar sampling tubes. Additional sample is required if MS/MSD is requested.	No preservative Sample is extruded into a vial with a magnetic stir bar and 5.0mL reagent water.	Freeze at -7°C to -20°C within 48 hours of collection.	Analysis must be completed within 14 days of collection date.
5030A Solid Bulk Jars	One (1) 2-4 oz. wide mouth jar for % moisture **AND** One bulk sample jar, usually 4 oz. or 8oz.	No preservative Sample is weighed into a vial with a magnetic stir bar and 5.0mL reagent water.	Cool to ≤6°C	Analysis must be completed within 14 days of collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatographs	Agilent	Lab uses models 6850 and 6890	Or equivalent system
P&T Concentrators	EST Analytical, Tekmar, OI	Tekmar 3000 series, Encon, Encon Evolution, and 4660 Eclipse	Or equivalent system
Data Systems	Agilent	Chemstation	Or equivalent system
Autosamplers	EST Analytical, OI	EST 8100, Centurion, Centurion WS, 4551	Or equivalent system
Mass Spectrometers	Agilent	5973 and 5975	Or equivalent system

9.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Agilent	J&W Scientific DB-624	20m x 0.18mm x 1um or equivalent
Trap	Supelco	Trap K and OI #10	Or equivalent

9.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	stop/go vials, various sizes	Supelco or equivalent
Balance, Analytical/Top Load	Able to measure to nearest 0.001g/0.01g	Mettler, OHaus or equivalent
Sample vials	40mL vials; pre-cleaned	Eagle Picher or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Methanol	Purge-and trap grade or equivalent
Sand	Or equivalent material to be used as a simulated soil matrix.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.1 Standard Definitions

Standard	Description	Comments
Tune Standard	4-Bromofluorobenzene (BFB) solution used to verify ion response ratios prior to analysis	Must inject between 5 and 50ng
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains required spiking compounds, at a minimum, and is used to prepare MS/MSD sets.	Same solution can be used for the LCS, MS/MSD and CCV.

10.2.2. Storage Conditions

Table 10.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock VOA calibration standards	o2si; catalog #122961-01; 250-1250ug/mL and #121106-02; 250-5000ug/mL; and #121093-04; 1000-2000ug/mL; and #020249-03; 2500ug/mL, or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Stock Gas calibration standards	o2si; catalog #120016-06; 250ug/mL and catalog #020229-09, 10,000ug/mL and #121093-04, 500/2000ug/mL, or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Intermediate VOA calibration standard	Refer to Section 10.2.3.1.	Solution good for 1 month from preparation	Same as stock standard.
Intermediate Gas calibration standard	Refer to Section 10.2.3.2.	Solution good for 1 week from preparation	Same as stock standard
Working VOA calibration standards	Refer to Section 10.2.3.3.	One-time use	Not applicable
Stock VOA ICV/Spiking standards	o2si; catalog #121092-02-SS; 250-5000ug/mL and #121091-06-SS, 250-5000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Intermediate VOA ICV/Spiking standard	Refer to Section 10.2.3.4.	Solution good for 1 month from preparation	Same as stock standard
Working ICV/Spiking standard	Refer to Section 10.2.3.5.	One-time use	Not applicable
Stock VOATune/ Surrogate standard	Restek; catalog #30240, 2500ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Stock VOA Internal standards	Restek; catalog #30241, 2500ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working Tune/Surrogate/Internal standard mix	Refer to Section 10.2.3.7.	Solution good for 1 month from preparation	Stored on autosampler under pressure in a 5mL vial.

10.2.3. Preparation Procedures

10.2.3.1. Intermediate VOA Calibration Standard Preparation (Example)

Dilute 1mL of o2si #122961-01 plus 1mL of o2si #121106-02 plus 100uL of o2si #020249-03 plus 1mL of o2si #121093-04 to 5.0mL with Methanol for a nominal concentration of 50mg/L.

10.2.3.2. Intermediate Gas Calibration Standard Preparation (Example)

Dilute 200uL of o2si #120016-06 plus 100uL of o2si #020229-09 plus 100uL of o2si #121093-04 to 1.0mL with Methanol for a nominal concentration of 50mg/L.

10.2.3.3. Working VOA Calibration Standards Preparation

Refer to Table 10.3 for examples of possible one-time use calibration standards.

Table 10.3 – Working Calibration Standards (examples only)

Standard	Int. VOA Cal. Standard amount	Int. Gas Cal. Standard amount	Final Total Volume	Final Concentration
Calibration Std 1	1uL	1uL	50mL	1ppb
Calibration Std 2	2uL	2uL	50mL	2ppb
Calibration Std 3	5uL	5uL	50mL	5ppb
Calibration Std 4	10uL	10uL	50mL	10ppb
Calibration Std 5	2uL	2uL	5mL	20ppb
Calibration Std 6 (CCV)	5uL	5uL	5mL	50ppb
Calibration Std 7	15uL	15uL	5mL	150ppb
Calibration Std 8	30uL	30uL	5mL	300ppb

10.2.3.4. Intermediate VOA ICV/Spiking Standard Preparation (Example)

Dilute 800uL of o2si #121092-02-SS plus 800uL of o2si #121091-06-SS to 4.0mL with Methanol for a final nominal concentration of 50mg/L.

10.2.3.5. Working ICV/Spiking Standard Preparation (Example)

Add 5uL of the Intermediate ICV standard per 5mL water for a final ICV concentration of 50ug/L.

10.2.3.6. Laboratory Control Sample (LCS) and Matrix Spike (MS/MSD) Preparation

10.2.3.6.1. **Aqueous LCS:** add 5uL of the Intermediate ICV/Spiking standard per 5mL reagent water for a nominal LCS concentration of 50ug/L.

10.2.3.6.2. **Aqueous MS:** add 5uL of the Intermediate ICV/Spiking standard per 5mL sample for a nominal MS concentration of 50ug/L.

10.2.3.6.3. **Low-level Soil LCS:** place 5 +/-0.5g of simulated soil matrix, 5mL reagent water and a stir bar into a vial. Add 5uL of the Intermediate ICV/Spiking standard for a nominal LCS concentration of 50ug/Kg.

10.2.3.6.4. **Low-level Soil MS:** place 5 +/-0.5g of sample, 5mL reagent water and a stir bar into a vial. Add 5uL of the Intermediate ICV/Spiking standard for a nominal MS concentration of 50ug/Kg.

10.2.3.6.5. Medium-level Soil LCS: place 4.8mL reagent water and 200uL methanol into a vial. Add 5uL of the Intermediate ICV/Spiking standard for an LCS concentration of 50ug/Kg.

10.2.3.6.6. Medium-level Soil MS: place a maximum of 200uL methanol sample extract into a vial and add enough reagent water to bring the final volume to 5mL. Add 5uL of the Intermediate ICV/Spiking standard for an LCS concentration of 50ug/Kg.

10.2.3.7. Working Tune/Surrogate/Internal Standard Preparation (Examples only, may vary)

Centurion/Centurion WS Autosamplers: Dilute 100uL of Restek #30240 plus 100uL of Restek #30241 to 5mL with Methanol for a final concentration of 50mg/L.

8100 Soil Autosamplers: Dilute 500uL of Restek #30240 plus 500uL of Restek #30241 to 5mL with Methanol for a final concentration of 250mg/L.

11. Calibration and Standardization

11.1. Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned by injecting 5-50ng BFB. This is done by analyzing a standard containing BFB. The BFB and calibration verification standard may be combined as long as both tuning and calibration verification acceptance criteria are met without interferences. Use the BFB mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected.

Mass (m/z)	Ion Abundance criteria
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	<2% of m/z 174
174	>50% of m/z 95
175	5 to 9% of m/z 174
176	95 to 101% of m/z 174
177	5 to 9% of m/z 176

The mass spectrum of BFB may be obtained by averaging three scans, the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using this approach and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other approaches suggested below:

1. A single scan within the BFB peak with background subtraction of a single scan no more than 20 scans prior to the elution of BFB.
2. An average of multiple scans within the BFB peak with background subtraction of a single scan no more than 20 scans prior to the elution of BFB.

If the ratios do not meet the criteria above, reanalyze the BFB tune. If the BFB still fails the criteria, instrument maintenance and/or preparation of new standards must be considered.

11.2. Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate

linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

11.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

11.4. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

11.5. The %RSD should be $\leq$20% for each target analyte.

11.6. For each calibration standard, all reported compounds that appear in Table 3 must meet the minimum response factor criteria shown.

11.7. If the percent relative standard deviation (%RSD) of the RFs for a compound is $\leq$20% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

11.7.1. If the % RSD for any compound is >20%, the analyst may employ a linear regression equation, non-weighted or weighted, that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.8. When calculating the calibration curve using the linear regression model, a minimum quantitation check on the viability of the calibration standard that corresponds to the reporting limit should be performed by re-fitting the response from the calibration standard that corresponds to the reporting limit back into the curve. The recalculated concentration of the reporting limit standard should be within +/-30% of the standard's true concentration. Reported compounds that fail this criteria must be qualified as estimated if the reported concentration is <2x the reporting limit. Alternatively, the reporting limit can be raised to the level of a calibration standard that meets the criteria when re-fitted against the curve.

11.9. Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be used and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of

contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be $\geq$0.99. Refer to Method 8000C for additional information regarding calibration.

11.10. If compounds fail to meet the criteria in Sections 11.5-11.9, the calibration fit must be set to average response factor and associated samples concentrations may be determined but they must be reported as estimated. In order to report non-detects, the compound must have been detected in the initial calibration standard that corresponds to the reporting limit.

11.11. Initial Calibration Corrective Action: If more than 10% of the compounds included with the initial calibration exceed the $\leq$20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternative curve fits, then the chromatographic system is considered too imprecise for analysis to begin. Instrument maintenance and/or preparation of new calibration standards must be considered prior to repeating the initial calibration procedure.

11.12. Each day that analysis is performed, the calibration standards and/or check standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.13. Initial Calibration Verification (ICV): The initial calibration curve should be verified immediately after performing the standard analysis using a second source standard (ICV) that is prepared using standards different from the calibration standards, with a concentration near the midpoint of the calibration range. The acceptance limits for the ICV are 70-130% recovery for all reported compounds, with the following exceptions:

Acetone	50-150%
Acrolein	50-150%
Bromomethane	50-150%
Iodomethane	50-150%
Methyl acetate	50-150%

11.14. ICV Corrective Action: If the ICV fails the criteria, another ICV may be analyzed. If the second ICV fails, a new initial calibration curve may be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Quantitative sample analysis should not proceed for those analytes that fail in the ICV or associated results must be qualified as estimated if analysis continues. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

11.15. Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a BFB tune that must meet the criteria in Section 10.1, followed by a Continuing Calibration Verification (CCV) standard. The CCV is normally prepared using the same standard solution used for the initial calibration but an ICV/LCS can be used as a CCV if it passes the required criteria for a CCV.

11.16. All target compounds in the CCV must be evaluated using a +/-20% variability criterion. Use percent difference when performing the average RF model calibration. Use percent drift when calibrating using a regression fit model.

$$\% \text{ Difference (\%D)} = \frac{\text{Calculated amount of standard} - \text{Expected amount of standard}}{\text{Expected amount of standard}} \times 100$$

$$\% \text{ Drift} = \frac{\text{Calculated concentration} - \text{Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

11.17. If the percent difference or percent drift for a compound is $\leq$20%, then the initial calibration for that compound is considered to be valid and sample analysis can continue. If the criterion is not met for more than 20% of the compounds included in the calibration, then corrective action must be taken prior to sample analysis.

In cases where compounds fail, they may still be reported as non-detects if the compound was detected in the initial calibration standard that corresponds to the reporting limit. For situations when the failed compound is present in samples, reported concentrations must be qualified as estimated values. Alternatively, the sample may be reanalyzed and reported for the compounds in question with a CCV that meets the criteria.

11.18. All reported compounds that appear in Table 3 must meet the minimum response factor criteria shown. If the minimum response factors are not met, the system should be evaluated and corrective action should be taken before sample analysis begins.

11.19. The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 10 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

11.20. CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. Alternatively, an ICV/LCS may be used as a CCV if it passes the CCV acceptance criteria. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. Exception: If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12. Procedures

12.1. Configure the purge & trap system and GC/MS system per manufacturer's instructions. All samples must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

12.2. Sample Preparation and Handling

12.2.1. Aqueous Samples

Water samples to be analyzed using the Centurion/OI autosampler require no sample preparation and are loaded as full 40mL VOA vials, unless they require a dilution. Refer to Section 7 for additional information regarding sample handling.

Water samples to be purged on the Archon/8100 autosampler are prepared by quickly measuring a 5mL aliquot of the sample using a 5mL gastight syringe and transferring it to a 40mL VOA vial.

This is done as quickly as possible to minimize analyte loss. The syringe is thoroughly rinsed inside and out with reagent water before measuring each sample.

Dilutions on aqueous samples must be prepared in a volumetric fashion. Sample aliquots are measured in either a volumetric pipet or gas-tight syringe and brought to volume in either a volumetric flask or gas-tight syringe.

After analysis, check the residue in the vial using pH paper. The pH should be <2 if HCl-preserved vials were used. Holding time for water samples with pH >2 is 7 days. Appropriately qualify on the sequence log and in LIMS any sample not meeting the pH requirement and/or holding time requirement. A stamp may be used to document on sequence logs that all water samples are pH<2 unless otherwise noted.

12.2.2. Soil Samples

12.2.2.1. Low-Level soils

Preferably, samples received for low level analysis should be contained in pre-weighed Terra Core vials with reagent water. Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial and correct for the label weight to determine the sample weight. The sample is ready for analysis. Refer to Section 7 for additional information regarding sample handling.

Alternatively, samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial either with or without 5mL reagent water and a stir bar. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight determined initially to determine the sample weight. The sample is ready for analysis.

Samples received in bulk soil jars are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of sample to the vial. Record the sample weight. Add 5mL of reagent water and a stir bar and cap the vial. The sample is ready for analysis.

12.2.2.2. Medium-Level soils

Preferably, samples received for medium-level analysis should be received in pre-weighed Terra Core vials with methanol as a preservative. Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial and correct for the label weight to determine the sample weight. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis. Refer to Section 7 for additional information regarding sample handling.

Alternatively, samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial with 5mL methanol. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight determined initially to determine the sample weight. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis.

Samples received in bulk soil jars are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add 5 +/-0.5g of sample to the vial. Record the sample weight. Add 5mL methanol and cap the vial. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL reagent water is used for analysis.

12.3. Qualitative Analysis: Compounds are identified as present when the following criteria are met:

12.3.1. The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

12.3.2. The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for the characteristic ions.

12.3.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.4. Quantitative Analysis: Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

12.5. Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

12.6. If the sample concentration exceeds the linear range of the analysis, the sample must be diluted and reanalyzed or reported as an estimated concentration.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required. 3) If contaminant is present in the sample at a concentration >10x the method blank, sample may be reported with qualification.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	Reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits RPD ≤20% Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Sample Duplicate (Dup)	Sample	One sample duplicate per batch of up to 20 samples if no MS/MSD.	RPD ≤20%	No corrective actions necessary. Qualify duplicate appropriately if RPD is out-of-control.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
As required by client only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed at the same dilution or more concentrated. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

13.2. Batch QC consisting of a Method Blank and LCS, at a minimum, is required for each batch of 20 or fewer samples for each matrix and preparation method combination (aqueous, low-level soil, and medium-level soil.).

13.3. Method Blank Preparation

13.3.1. Waters on Archon autosamplers: The Method Blank consists of a 40mL VOA vial containing 5mL reagent water.

13.3.2. Waters on Centurion autosamplers: The Method Blank consists of an HCl-preserved 40mL VOA vial filled completely with reagent water.

13.3.3. Low-level soils: The Method Blank consists of a 40mL VOA vial containing 5 +/-0.5g simulated soil matrix and 5mL reagent water and a stir bar.

13.3.4. Medium-level soils: The Method Blank consists of a 40mL VOA vial containing 4.8mL reagent water and 200uL methanol.

13.4. Laboratory Control Sample (LCS) and Matrix Spike (MS/MSD) Preparation: Refer to Section 10.2.3.6.

13.5. Allowable Marginal Exceedances: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: As allowed by client or program, the LCS may be outside the control limits but ≥10% recovery for up to four additional volatile compounds with the exception of benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, total xylenes and any requested oxygenate without corrective action.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = (X_s)(D) \qquad \text{Solid Sample (ug/kg)} = \frac{(X_s)(V_f)(D)}{(W_s)}$$

Where: X_s = On-column concentration of the analyte, ug/L
V_f = Final volume, L
D = Dilution factor
W_s = Weight of solid sample, kg

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

14.2. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where: R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where: R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where: RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control of Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL and/or LOD/LOQ verification study must be conducted annually for each matrix per instrument.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. GC columns and chromatographic conditions may differ from those recommended.

19.2. Calibration solutions are purchased as certified standards.

20. Instrument/Equipment Maintenance

20.1. Refer to maintenance log and/or instrument manufacturer's instructions.

21. Troubleshooting

21.1. Refer to maintenance log and/or instrument manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

24. Pollution Prevention

24.1. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24.2. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000C, 8260C, 5030A, 5030B, and 5035A.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Method 8260C Target Compounds and Reporting Limits
26.2. Table 2: Characteristic Ions and Internal Standard Association of Target Compounds
26.3. Table 3: Minimum Response Factor Criteria

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-029-rev.17	1. Section 3.1: added reference to MDLs 2. Section 9.1: added simulated soil matrix. 3. Section 9.2.3.6: added detail for preparation of LCS and MS to include simulated soil matrix. 4. Table 12.1: revised method blank corrective action. 5. Section 12.2.3: revised low-level soil method blank prep to include simulated soil matrix. 6. Inserted new Method Modifications section.	24Sep2012
S-IN-O-029-rev.18	1. Cover: changed 8260B to 8260C and added actual effective date. 2. Section 1.1: changed 8260B to 8260C 3. Section 4: revised to reflect 8260C 4. Table 8.3: revised balance specifications to match practice 5. Table 9.2: revised for standard mixes currently in use. 6. Section 9.2.3: revised recipes for standard mixes currently in use, changed approx. 5g to 5 +/- 0.5g, added batch QC details for medium-level soils, and detailed surrogate prep for both types of autosampler. 7. Section 10.1: added BFB acquisition guidance. 8. Section 10.13: added as guidance for evaluation of ICAL standards. 9. 10.5 - 10.21: revised to comply with Method 8260C, clarified use of LCS as CCV, and clarified requirement to correct vial TC vial weight for the label weight. 10. Section 11.2.2: added that tared soil vials need to be corrected for label weight and changed approx. 5g to 5 +/- 0.5g. 11. Section 11.5: added as a reference to the Manual Integrations SOP. 12. Section 11.6: added to require over range samples be diluted and reanalyzed or qualified as estimated. 13. Section 12.2: added to clarify the requirement for batch QC. 14. Section 12.3.2: clarified that blank is to be prepared using an HCl preserved vial. 15. Section 12.3.3: changed approx. 5g to 5 +/- 0.5g and added stir bar. 16. Section 13.1: added optional LOD/OQ verification. 17. Section 16.1: removed reference to 8000B and 8260A and changed 8260B to 8260C. 18. Section 17: changed 8260B to 8260C and added Table 3 attachment	31Oct2013

S-IN-O-029-rev.19	1. Converted to Corporate 27-section format. 2. Cover page: changed phone number and revised document control format. 3. Table 7.1: updated temperature format and preservation for 5035A. 4. Section 9.1: updated to include OI instrumentation. 5. Section 9.2: updated to include OI instrumentation. 6. Table 10.2: updated to current standards in use. 7. Section 10.2.3: updated to current standard preparation procedures. 8. Section 11: removed equations for different curve fits. 9. Section 11.7: added option of weighted linear. 10. Section 11.10: added requirement to set failing compounds to average fit. 11. Section 12.2.1: added OI and indicated that sample pH should be <2 if HCl vials were used. 12. Table 13.1: added an exception to MB when sample concentration is>10x MB concentration. Added sample duplicate to table and added RPD criteria. 13. Section 14: equations for water and solid final concentration fixed. 14. Table 1: added compounds for consistency between tables. 15. Table 2: added internal standard association and updated 1,2,3-TCP ions. 16. Table 3: revised minimum RF for TCE and PCE.	23Nov2016

Table 1: Method 8260C Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L)	RL soil Low-level (ug/kg)	RL soil Medium-level (ug/kg)
Dichlorodifluoromethane	5	5	125
Chloromethane	5	5	125
Vinyl Chloride	2	5	125
Bromomethane	5	5	125
Chloroethane	5	5	125
Trichlorofluoromethane	5	5	125
Acrolein	50	100	2500
1,1,2-Trichlorotrifluoroethane	5	5	125
1,1-Dichloroethene	5	5	125
Acetone	100	100	2500
Iodomethane	10	100	2500
Carbon Disulfide	10	10	250
Methylene Chloride	5	20	500
Acrylonitrile	100	100	2500
Methyl tert-butyl ether	4	5	125
trans-1,2-Dichloroethene	5	5	125
Vinyl Acetate	10	100	2500
1,1-Dichloroethane	5	5	125
2-Butanone (MEK)	25	25	625
cis-1,2-Dichloroethene	5	5	125
2,2-Dichloropropane	5	5	125
Bromochloromethane	5	5	125
Chloroform	5	5	125
Cyclohexane	100	100	2500
1,1,1-Trichloroethane	5	5	125
Carbon Tetrachloride	5	5	125
1,1-Dichloropropene	5	5	125
Benzene	5	5	125
1,2-Dichloroethane	5	5	125
Trichloroethene	5	5	125
Methylcyclohexane	50	50	1250
1,2-Dichloropropane	5	5	125
Dibromomethane	5	5	125
Bromodichloromethane	5	5	125
cis-1,3-Dichloropropene	5	5	125
4-Methyl-2-pentanone (MIBK)	25	25	625
Toluene	5	5	125
trans-1,3-Dichloropropene	5	5	125
Ethyl Methacrylate	100	100	2500
1,1,2-Trichloroethane	5	5	125
Tetrachloroethene	5	5	125
1,3-Dichloropropane	5	5	125
2-Hexanone	25	100	2500
Dibromochloromethane (Chlorodibromomethane)	5	5	125
1,2-Dibromoethane (EDB)	5	5	125
Chlorobenzene	5	5	125
1,1,1,2-Tetrachloroethane	5	5	125
Ethylbenzene	5	5	125
m&p-Xylene	5	5	125
o-Xylene	5	5	125

Analyte	RL water (ug/L)	RL soil Low-level (ug/kg)	RL soil Medium-level (ug/kg)
Styrene	5	5	125
Isopropylbenzene	5	5	125
Bromobenzene	5	5	125
trans-1,4-Dichloro-2-butene	100	100	2500
Bromoform	5	5	125
1,1,2,2-Tetrachloroethane	5	5	125
1,2,3-Trichloropropane	5	5	125
n-Propylbenzene	5	5	125
2-Chlorotoluene	5	5	125
1,3,5-Trimethylbenzene	5	5	125
4-Chlorotoluene	5	5	125
tert-Butylbenzene	5	5	125
1,2,4-Trimethylbenzene	5	5	125
sec-Butylbenzene	5	5	125
1,3-Dichlorobenzene	5	5	125
p-Isopropyltoluene	5	5	125
1,4-Dichlorobenzene	5	5	125
n-Butylbenzene	5	5	125
1,2-Dichlorobenzene	5	5	125
1,2-Dibromo-3-chloropropane	10	10	250
1,2,4-Trichlorobenzene	5	5	125
Hexachlorobutadiene	5	5	125
Naphthalene	5	5	125
1,2,3-Trichlorobenzene	5	5	125
2-Methylnaphthalene	10	10	250

[1]Target Compounds and Reporting Limits are subject to change.

Table 2: Characteristic Ions and Internal Standard Association of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Group 1 – Fluorobenzene (IS)	**96**	**-**
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
Trichlorofluoromethane	101	103
Acrolein	56	55
1,1,2-Trichlorotrifluoroethane	101	151
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Iodomethane	142	127
Carbon Disulfide	76	78
Methylene Chloride	84	86, 49
Acrylonitrile	53	52, 51
Methyl tert-butyl ether	73	57
trans-1,2-Dichloroethene	96	61, 98
Vinyl Acetate	43	86
1,1-Dichloroethane	63	65, 83
2-Butanone (MEK)	43	57, 72
cis-1,2-Dichloroethene	96	61, 98
2,2-Dichloropropane	77	97
Bromochloromethane	49	128
Chloroform	83	85
Dibromofluoromethane (Surr)	113	111
Cyclohexane	56	84, 41
1,1,1-Trichloroethane	97	99, 61
Carbon Tetrachloride	117	119, 121
1,1-Dichloropropene	75	110, 77
Benzene	78	52, 77
1,2-Dichloroethane	62	98, 64
Trichloroethene	95	97, 130, 132
Methylcyclohexane	55	69, 83
1,2-Dichloropropane	63	62, 112
Dibromomethane	93	95, 174
Bromodichloromethane	83	85, 127
Group 2 – Chlorobenzene-d5 (IS)	**117**	**82, 119**
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone (MIBK)	43	58, 85
Toluene-d8	98	99, 100
Toluene	91	92
trans-1,3-Dichloropropene	75	77
Ethyl Methacrylate	69	99, 114
1,1,2-Trichloroethane	83	97, 85
Tetrachloroethene	166	129, 168
1,3-Dichloropropane	76	78
2-Hexanone	43	58, 100
Dibromochloromethane (Chlorodibromomethane)	129	127
1,2-Dibromoethane (EDB)	107	109
Chlorobenzene	112	77, 114

Analyte	Primary Ion	Secondary Ion(s)
Group 2 – Chlorobenzene-d5 (IS) Continued	**117**	**82, 119**
1,1,1,2-Tetrachloroethane	131	133, 119
Ethylbenzene	106	91
m&p-Xylene	106	91
o-Xylene	106	91
Styrene	104	78
Isopropylbenzene	105	120
4-Bromofluorobenzene (Surr)	95	174, 176
Bromobenzene	77	156, 158
trans-1,4-Dichloro-2-butene	53	88, 75
Group 3 – 1,4-Dichlorobenzene-d4 (IS)	**152**	**115, 150**
Bromoform	173	175, 254
1,1,2,2-Tetrachloroethane	83	131, 85
1,2,3-Trichloropropane	110	75, 77
n-Propylbenzene	91	120
2-Chlorotoluene	91	126
1,3,5-Trimethylbenzene	105	120
4-Chlorotoluene	126	91
tert-Butylbenzene	119	91, 134
1,2,4-Trimethylbenzene	105	120
sec-Butylbenzene	105	134
1,3-Dichlorobenzene	146	111, 148
p-Isopropyltoluene	119	134, 91
1,4-Dichlorobenzene	146	111, 148
n-Butylbenzene	91	92, 134
1,2-Dichlorobenzene	146	111, 148
1,2-Dibromo-3-chloropropane	155	75, 157
1,2,4-Trichlorobenzene	180	182, 145
Hexachlorobutadiene	225	223, 227
Naphthalene	128	127
1,2,3-Trichlorobenzene	180	182, 145
2-Methylnaphthalene	142	141, 115

[2]Subject to change.

Table 3: Minimum Response Factor Criteria

Analyte	Minimum Response Factor (RF)
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
Methylene Chloride	0.100
1,1-Dichloroethene	0.100
trans-1,2-Dichloroethene	0.100
1,1-Dichloroethane	0.200
cis-1,2-Dichloroethene	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon Tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
*Trichloroethene	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
Toluene	0.400
1,1,2-Trichloroethane	0.100
*Tetrachloroethene	0.100
Dibromochloromethane (Chlorodibromomethane)	0.100
1,2-Dibromoethane (EDB)	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
m&p-Xylene	0.100
o-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2,4-Trichlorobenzene	0.200
trans-1,3-Dichloropropene	0.100
cis-1,3-Dichloropropene	0.200
*Acetone	0.010
*2-Butanone (MEK)	0.010
*4-Methyl-2-pentanone (MIBK)	0.050
*2-Hexanone	0.050
Methyl tert-butyl ether	0.100
Carbon Disulfide	0.100
1,2-Dibromo-3-chloropropane	0.050
Methylcyclohexane	0.100
1,1,2-Trichlorotrifluoroethane	0.100
Methyl acetate	0.100

*Alternate minimum RF criteria based on compound performance.

ATTACHMENT C-17

THE DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS)
PACE, INDIANAPOLIS

Pace Analytical Services, LLC
7726 Moller Road
Indianapolis, IN 46268
Phone: 317.228.3100
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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS)

REFERENCE METHOD: EPA SW-846 METHOD 8082A

SOP NUMBER:	S-IN-O-050-rev.16
EFFECTIVE DATE:	May 1, 2018
SUPERSEDES:	S-IN-O-050-rev.15

APPROVAL

General Manager — April 27, 2018 Date

Quality Manager — April 27, 2018 Date

Department Manager — April 26, 2018 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of polychlorinated biphenyls (PCBs) in aqueous and solid samples, including oils and wipes, while meeting the requirements specified in EPA method 8082A.

2. Summary of Method

2.1. Samples are extracted using a technique and solvent system appropriate for the given matrix.

2.2. Cleanup steps may be applied to the extract, if necessary, depending on the nature of the matrix interferences and the target analytes.

2.3. The extract is analyzed by a gas chromatograph fitted with an electron capture detector (ECD). Aroclor identification is confirmed by a secondary column.

3. Scope and Application

3.1. Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns than those of Aroclor standards.

3.2. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC systems and interpretation of GC PCB data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This procedure is used to determine the concentrations of PCBs as Aroclors in extracts from solid, aqueous, oil and wipe matrices.

5. Limits of Detection and Quantitation

5.1. The list of reporting limits can be found in Table 1. Reporting limits may vary as a function of volume or weight of sample extracted and extract final volume. Refer to LIMS for method detection limits

6. Interferences

6.1. Matrix interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware. These interferences lead to discrete artifacts or elevated baselines in gas chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates, which are easily extracted during lab operations. Avoiding the use of plastics in the lab can best minimize interferences from phthalates.

6.2. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled. Cleanup procedures may be used to remove such interferences. Refer to the appropriate cleanup SOPs if extract cleanup to remove interferences is required.

6.3. Equipment

Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage, and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	100mL widemouth amber glass bottle	None required	Cool to $\leq 6^{o}C$	Extract within 6 months of collection and analyze within 40 days of extraction
Solid	>100 grams in 4 or 8oz glass jar	None required	Cool to $\leq 6^{o}C$	Extract within 6 months of collection and analyze within 40 days of extraction
Oils	>10 grams in a glass container	None required	Cool to $\leq 6^{o}C$	Extract within 6 months of collection and analyze within 40 days of extraction
Wipes	One wipe per glass container	None required	Cool to $\leq 6^{o}C$	Extract within 6 months of collection and analyze within 40 days of extraction

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
GC	Agilent, 7890A LTM module optional or equivalent	Equipped with dual ECD Detectors; dual columns, and dual injectors, or equivalent system.
Autosampler	Agilent, 7693 or equivalent	150 position
Data System	Chemstation acquisition; Target integrations	Or equivalent software

Equipment	Vendor	Description / Comments
GC Columns	Restek, DB5-MS or DB35-MS or equivalent RTX-CLP1 and RTX-CLP2 or equivalent	Fused silica; 30 meters, 0.32mm ID, 0.25um film thickness, or equivalent column Fused silica: 30m x0.32mm ID x 0.32um and 30m x 0.32mm x 0.25um, or equivalent
LTM GC Columns	Restek, DB-35-MS LTM or equivalent Restek, DB5-MS LTM or equivalent	Fused silica; 15 meters, 0.32mm ID, 0.25um film thickness, or equivalent column Fused silica; 15 meters, 0.32mm ID, 0.25um film thickness, or equivalent column

9.2. General Supplies

Item	Vendor	Description
Glass syringes	Hamilton or equivalent	Various sizes
Glass vials	Fisher or equivalent	20mL volume with Teflon lined caps
Autosampler vials	Fisher or equivalent	2mL volume with Teflon lined crimp tops
Micro-inserts	Hewlett Packard or equivalent	Various sizes

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Hexane	Pesticide or reagent grade or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, initial calibration verification and continuing calibration verification.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for required target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	CCV
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the calibration curve.	ICV

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock PCB Calibration standards	Ultra Scientific 1016 cat#EPA-1282, 1260 cat#EPA-1362, each at 1000ug/mL, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate PCB Calibration standards	Refer to Section 10.2.3.1	Solution good for 6 months from preparation.	Refrigerate
Working PCB Calibration standards	Refer to Section 10.2.3.2	Solution good for 6 months from preparation.	Refrigerate
Stock PCB ICV standard	Restek 1016/1260 cat#32039, each at 1000ug/mL, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate PCB ICV standard	Refer to Section 10.2.3.3	Solution good for 6 months from preparation.	Refrigerate
Working PCB ICV standard	Refer to Section 10.2.3.4	Solution good for 6 months from preparation.	Refrigerate
Stock PCB Surrogate standard	Ultra; catalog #ISM-320; TCMX; 200ug/mL, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock Single Point PCB Calibration Standards	Ultra Scientific 1221 cat #EPA-1292, 1232 cat #EPA-1302, 1242 cat #EPA-1312, 1248 cat #EPA-1342, 1254 cat #EPA-1352, 1262 cat #EPA-1372 and 1268 cat #EPA-1382, each at 1000ug/mL, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Intermediate Single Point PCB Calibration Standards	Refer to Section 10.2.3.5	Solution good for 6 months from preparation.	Refrigerate
Working Single Point PCB Calibration Standards	Refer to Section 10.2.3.6	Solution good for 6 months from preparation.	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1. Intermediate PCB Calibration Standard Preparation

Dilute 100uL of the Stock Aroclor 1016 Calibration standard (1000ug/mL), 100uL of the Stock Aroclor 1260 Calibration standard (1000ug/mL) and 50uL of the Stock PCB Surrogate standard (200ug/mL) to 50mL with Hexane for a final Aroclor 1016/1260 concentration of 2ug/mL and a final surrogate concentration of 0.2ug/mL. This recipe can also be used to prepare an intermediate calibration standard for other Aroclors.

10.2.3.2. Working PCB Calibration Standard Preparation

The following are examples of calibration standard concentrations and could vary based on requirements:

Standard	Intermediate PCB Cal. Std. amount	Final Volume in Hexane	Final PCB Conc, ug/L	Final Surr Conc, ug/L
CAL1	2.5uL	1mL	5	0.5
CAL2	5uL	1mL	10	1
CAL3	25uL	1mL	50	5
CAL4	50uL	1mL	100	10
CAL5 (CCV)	250uL	1mL	500	50
CAL6	300uL	800uL	750	75
CAL7	500uL	1mL	1000	100

10.2.3.3. Intermediate PCB ICV Standard Preparation

Dilute 20uL of the Stock PCB ICV standard (1000ug/mL) and 10uL of the Stock PCB Surrogate standard (200ug/mL) to 20mL with hexane for a final Aroclor 1016/1260 concentration of 1ug/mL and a final surrogate concentration of 0.1ug/mL. This recipe can also be used to prepare an intermediate ICV standard for other Aroclors.

10.2.3.4. Working PCB ICV Standard Preparation

Dilute 500uL of the Intermediate PCB ICV standard (1/0.1ug/mL) to 1mL with hexane for a final Aroclor 1016/1260 concentration of 500ug/L and a final surrogate concentration of 50ug/L.

10.2.3.5. Intermediate Single Point PCB Calibration Standard Preparation

The following are examples of intermediate single point calibration standards and could vary based on requirements:

Standard	Stock Single Point PCB Calibration Standard amount	Stock PCB Surr. amount	Final Volume in Hexane	Final PCB Concentration, ug/mL	Final Surr Concentration, ug/mL
Working 1221/1254	10uL each	5uL	20mL	0.5 each	0.05
Working 1232/1262	10uL each	5uL	20mL	0.5 each	0.05
Working 1242/1268	10uL each	5uL	20mL	0.5 each	0.05
Working 1248	10uL	5uL	20mL	0.5	0.05

10.2.3.6. Working Single Point PCB Calibration Standard Preparation

The following are examples of working single point calibration standards and could vary based on requirements:

Standard	Intermediate Single Point PCB Calibration Standard amount	Final Volume in Hexane	Final PCB Concentration, ug/L	Final Surr Concentration, ug/L
Working 1221/1254	200uL	1mL	100 each	10
Working 1232/1262	200uL	1mL	100 each	10
Working 1242/1268	200uL	1mL	100 each	10
Working 1248	200uL	1mL	100	10

11. Calibration

11.1. Before the initial calibration standards are injected, it is advisable to perform routine injection port and column maintenance due to the sensitivity of the ECD detector.

11.2. **Initial Calibration**: Initial calibration standards are introduced into the GC from the lowest to highest concentration by direct injection. Five calibration points, at a minimum, are analyzed to evaluate linearity. The lowest calibration standard must be at or below the required reporting limit. Refer to the Quality Manual for more information regarding calibration curves.

11.2.1. A mixture of Aroclor 1016 and 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. An initial 5-point calibration is performed using the mixture of Aroclors 1016 and 1260.

11.2.2. Standards of the other Aroclors are necessary for pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to demonstrate the linearity of the detector, these single standards of the remaining Aroclor may be used to determine a single-point calibration factor for each Aroclor. The standards for these other Aroclors should be analyzed before the analysis of any samples, and before the analysis of the Aroclor 1016/1260 initial calibration curve.

11.2.3. In situations where only one or a few Aroclors are of interest for a specific project, the analyst may employ a five-point initial calibration of each of the Aroclors of interest and not use the 1016/1260 mixture or the pattern recognition standards.

11.3. Record the peak area (or height) for each Aroclor peak to be used for quantitation. A minimum of 3 peaks must be chosen for each Aroclor, preferably 5 peaks are used. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. A book of reference chromatograms for each Aroclor has been created for each instrument. Refer to these books for the preferred peaks used for calibration of each Aroclor.

11.4. Calculate the Calibration Factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards using the calculation below:

$$CF = \frac{\text{Peak Area or Height in the Standard}}{\text{Mass of the Standard Injected, ng}}$$

Five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the 5 peaks chosen for this mixture. The single standard for each of the other Aroclors will generate at least 3 calibration factors, one for each selected peak.

11.5. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{CF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
CF_{avg} = Mean of CFs for a peak

11.6. If the %RSD of the CFs is $\leq$ 20% over the calibration range, then the slopes of the lines for each standard are sufficiently close to one another and the average CF may be used to determine sample concentrations.

11.7. If any %RSD is >20%, the analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.8. When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the calibration model chosen for this mixture must be applied to the other 5 Aroclors for which only single standards are analyzed. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate linearity.

11.9. **Initial Calibration Corrective Action:** If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.10. Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.11. **Initial Calibration Verification (ICV):** In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed

ICV according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. The ICV recoveries are evaluated against a default acceptance range of 70-130% recovery.

11.12. ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

11.13. Daily and Continuing Calibration: Verify the initial calibration each 12-hour shift by injecting a Continuing Calibration Verification (CCV) standard prior to conducting any sample analyses.

The CCV must also be analyzed at intervals of once every 20 client samples and at the end of the analysis sequence. For Aroclor analyses, the CCV standard should be a mixture of Aroclors 1016 and 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition.

11.14. For initial calibrations that employed average calibration factor, the calibration factor (CF_v) for each analyte calculated from the CCV must not exceed a %Difference (%D) of more than +/-20% when compared to the CF_{avg} from the initial calibration curve. For initial calibrations that employed a linear calibration, the % Drift for each analyte calculated from the CCV must be within +/-20% in order to use the calibration model to quantitate sample results.

$$\% \text{ Drift} = \frac{\text{Calculated concentration} - \text{Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

11.15. CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12. Procedures

12.1. All sample extracts must be analyzed at room temperature and the system must be tuned and calibrated as per Section 11, and free of contamination before samples are analyzed.

12.2. Gas Chromatography conditions: Configure the GC per manufacturer's instructions.

12.3. Inject aliquots of all sample extracts and quality control into the GC under the same operating conditions as used for the calibration standards. The sample vials are loaded onto the autosampler that is programmed via the data system to inject the necessary volume.

12.4. Qualitative Analysis: Compounds are identified as present when the following criteria are met:

12.4.1. Absolute retention times are used for the identification of PCBs as Aroclors. Retention time windows are established on both the primary and the confirmation column to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize

the occurrence of both false positive and false negative results. To establish retention time windows, make three or more injections of a standard over the course of a 72-hour period, at a minimum. Record the retention time in minutes for the major peaks and surrogate to three decimal places. Calculate the mean and standard deviation of the absolute retention times of the standard. The retention time window is defined as +/-3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

12.4.2. Establish the center of the retention time window for each major peak and surrogate by using the absolute retention time for each major peak and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

12.4.3. It may also be useful to establish the center of the retention time window for single point standards by using the absolute retention time for each major peak and surrogate from the single point standards analyzed at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the single point standards analyzed with the initial calibration.

12.4.4. Tentative identification of a PCB compound is made when peaks from the sample extract fall within the established retention time windows for Aroclor peaks. The results of a single column/single injection analysis may be confirmed, if necessary, on a second, dissimilar, GC column. In order to be used for confirmation, retention time windows must have been established for the second GC column. In addition the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis. When the dual-column approach is employed, the target Aroclors are identified and confirmed when they meet the identification criteria on both columns. When confirmation is made on a second column, that analysis should meet all of the QC criteria described above for calibration, retention times, etc.

12.4.5. A book of reference chromatograms for each Aroclor has been created for each instrument. Refer to these books for the preferred peaks and ratios between peaks that are characteristic of each Aroclor. When interferences are present, degradation has occurred, or multiple Aroclors are suspected, the following tools are helpful for proper identification:

- Overlays of the sample chromatogram with chromatograms of Aroclor standards
- Comparison of characteristic peak retention times with Aroclor standards
- Comparison of the ratio between characteristic peaks with ratios of Aroclor standards
- Comparison with historical results, if available
- Analyst judgment and consultation with other experienced analysts
- Consistent application of evaluation tools

12.5. Quantitative Analysis

12.5.1. The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

12.5.2. Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen and the calibration model established from the multi-point calibration of 1016/1260. A concentration is determined using each of the characteristic peaks and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor. Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard or calibration standards interspersed within the samples. The results from these bracketing standards must meet the calibration verification criteria in Sections 11.13 through 11.15.

12.5.3. A book of reference chromatograms for each Aroclor has been created for each instrument. Refer to these books for the preferred peaks used for quantitation of each Aroclor. When interferences are present or degradation has occurred, peaks yielding concentrations or areas that are dissimilar to the others may be excluded. When multiple co-eluting Aroclors are suspected, quantitation of Aroclor(s) should be based on the best match with established Aroclor patterns as determined using the tools outlined in Section 12.4.5.

12.5.4. When PCB concentrations exceed the calibration range, the sample extract should be rerun at a dilution or the result must be qualified as estimated.

12.5.5. Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

13. Quality Control

13.1. Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or sodium sulfate	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample results are non-detect, the sample results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

Aqueous Sample (ug/L) = $\frac{(X_s)(V_f)(D)}{(V_i)} * 1000$ **Solid Sample (ug/kg)** = $\frac{(X_s)(V_f)(D)}{(W_s)} * 1000$

Where: X_s = On-column concentration of the analyte in ug/mL
V_f = Final volume of extract in Liters
D = Dilution factor of concentrated extract
V_i = Volume of aqueous sample extracted in Liters
W_s = Weight of solid sample extracted in kilograms

Moisture corrected concentration = $\frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$

Oil Sample (mg/kg) = $\frac{(X_s)(V_f)(D)}{(W_s)}$ **Wipe Sample (Total ug)** = $(X_s)(V_f)(D)$

Where: X_s = On-column concentration of the analyte in ug/mL
V_f = Final volume of extract in milliliters
D = Dilution factor of concentrated extract
W_s = Weight of solid sample extracted in kilograms

14.2. LCS equation

R = (C/S) * 100

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD calculations:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. MDLs must be conducted per EPA *Definition and Procedure for the Determination of the Method Detection Limit, Revision 2*; December 2016.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability (DOC) study.

19. Method Modifications

19.1. Extracts are not stored in the dark. Sample extracts are analyzed within a few days of extraction and are not exposed to light for an extended period of time.

19.2. A standard of the DDT analogs is not analyzed and evaluated because interference from DDT and its breakdown components is more applicable to congener analysis than Aroclor analysis. Analysis of the DDT analogs is not considered a method requirement.

19.3. Single point Aroclor standards may be combined in the same way that Aroclors 1016 and 1260 are combined.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment.

Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in the Waste Handling or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Wastes", EPA SW-846, methods 8082A, 8000C.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. NELAC/TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Analytes and reporting limits for the analysis of PCBs by 8082A.

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-050-rev.13	1. Section 2.1: removed specific method references for extraction procedures. 2. Section 3.1: created a table for reporting limits of each matrix and added 1262, 1268 and Total PCBs. 3. Table 7.1: revised hold time to extraction to 6 months. 4. Table 8.1: updated equipment and columns information. 5. Table 9.3: updated standards in use and storage conditions. 6. Section 9.2.3: updated standards used and preparation. 7. Section 10.2.2: clarified that single points should be analyzed before ICAL. 8. Section 10: removed quadratic curve fit criteria. 9. Section 11: removed calculations for average and linear curve fits. 10. Section 14: clarified method modifications.	10Aug2015
S-IN-O-050-rev.14	1. Converted to 27-section format. 2. Section 5: moved table of reporting limits to Table 1 attachment. 3. Table 7.1: revised storage temperature format. 4. Section 9.1: updated LTM column information. 5. Section 10.2.3: updated standard preparation to reflect current procedures. Added intermediate combined single point standards. 6. Section 14: corrected equations for aqueous and solid samples to put them in like terms with instrument output. Added equations for oil and wipe samples. 7. Section 19: added modification for combination of single point Aroclors. 8. Section 25.3: added years 2003 and 2009 to TNI reference. 9. Section 26: added reference to Table 1. 10. Table 1: added table for reporting limits.	27Jul2017
S-IN-O-050-rev.15	1. Section 8.1: removed reference to Glossary section of QAM. 2. Section 10.2.1: removed reference to LCS and MS/MSD standards. 3. Section 11.3: added language for example chromatograms and preferred Aroclor peaks used to calibration. 4. Section 12.4.5: added language and a list of tools to help in the identification of Aroclors in a complex matrix. 5. Section 12.5.3: added language to help in the quantitation of Aroclors in a complex matrix. 6. Section 18.1: updated MDL language to new EPA procedure.	7Mar2018
S-IN-O-050-rev.16	1. Table 7.1: updated collection per sample for aqueous samples to reflect RVE. 2. Section 9.1: updated GC columns. 3. Section 10.2.3.2: added lower CAL1 to example ICAL. 4. Section 25.3: added NELAC to reference.	26Apr2018

Table 1: Analytes and Reporting limits for PCBs analyzed by 8082A

Analyte	CAS Number	Water Reporting Limit	Soil Reporting Limit	Oil Reporting Limit	Wipes Reporting Limit
PCB-1016 (Aroclor 1016)	12674-11-2	0.1 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB-1221 (Aroclor 1221)	11104-28-2	0.2 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB-1232 (Aroclor 1232)	11141-16-5	0.1 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB-1242 (Aroclor 1242)	53469-21-9	0.1 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB-1248 (Aroclor 1248)	12672-29-6	0.1 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB-1254 (Aroclor 1254)	11097-69-1	0.1 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB-1260 (Aroclor 1260)	11096-82-5	0.1 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB-1262 (Aroclor 1262)	37324-23-5	0.1 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB-1268 (Aroclor 1268)	11100-14-4	0.1 ug/L	100 ug/kg	1 mg/kg	0.5 total ug
PCB, Total (Aroclor)	1336-36-3	0.2 ug/L	100 ug/kg	1 mg/kg	0.5 total ug

ATTACHMENT C-18

SEPARATORY FUNNEL EXTRACTION
PACE, INDIANAPOLIS

Pace Analytical Services, LLC
7726 Moller Road
Indianapolis, IN 46268
Phone: 317.228.3100
Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

SEPARATORY FUNNEL EXTRACTION
REFERENCE METHOD: EPA SW-846 METHOD 3510C

SOP NUMBER:	S-IN-O-054-rev.18
EFFECTIVE DATE:	July 23, 2018
SUPERSEDES:	S-IN-O-054-rev.17

APPROVAL

General Manager — July 16, 2018 Date

Quality Manager — July 9, 2018 Date

Department Manager — July 16, 2018 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for extracting non-volatile and semi-volatile organic compounds from groundwater and surface water samples in a separatory funnel while meeting the requirements specified in SW-846 Method 3510C.

2. Summary of Method

2.1. A measured volume of sample~~,~~ normally about 1 liter, is serially extracted with solvent in a separatory funnel. Reduced sample volumes may be used providing that the ratio of sample to solvent remains consistent with the ratio indicated in Method 3510C. Reduced sample volume extraction may also be referred to as Reduced Volume Extraction (RVE). Some extractions also require the monitoring and adjusting of the pH of the sample. The extract is separated from the sample and is concentrated, followed by cleanup, if necessary, or analysis.

3. Scope and Application

3.1. Applicable compounds, volumes/weights utilized, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of separatory funnel equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This procedure is for extracting water insoluble or slightly water soluble organic compounds from groundwater, surface water and other aqueous samples using methylene chloride as the extraction solvent.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Solvents, reagents and glassware can all contribute to compound artifacts or raised baselines; both conditions that can affect chromatography. Analyzing method blanks is therefore crucial in determining the presence of contaminants.

6.2. Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L, 125mL widemouth, or equivalent.	None	Cool to ≤6°C	Samples must be extracted within 7 days of collection date and extracts must be analyzed within 40 days of extraction date. Samples for **PCB** analysis must be extracted within 6 months of collection date and extract must be analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
N-EVAP concentrator	Organomation	Or equivalent equipment
Zymark concentrator (2) and glassware	Zymark	Or equivalent equipment
Shaker Tables	Glass-Col	Or equivalent equipment

9.2. General Supplies

Item	Description
Separatory Funnels	2L or 125mL, Teflon, with PTFE stopcocks and Teflon lids or equivalent
Glass beakers	400mL Pyrex or equivalent
Autosampler vials	~2mL, clear glass with aluminum crimp-top seals
Micro-syringes	Various sizes
Glass funnels	
Glass wool	
Graduated cylinders	Glass, Class A
Kuderna-Danish Concentrator Sets	250mL or 500mL flask with 10mL concentrator tube and 3-ball Snyder column
Heated water bath	Temperature controlled
Boiling Chips	Teflon or equivalent
Pasteur pipettes	For testing sample pH
pH paper	pH range 1-12
Glass stirring rods	For breaking up emulsions
Glass tubes	Disposable, 20x150mm or equivalent
Filter paper	For filtration of extract
Pipettes	Volumetric, Class A, various sizes

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Sodium Sulfate	Anhydrous, granular 10-60 mesh, meets ACS specs or equivalent. Rinse thoroughly with methylene chloride and allow it to dry prior to use.
Methylene Chloride	Extraction solvent, pesticide grade or equivalent
Acetone	Extraction solvent, pesticide grade or equivalent
Hexane	Exchange solvent, pesticide grade or equivalent
Sulfuric acid solution (1:1)	Reagent grade
Sodium Hydroxide solution (10N)	Dissolve 400g sodium hydroxide pellets into 1L of reagent water or purchase premade

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for preparing LCS, MS, and MSD samples and for spiking surrogate into all samples.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogates are added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains all target analytes.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock BNA RVE spike standard	1. Restek; catalog # 31004, B/N spike; 1000ug/mL, or equivalent 2. Restek; catalog # 31014, Acid spike; 2000ug/mL, or equivalent 3. Restek; catalog # 561763, Custom PAH spike; 5000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working BNA RVE spike standards	Refer to Section 10.2.3.1	Good for 6 months from preparation date	Refrigerate
Stock/Working BNA RVE surrogate standard	O2si; catalog # 110004-83-1L; 100ug/mL, or equivalent. Use 100uL for each BNA RVE.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock Full-list BNA RVE spike standard	1. SVOA Mega Mix; Restek; cat#31850; 1000ug/mL, or equivalent 2. 8270 Mix 1; Restek; cat#572178, 2000ug/mL, or equivalent 3. 8270 Mix 2: Restek; cat#572448, 2000ug/mL, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working Full-list BNA RVE spike standard	Refer to Section 10.2.3.2	Good for 6 months from preparation date	Refrigerate

Standard Type	Description	Expiration	Storage
Stock PCB RVE spike standard	Restek; catalog #32039, Aroclors 1016/1260; 1000ug/mL, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PCB RVE spike standard	Refer to Section 10.2.3.3	Good for 6 months from preparation date	Refrigerate
Stock PCB/8081 RVE surrogate standard	Restek; catalog#32457, TCMX/DCB mix; 200ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PCB/8081 RVE surrogate standard	Refer to Section 10.2.3.4	Good for 6 months from preparation date	Refrigerate
Stock 8081 RVE spike standard	Restek; catalog #32292, 8-80ug/mL of each compound, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8081 RVE spike standard	Refer to Section 10.2.3.5	Good for 6 months from preparation date	Refrigerate
Stock DRO spike standard	Restek; catalog # 31258, Diesel #2 standard; 50,000ug/mL, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO spike standard	Refer to Section 10.2.3.7	Good for 6 months from preparation date	Refrigerate
Stock DRO surrogate standard	Restek; catalog # 31487, Pentacosane; 10,000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working DRO surrogate standard	Refer to Section 10.2.3.8	Good for 6 months from preparation date	Refrigerate
Stock PAH-SIM RVE spike standard	Restek; catalog # 31622, Cal. Mix 5; 2000ug/mL of each compound, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PAH-SIM RVE spike standard	Refer to Section 10.2.3.9	Good for 6 months from preparation date	Refrigerate
Stock PAH-SIM RVE surrogate standard	Restek; catalog # 31062, B/N surrogate; 5000ug/mL of each compound, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working PAH-SIM RVE surrogate standard	Refer to Section 10.2.3.10	Good for 6 months from preparation date	Refrigerate
Stock/Working Scan/SIM Combo RVE spike standard	O2si, catalog #114072-06, 10-100ug/mL or equivalent. Use 100uL for the RVE LCS, MS, and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Freeze after opening.
Stock/Working Scan/SIM Combo RVE surrogate spike standard	O2si, catalog #114071-06; 10-100ug/mL or equivalent. Use 100uL for the RVE LCS, MS, and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Freeze after opening.
Stock TCLP BNA RVE spike standard	1. Restek; catalog # 31028, TCLP B/N spike; 2000ug/mL of each compound, or equivalent 2. Restek; catalog # 31027, TCLP Acid spike; 2000ug/mL of each compound, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working TCLP BNA RVE spike standard	Refer to Section 10.2.3.11	Good for 6 months from preparation date	Refrigerate

Standard Type	Description	Expiration	Storage
Stock 8141 spike standard	Ultra; catalog #CUS-12835, 100ug/mL of each compound or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock Dichlorvos spike standard	Ultra; catalog #PST-380H1000, 1000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8141 spike standard	Refer to Section 10.2.3.12	Good for 2 months from preparation date	Refrigerate
Stock 8141 surrogate standard	AccuStandard; catalog #M-507-1S-10X, Triphenylphosphate 5000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8141 surrogate standard	Refer to Section 10.2.3.13	Good for 2 months from preparation date	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1. Working BNA RVE Spike Standard Preparation

Dilute 2.5mL of the stock Acid spike standard (2000ug/mL), 5mL of the stock B/N spike standard (1000ug/mL) and 1mL of the stock Custom PAH spike (5000ug/mL) to 50mL with acetone for a final concentration of 100ug/mL. Add 100uL of this working standard to each BNA RVE LCS, MS and MSD.

10.2.3.2. Working Full-list BNA RVE Spike Standard Preparation

Dilute 5mL of Stock Mega Mix(1000ug/mL), 2.5mL of Stock Mix #1 (2000ug/mL) and 2.5mL of Stock Mix #2 (2000ug/mL) to 50mL in acetone for a final concentration of 100ug/mL. Add 100uL of this working full-list spike standard to each BNA RVE LCS, MS, and MSD.

10.2.3.3. Working PCB RVE Spike Standard Preparation

Dilute 100uL of the Stock PCB standard (1000ug/mL) to 1000mL with acetone for a final concentration of 1ug/mL. Add 500uL of this working spike to each PCB RVE LCS, MS and MSD.

10.2.3.4. Working PCB/8081 RVE Surrogate Standard Preparation

Dilute 5mL of Stock 8081/PCB Surrogate Standard (200ug/mL) to 400mL in acetone for a final concentration of 2.5ug/mL. Add 100uL of this working standard to each PCB/8081 RVE sample, Method Blank, LCS, MS and MSD.

10.2.3.5. Working 8081 RVE Spike Standard Preparation

Dilute 5mL of 8081 Stock Spike Standard (8-80ug/mL) to 20mL in acetone for a final concentration of 2-20ug/mL. Add 50uL of this working spike standard to each 8081 RVE LCS, MS and MSD.

10.2.3.6. Working DRO Spike Standard Preparation

Dilute 10mL of the stock DRO surrogate standard (50,000ug/mL) to 200mL with acetone for a final concentration of 2500ug/mL. Add 1mL of this working spike to each DRO/ERO/OHIO MOD LCS, MS and MSD.

10.2.3.7. Working DRO Surrogate Standard Preparation

Dilute 7.5mL of the stock DRO surrogate standard (10,000ug/mL) to 500mL with acetone for a final concentration of 150ug/mL. Add 1ML of this working surrogate to each DRO/ERO/Ohio mod sample, Method Blank, LCS, MS and MSD.

10.2.3.8. Working PAH-SIM RVE Spike Standard Preparation

Dilute 5mL of the stock PAH-SIM RVE spike standard (2000ug/mL) to 200mL with acetone for a final concentration of 50ug/mL. Add 20uL of this working spike to each PAH-SIM RVE LCS, MS and MSD.

10.2.3.9. Working PAH-SIM RVE Surrogate Standard Preparation

Dilute 5.0mL of the stock PAH-SIM RVE surrogate standard (5000ug/mL) to 500mL with acetone for a final concentration of 50ug/mL. Add 20uL of this working surrogate to each PAH-SIM RVE sample, Method Blank, LCS, MS and MSD.

10.2.3.10. Working TCLP BNA RVE Spike Standard Preparation

Dilute 5mL of the stock TCLP acid spike standard (2000ug/mL) and 5mL of the stock TCLP B/N spike standard (2000ug/mL) to 100mL with acetone for a final concentration of 100ug/mL. Add 100uL of this working spike to each TCLP BNA RVE LCS, MS and MSD.

10.2.3.11. Working 8141 Spike Standard Preparation

Dilute 1mL of the Stock 8141 spike standard (100ug/mL) and 100uL of Stock Dichlorvos standard (1000ug/mL) to 5mL in acetone for a final concentration of 20000ug/L. Add 100uL of this working spike to each 8141 LCS, MS and MSD.

10.2.3.12. Working 8141 Surrogate Standard Preparation

Dilute 1mL of the Stock 8141 Surrogate Standard (5000ug/mL) to 50mL in acetone for a final concentration of 100ug/mL. Add 25uL of this working surrogate standard to each 8141 sample, Method Blank, LCS, MS and MSD.

11. Calibration

11.1. Not applicable to this SOP.

12. Procedures

12.1. Make sure that all glassware and Teflon separatory funnels used for this procedure have been properly washed. All washed glassware must be rinsed prior to use with acetone to remove residual water and rinsed with methylene chloride to remove any residual contaminants.

12.2. Measure the initial pH of each sample using wide range pH paper by dipping a clean disposable Pasteur pipette into each sample and touching the pipette to a piece of pH paper. Record the initial pH in the extraction log.

12.3. A nominal sample volume of 100mL of aqueous sample is routinely extracted for Reduced Volume Extractions (RVE); otherwise, a nominal sample volume of 1L is used. Refer to Table 1 – Extraction

Conditions for more information. For samples expected to contain high concentrations of analytes, use a smaller aliquot of sample diluted to 1L or 100mL with reagent water.

12.4. For each extraction batch of 20 or fewer samples, prepare a method blank by placing 1L or 100mL of reagent water in to a labeled separatory funnel on the black separatory funnel racks, or on the trellis. The method blank will be used to check for contamination in the system.

12.5. For each extraction batch of 20 or fewer samples, prepare an LCS by placing 1L or 100mL of reagent water in to a labeled separatory funnel on the black separatory funnel rack, or on the trellis. Spike the reagent water with the appropriate amount of spike solution. The LCS will be used to determine the efficiency of the extraction method.

12.6. If the entire contents of the sample bottle are to be extracted, mark the level of sample on the outside of the bottle for later volume determination. If only an aliquot of the sample is needed, measure the desired volume using a Class A graduated cylinder and record the volume in mLs. Transfer the sample from the sample bottle or graduated cylinder into a clean separatory funnel on the rack or the trellis.

12.7. For each extraction batch of 20 or fewer samples, prepare a matrix spike (MS) and matrix spike duplicate (MSD) in separate, labeled separatory funnels whenever available sample volume allows.

12.8. Add the appropriate **surrogate** solution to each method blank, sample, LCS, MS and MSD. Add the appropriate **spiking** solution to the LCS, MS and MSD. Refer to the standard preparation log and the sample preparation log for details regarding the appropriate surrogate and spiking solutions and volumes to be used for each method.

12.9. Adjust the sample pH, if necessary, to the pH indicated in Table 1 using 1:1 Sulfuric Acid or 10N Sodium Hydroxide. The pH is checked by dipping the tip of a disposable glass pipet into each well-mixed sample and placing the tip onto the pH paper to obtain a pH measurement.

12.10. Rinse the sample bottle (or graduated cylinder) with the first 60mL portion of extraction solvent for a 1L sample or the first 6mL portion of extraction solvent for a 100mL sample and transfer the rinsate to the separatory funnel. If the sample was transferred to the separatory funnel directly from the sample bottle, refill the bottle to the mark made in Section 12.6 with water and then measure the volume of sample that was in the bottle using a Class A graduated cylinder and record the volume in mLs as the initial sample volume.

12.11. Seal the separatory funnels with Teflon lids and shake for two minutes with periodic venting. This can be done manually or on an automatic shaker. When using the automatic shaker that holds separatory funnels in an inverted position, the stopcocks of the separatory funnels can be left open as an alternative to periodic venting. **NOTE**: Methylene chloride may cause excessive pressure in the separatory funnel. It is recommended to shake slightly and vent before placing funnels on an automatic shaker.

12.12. Return the 2L separatory funnels to the trellis and allow the solvent layer to separate from the aqueous phase. The 125mL separatory funnels remain in the automatic shaker for draining. If an excessive emulsion is present in the solvent layer, it can be broken up manually by using a clean glass stirring rod. If this is not successful, the sample can be drained into a clean secondary container (i.e. VOA vial) and transferred to a centrifuge tube. The extract can be centrifuged and then decanted into the drying funnel.

12.13. Drain the solvent layer through a drying funnel consisting of a clean funnel containing a plug of clean glass wool topped with a portion of clean sodium sulfate. The solvent should be collected in labeled beakers, labeled KD glassware, or labeled glass tubes.

12.14. Repeat the extraction two additional times with 60mL or 6mL aliquots of solvent added for each

extraction. Drain the solvent through the drying funnel after each extraction. After the final solvent extraction has been collected, rinse the funnel with methylene chloride and remove the drying funnel.

12.15. If extraction at a secondary pH is required, add acid or base as necessary and serially extract the sample, as described in Sections 12.11-12.14, at the adjusted pH. Collect all sample extract fractions together for concentration. Refer to Table 1 for extraction conditions.

12.16. **Concentration procedure for 8270 BNA, 8270 Scan/SIM Combo, and TCLP samples:** Pour the extract into a labeled Kuderna-Danish concentrator with a concentrator tube securely attached. Add one or two clean boiling chips to the KD flask and attach a 3-ball Snyder column. Place the KD apparatus on a hot water bath so that the flask is partially immersed in the water. At the proper rate of distillation, the balls of the Snyder column should actively chatter but the chambers should not flood with solvent. Adjustment of the angle of the apparatus and the water temperature may be necessary to make the boiling more efficient. When the apparent volume of the extract reaches 4-6mL, remove the apparatus from the water bath and allow it to cool. Once cooled, carefully disassemble the KD apparatus rinsing each joint into the concentrator tube with a small amount of extraction solvent. Place the concentrator tube into the N-Evap and further concentrate the extract until the apparent volume is slightly below 1mL. Continue to Section 12.20.

12.17. **Concentration of PAH-SIM LVE samples:** Pour the extract into a labeled 20x150mm glass tube and place the tube on the N-Evap concentrator in a warm water bath (about 40^{o}C) and evaporate the solvent volume using a gentle stream of nitrogen. The tube should be positioned so that water will not condense into the sample and the sample should not be allowed to go below 0.5mL, this could lead to losses of semi-volatile compounds.

12.18. **Concentration procedure for all other samples:** Pour the entire sample extract into a labeled Zymark extractor tube and place in the Zymark concentrator apparatus. Adjust the settings per manufacturer's instructions. When the apparent volume is slightly below the intended final volume, remove the concentrator from the apparatus and allow it to cool.

12.19. If a solvent exchange is required, see Table 1, add 50mL of the exchange solvent to the Zymark tube. Concentrate the extract to slightly below the intended final volume, remove from the water bath and allow it to cool.

12.20. If further concentration is necessary for any sample extract, nitrogen blowdown can be performed. For this procedure, place the concentrator tube on the N-Evap concentrator in a warm water bath (about 40^{o}C) and evaporate the solvent volume using a gentle stream of nitrogen. The tube should be positioned so that water will not condense into the sample and the sample should not be allowed to go below 0.5mL, this could lead to the loss of semi-volatile compounds.

12.21. Prepare a calibrated vial by volumetrically dispensing the required volume of the solvent being used into a vial and securely capping the vial to eliminate evaporation. The calibrated vial must be prepared daily using a Class A pipet. Quantitatively transfer the sample extract from the Zymark tube or concentrator tube to a vial. Bring the sample extract in the vial to the required final volume listed in Table 1 by visually comparing the sample extract vial volume to the calibrated vial volume. Securely cap the sample extract vial. Store all extracts in the appropriate storage cooler. For extracts that will not concentrate to the usual final volume, use the procedure described above to bring the extract to the next higher practical volume for which a calibrated vial can be prepared.

12.22. Refer to appropriate cleanup SOPs if extract cleanup is required.

13. Quality Control

13.1. Refer to the SOP for the determinative method for batch quality control acceptance criteria and corrective actions.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. Analysts performing this method must document acceptable accuracy and precision by passing a demonstration of capability study (DOC) on an annual basis.

19. Method Modifications

19.1. Spikes not added to graduated cylinder or sample bottle but instead added to the separatory funnel.

20. Instrument/Equipment Maintenance

20.1. Refer to manufacturer's instructions.

21. Troubleshooting

21.1. Refer to manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3510 "Separatory Funnel Extraction".

25.2. Pace Analytical Quality Manual; latest revision.

25.3. NELAC/TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Extraction Conditions

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-054-rev.13	1. Effective date added to cover page and body header. 2. Table 9.1: added preparation of sodium sulfate for use by solvent rinsing. 3. Section 11: revised to add new process of determining sample volume prior to extraction using Class A graduated cylinder. Removed language referring to the previous method of sample volume determination. Reordered sections to reflect the addition of surrogates and spike prior to the addition of solvent. Added a reference to extract cleanup SOPs. 4. Section 13.1: added optional LOD/LOQ verification. 5. Section 14: removed previous method modification of determining sample volume post extraction.	03Nov2013
S-IN-O-054-rev.14	1. Section 3.2: added Pesticides. 2. Section 9.2.2: added detail for 8081 and 8141 standards 3. Section 9.2.3: added detail for 8081 and 8141 standards 4. Table 1: added detail for 8081 and 8141 extractions.	28Feb2015
S-IN-O-054-rev.15	1. Table 7.1: revised holding time for extraction of samples for PCB analysis. 2. Table 9.3: updated storage conditions for standards and added Combo LVE spike. 3. Section 12: removed equations for LCS, RSD, MS. 4. Section 13: removed MDL study requirement.	02Sep2015
S-IN-O-054-rev.16	1. Converted to 27 section format. 2. Table 7.1: updated storage temperature format. 3. Table 10.3: updated standard descriptions. 4. Section 10.2.3: updated standard preparation where needed. 5. Section 12: separated instructions for method blank, LCS and MS/MSD. Specified "first portion" of solvent used to rinse cylinder or bottle. 6. Section 19: removed modification for some samples getting two extractions at each pH. 7. Section 25.3: added years 2003 and 2009 to TNI reference. 8. Table 1: removed columns for # of extractions.	05Sep2017

S-IN-O-054-rev.17	1. Section 2.1: changed LVE reference to RVE for reduced volume extraction. 2. Section 10.2.1: removed reference to calibration standards. 3. Table 10.3: added Dichlorvos stock standard, changed LVE to RVE in all instances, and removed references to full-volume analysis where needed. 4. Section 10.2.3: updated standard preparation procedures where needed. 5. Section 12: changed LVE to RVE in all instances. Changed ring stand language to separatory funnel rack or trellis. Added language to require a method blank, LCS and MS/MSD for each extraction batch of 20 or fewer samples. Added an alternative to periodic venting for inverted separatory funnels on certain automatic shakers. 6. Section 13: removed table 13.1 and referred to SOP for the determinative method. 7. Section 25.3: added years 2003 and 2009 to TNI reference. 8. Table 1: added nominal sample volume column and changed LVE to RVE in all instances.	26Apr2018
S-IN-O-054-rev.18	1. Section 9.2: added 250mL KD as an option. 2. Table 10.3: updated DRO standards. 3. Section 12.6: added procedure for marking bottle for later initial sample volume determination. 4. Section 12.10: added procedure for initial sample volume determination when entire bottle content is used. 5. Section 25.3: added NELAC to reference. 6. Table 1: updated DRO information.	8Jul2018

Table 1 – Extraction Conditions

Determinative Method	Nominal Sample Volume Extracted	Initial Extraction pH	Secondary Extraction pH	Extraction Solvent	Exchange Solvent	Final Extract Volume (mL)
8015 DRO (includes ERO and Ohio mod)	1000mL	<2	N/A	Methylene Chloride	N/A	1
8081 OC PEST RVE	100mL	5-9	N/A	Methylene Chloride	Hexane	10
8082 PCB RVE	100mL	5-9	N/A	Methylene Chloride	Hexane	10
8141 OP PEST	1000mL	As received	N/A	Methylene Chloride	Hexane	10
8270 PAH-SIM RVE	100mL	>11	N/A	Methylene Chloride	N/A	1
8270 BNA RVE	100mL	<2	>11	Methylene Chloride	N/A	1
8270 BNA Scan/SIM Combo RVE	100mL	<2	>11	Methylene Chloride	N/A	1
8270 TCLP BNA RVE	10mL	<2	>11	Methylene Chloride	N/A	1

ATTACHMENT C-19

THE DETERMINATION OF SEMI-VOLATILE COMPOUNDS BY GC/MS
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SEMI-VOLATILE COMPOUNDS BY GC/MS
REFERENCE METHOD: EPA SW-846 METHOD 8270C

SOP NUMBER:	S-IN-O-068-rev.15
EFFECTIVE DATE:	November 27, 2017
SUPERSEDES:	S-IN-O-068-rev.14

APPROVAL

General Manager — November 22, 2017 Date

Quality Manager — November 22, 2017 Date

Department Manager — November 22, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of semi-volatile organic compounds in sample extracts while meeting the requirements specified in EPA method 8270C.

2. Summary of Method

2.1. Semi-volatile compounds are introduced into a gas chromatograph by injection of a sample extract onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes that are then detected with a mass spectrometer. Identification of the analytes is made by comparing their mass spectra with spectra of known standards. Quantitation is accomplished by comparing the response of a major ion relative to the internal standard response using a multi-point calibration curve.

2.2. Method 8270C provides chromatographic conditions for the detection of semi-volatile compounds in organic extracts. Aqueous samples are extracted using SW-846 method 3510 Separatory Funnel Extraction or other applicable method. Solid samples are extracted using SW-846 method 3546 Microwave Extraction or other applicable method.

3. Scope and Application

3.1. This method can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, phenols, and nitrophenols.

3.2. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent extraction and exhibits poor chromatographic behavior. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosdiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3.3. The following compounds are analyzed using this procedure but are not 8270C listed compounds: Carbazole, Biphenyl (Diphenyl), Caprolactam, Atrazine, Benzaldehyde, Diethyl Aniline, 2,3-Dichloroaniline, 1-Methylnaphthalene and 4-Chlorobenzotrifluoride.

3.4. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.5. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS equipment and the interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This procedure is applicable to extracts prepared from many types of solid waste, soils, air sampling media, and water samples.

5. Limits of Detection and Quantitation

5.1. The list of compounds and reporting limits analyzed for method 8270C is found in Table 1. Other compounds may be reported upon completion of appropriate validation procedures. Refer to the LIMS for method detection limits.

6. Interferences

6.1. Glassware for the preparatory steps for this method should be thoroughly cleaned and rinsed. Soap products can leave phthalates on the glassware that may appear in the analytical data. Hits for phthalates should be closely scrutinized and continuous hits should warrant checking on the glassware cleaning procedure.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L or 125mL widemouth, or equivalent.	None required	Cool to $\leq 6^{o}C$	Samples must be extracted within 7 days of collection date and analyzed within 40 days of extraction date.
Solid	> 200 grams in 4oz or 8oz glass jar	None required	Cool to $\leq 6^{o}C$	Samples must be extracted within 14 days of collection date and analyzed within 40 days of extraction date.

Samples and sample extracts must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation

Equipment	Description / Comments
Gas Chromatographs	Hewlett Packard/Agilent 6890/7890 or equivalent system
Data Systems	Hewlett Packard/Agilent Chemstation or equivalent system
Autosamplers	Hewlett Packard/Agilent or equivalent system
Mass Spectrometers	Hewlett Packard/Agilent 5973/5975. Or equivalent system.

9.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Restek	Rxi-5 Sil MS	30m x 0.25mm or equivalent column

9.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials (clear vials)	Supelco or equivalent
Autosampler vials	1.8mL clear vials	Or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	Pesticide grade or equivalent
Acetone	Pesticide grade or equivalent
Methanol	Pesticide grade or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions and vendors

Standard	Description	Comments
Tuning Standard	Standard used to tune the mass spectrometer	DFTPP solution
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains method required spiking compounds, at a minimum, and is used for spiking MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

10.2.2. Details and Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock 8270 Mega Mix calibration standard	Restek; catalog #31850, 1000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 Mix #1 calibration standard	Restek; catalog #572178, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 Mix #2 calibration standard	Restek; catalog #572448, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.

Standard Type	Description	Expiration	Storage
Intermediate 8270 Calibration standard	Refer to Sections 10.2.3.1	Expires 6 months from date of preparation.	Refrigerate
Working 8270/Intermediate 8270 LVE calibration standards	Refer to Section 10.2.3.2	Expires 6 months from date of preparation	Refrigerate
Working 8270 LVE calibration standards	Refer to Sections 10.2.3.3	Expires 6 months from date of preparation.	Refrigerate
Stock 8270 ICV standard-Stock A	NSI; catalog #C-701, 1000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-Stock B	NSI; catalog #C-402, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-Stock C	NSI; catalog #C-639, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-Stock D	NSI; catalog #541H, 2000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock 8270 ICV standard-Lilly ICV	Cresent; catalog #CCS-2579, 1000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270/Intermediate 8270 LVE ICV standard	Refer to Section 10.2.3.4	Expires 6 months from date of preparation.	Refrigerate
Working 8270 LVE ICV standard	Refer to Section 10.2.3.5	Same expiration date as Intermediate 8270 LVE ICV standard	Refrigerate
Stock internal standards	Restek; catalog # 31006; 4000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270 LVE internal standards	Refer to Section 10.2.3.6	Expires 6 months from date of preparation.	Refrigerate
Stock Surrogate standards	Accustandard; catalog #M-8270-SS; 4000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Stock DFTPP Tuning Standard	O2si; catalog #113000-01, 50ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
Working 8270 DFTPP LVE Tuning Standard	Refer to Section 10.2.3.7	Expires 6 months from date of preparation.	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1 Intermediate 8270 Calibration Standard Preparation

Dilute 1000uL of Stock 8270 MegaMix calibration standard (1000ug/mL), 500uL of Stock 8270 Mix #1 (2000ug/mL), 500uL of Stock 8270 Mix #2 (2000ug/mL) and 250uL of Stock Surrogate standard (4000ug/mL) to 5mL with Methylene Chloride for a final concentration of 200ug/mL

10.2.3.2 Working 8270/Intermediate 8270 LVE Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Int. 8270 Cal. Std. Amount	Final Volume in Methylene Chloride	Final Nominal Cal Std Concentration	Stock Internal Standard amount added to Cal. Std.	Internal Standard Concentration
8270 Cal Std 1	25uL	1000uL	5ug/mL	10uL	40ug/mL
8270 Cal Std 2	50uL	1000uL	10ug/mL	10uL	40ug/mL
8270 Cal Std 3	100uL	1000uL	20ug/mL	10uL	40ug/mL
8270 Cal Std 4	250uL	1000uL	50ug/mL	10uL	40ug/mL
8270 Cal Std 5	400uL	1000uL	80ug/mL	10uL	40ug/mL
8270 Cal Std 6	500uL	1000uL	100ug/mL	10uL	40ug/mL
8270 Cal Std 7	750uL	1000uL	150ug/mL	10uL	40ug/mL

10.2.3.3 Working 8270 LVE Calibration Standards

Prepare using the Working 8270/Intermediate 8270 LVE Calibration Standards from Section 10.2.3.2. The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate Cal. Std. ID from Section 10.2.3.2	Intermediate Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Internal Standard Concentration
8270 LVE Cal. Std 1	8270 Cal Std 1	100uL	1mL	0.5ug/mL	4ug/mL
8270 LVE Cal. Std 2	8270 Cal Std 2	100uL	1mL	1.0ug/mL	4ug/mL
8270 LVE Cal. Std 3	8270 Cal Std 3	100uL	1mL	2.0ug/mL	4ug/mL
8270 LVE Cal. Std 4	8270 Cal Std 4	100uL	1mL	5.0ug/mL	4ug/mL
8270 LVE Cal. Std 5	8270 Cal Std 5	100uL	1mL	8.0ug/mL	4ug/mL
8270 LVE Cal. Std 6	8270 Cal Std 6	100uL	1mL	10ug/mL	4ug/mL
8270 LVE Cal. Std 7	8270 Cal Std 7	100uL	1mL	15ug/mL	4ug/mL

10.2.3.4 Working 8270/Intermediate 8270 LVE ICV Standard Preparation

Dilute 50uL of 8270 ICV Standard-Stock A (1000ug/mL), 25uL of 8270 ICV Standard-Stock B (2000ug/mL), 25uL of 8270 ICV Standard-Stock C (2000ug/mL), 25uL of 8270 ICV Standard-Stock D (2000ug/mL), 50uL of 8270 ICV Standard-Lilly ICV (1000ug/mL), and 25uL of Stock Surrogate Standard (4000ug/mL) to 1mL with methylene chloride for a final concentration of 50-100ug/mL.

10.2.3.5 Working 8270 LVE ICV Standard Preparation

Dilute 100uL of the Intermediate 8270 LVE ICV Standard (50-100ug/mL) to 1mL with methylene chloride for a final concentration of 5-10ug/mL.

10.2.3.6 Working 8270 LVE Internal Standards Preparation

Dilute 300uL of the Stock internal standards (4000ug/mL) to 3mL with methylene chloride for a final concentration of 400ug/mL.

10.2.3.7 Working DFTPP LVE Tuning Standard Preparation

Dilute 200uL of the Stock DFTPP Tuning Standard (50ug/mL) to 1mL with methylene chloride for a final DFTPP concentration of 10ug/mL.

11. Calibration

11.1. DFTPP Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned using a 50ng injection of DFTPP. Analysis must not begin until the tuning criteria are met. Use the DFTPP mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected. The 12-hour window during which standards and samples may be analyzed begins with the injection of DFTPP. All subsequent standards, samples MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

Mass (m/z)	Ion Abundance criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

If the DFTPP ratios do not meet the criteria, reanalyze the DFTPP tune. If the DFTPP still fails the criteria, autotune adjustment, instrument maintenance, and/or preparation of new standards must be considered.

The mass spectrum of DFTPP may be obtained by averaging three scans, the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using this approach and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak. Alternatively, the analyst may use other approaches suggested below:

1. A single scan within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.
2. An average of multiple scans within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.

11.2. Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

11.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

11.4. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

11.5. The %RSD should be should be ≤15% for each target analyte. However, the %RSD for each individual Calibration Check Compound (CCC) must be ≤30%. If the RSD of any CCC is >30%, then the chromatographic system is too reactive and instrument maintenance or preparation of new standards may be necessary before attempting recalibration. The CCCs are:

Base/Neutral Fraction	**Acid Fraction**
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

11.6. System Performance Check Compounds (SPCCs) are checked for a minimum average response factor (RF_{avg}) to determine potential instability and/or degradation caused by deterioration of instrument conditions or standard material. The minimum RF_{avg} for the semivolatile SPCCs are as follows:

N-nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Instrument maintenance or preparation of new standards may be necessary. The SPCC criteria must be met for sample analysis to begin.

11.7. If the percent relative standard deviation (%RSD) of the RFs for a compound is ≤15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

11.8. If the % RSD for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure

of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.9. Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be $\geq$ 0.99. Refer to Method 8000C for additional information regarding calibration.

11.10. Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.11. Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably. Additionally, examination of signal-to-noise ratio of analytes in low-level standards may be a useful diagnostic tool to monitor instrument performance. Signal-to-noise ratio is defined as the ratio of the analyte signal to the noise measured on a blank. It is recommended but not required that the signal-to-noise ratio be at least three to confirm detection.

11.12. Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. The ICV recoveries are evaluated against a default acceptance range of 70-130% recovery. Alternative acceptance limits may be appropriate for some compounds.

11.13. ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

11.14. Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a DFTPP tune verification as described in Section 10.1, followed by a Continuing Calibration Verification

(CCV) standard.

11.15. If the % difference (%D) or % Drift for each CCC is ≤20%, then the initial calibration is assumed to be valid. The response factors for all SPCCs in the CCV standard must meet the criteria in Section 10.6. The % difference (%D) or % Drift for each non-CCC compound should be ≤40%. If non-CCC compounds fail to meet this criterion, the concentrations above the reporting limit in associated samples must be qualified as estimated.

11.16. The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

11.17. CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12. Procedures

12.1. All sample extracts must be analyzed at room temperature and the system must be tuned and calibrated as per Section 10, and free of contamination before samples are analyzed.

12.2. Gas Chromatography conditions: Configure the GC/MS per manufacturer's instructions.

12.3. The 1mL extract obtained from sample preparation for 8270 should be fortified with 10uL of the Stock 8270 internal standard (4000ug/mL) just prior to analysis such that 40ng of internal standard is injected onto the column. The 1mL extract obtained from sample preparation for 8270 LVE should be fortified with 10uL of the Working 8270 LVE internal standard (400ug/mL) just prior to analysis such that 4ng of internal standard is injected onto the column. Analyze each 8270 extract by injecting 2uL onto the column. Analyze each 8270 LVE extract by injecting 4uL onto the column.

12.4. Qualitative Analysis: Compounds are identified as present when the following criteria are met:

12.4.1. The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

12.4.2. The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for characteristic ions.

12.4.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.5. Quantitative analysis: Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. Calculations are subject to change based on the data reduction software used. Extract concentrations that exceed the upper calibration range must be diluted and reanalyzed or qualified as estimated. Additional internal standard must be added to the diluted extract to maintain the same concentration as the calibration standards.

12.6. Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

12.7. If the sample concentration exceeds the linear range of the analysis, the sample must be diluted and reanalyzed or reported as an estimated concentration.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits. Refer to Sections 13.2 and 13.3 for additional information.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) If only one surrogate fails and it is >10% recovery, re-extraction is not required but data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
As required by client only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed undiluted or more concentrated. The laboratory may only dilute a sample prior to reanalysis if matrix interference is present. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

13.2. The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS.

13.3. Allowable Marginal Exceedances: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken. The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: As allowed by client, the LCS shall be allowed to be outside the control limits but ≥10% for hexachlorocyclopentadiene, N-nitrosodimethylamine, pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, and 4-chloroaniline without corrective action. The LCS shall be allowed to be outside the control limits but >10% for up to four additional compounds, with the exception of any PAH, without corrective action.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_f)(D)}{(V_i)} \qquad \text{Solid Sample (ug/kg)} = \frac{(X_s)(V_f)(D)}{(W_s)}$$

Where:
X_s = Concentration of the analyte from the instrument, ug/mL
V_f = Final volume of extract, mL
D = Dilution factor of extract
V_i = Volume of aqueous sample extracted, L
W_s = Weight of solid sample extracted, kg

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

14.2. LCS equation

R = (C/S) * 100

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD calculations:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL study and/or LOD/LOQ verification must be conducted annually for each matrix per instrument.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Standards are purchased as certified stock solutions and not prepared from neat materials.

19.2. Microwave Method 3546 is used for the preparation of solid samples for analysis by 8270C.

19.3. Phenol-d5 is used as a surrogate instead of Phenol-d6.

19.4. Extract final volumes, volume of internal standards added to extracts, and volume of extract injected into the instrument may vary from those identified in Method 8270C.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846 Methods 8000C and 8270C.

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Table 1: Target Compounds and Reporting Limits

26.2. Table 2: Characteristic Ions of Target Compounds

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-068-rev.13	1. Cover page: added actual effective date. 2. Sections 10.8 and 10.9: removed SW-846 equations and made reference to Method 8000C. 3. Section 10.11: added as guidance for evaluation of ICAL standards. 4. Section 10.12: added that alternative limits may be appropriate for some ICV compounds. 5. Section 10.15: added criteria of <40% for non-CCC compounds in CCV. 6. Section 11.6: added as a reference to the Manual Integrations SOP. 7. Section 11.7: added to require over range samples be diluted and reanalyzed or qualified as estimated. 8. Sections 11.8-11.10: replaced Target equations with SW-846 equations. 9. Table 12.1: corrected references in LCS acceptance criteria section and removed client-specific reference in internal standard section. 10. Section 12.3 Note: removed client-specific reference. 11. Section 13.1: added optional LOD/LOQ verification. 12. Table 1: updated some soil RLs.	01Nov2013
S-IN-O-068-rev.14	1. Converted to SOT format with 27 sections. 2. Cover page: changed phone number and revised document control format. 3. Section 3: added a list of compounds that are analyzed but not listed in the method. 4. Section 9.2: updated column details 5. Table 10.3: updated standard details and storage conditions. 6. Section 10.2.3: updated several standard preparation procedures. 7. Section 12: removed calculations for curve fit types. 8. Updated Tables 1 and 2.	07Dec2015
S-IN-O-068-rev.15	1. Table 7.1: revised storage temperature format. 2. Table 10.3: updated to current standard IDs. 3. Section 10.2.3: revised some recipes to match current practice. 4. Section 14.1: corrected equations to be in like terms with instrument output. 5. Section 25.3: added years 2003 and 2009 to TNI reference. 6. Table 1: changed 5 ug/L RLs to 10 ug/L.	20Nov2017

Table 1: Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L)	RL soil (ug/kg)
Phenol	10	330
Bis (2-chloroethyl) ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
Benzyl Alcohol	20	660
1,2-Dichlorobenzene	10	330
2-Methylphenol (o-Cresol)	10	330
Bis (2-chloroisopropyl)ether	10	330
3&4-Methylphenol (m&p-Cresol)	20	660
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Benzoic Acid	50	1600
Bis(2-chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	20	660
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol	20	660
1-Methylnaphthalene	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	10	330
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1600
Dimethyl phthalate	10	330
Acenaphthene	10	330
Acenaphthylene	10	330
2,4-Dinitrophenol	50	1600
4-Nitrophenol	50	1600
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	50	1600
Diethyl phthalate	10	330
4-Chlorophenyl phenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	50	1600
4,6-Dinitro-2-methylphenol	50	1600
N-Nitrosodiphenylamine	10	330
4-Bromophenyl phenyl ether	10	330

Analyte	RL water (ug/L)	RL soil (ug/kg)
Hexachlorobenzene	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Anthracene	10	330
Di-n-butyl phthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butyl benzyl phthalate	10	330
3,3'-Dichlorobenzidine	20	660
Benzo(a)anthracene	10	330
Chrysene	10	330
Bis(2-ethylhexyl)phthalate	10	330
Di-n-octyl phthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	170
Indeno(1,2,3-cd)pyrene	10	330
Dibenz(a,h)anthracene	10	170
Benzo(g,h,i)perylene	10	330
N-Nitrosodimethylamine	10	330
Pyridine	10	1600
Benzidine	20	330
Acetophenone	10	330
2,6-Dichlorophenol	10	330
1,2-Diphenylhydrazine	10	330
2-Picoline	50	1600
1,2,4,5-Tetrachlorobenzene	10	330
1,3-Dinitrobezene	50	1600
2,3,4,6-Tetrachlorophenol	10	330

[1] Target Compounds and Reporting Limits are subject to change.

Table 2: Characteristic Ions of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Group 1 - 1,4-Dichlorobenzene-d4 (IS)	152	150, 115
N-Nitrosodimethylamine	42	74,44
Pyridine	79	52
2-Picoline	93	66, 92
4-Chlorobenzotrifluoride[3]	180	161,182
2-Fluorophenol (S)	112	64
Phenol-d5 (S)	99	71, 42
Benzaldehyde	77	105, 106
Phenol	94	65.66
Aniline	93	66, 65
Bis(2-Chloroethyl) ether	93	63.95
2-Chlorophenol	128	64,130
n-Decane[3]	57	43, 142
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
Benzyl alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 111
Bis(2chloro1methylethyl) ether[3]	45	77, 121
Bis(2-chloroisopropyl) ether	45	77, 121
3&4-methylphenol (m&p cresol)	108	107,77
Acetophenone	105	77, 120
N-Nitroso-di-n-propylamine	70	130, 101
Hexachloroethane	117	201, 199
Group 2 - Naphthalene-d8 (IS)	136	68
Nitrobenzene-d5 (S)	82	128, 54
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	122	107, 121
Bis(2-chloroethoxy) methane	93	95, 123
Benzoic Acid	105	122, 77
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 102
Apha-Terpineol	59	93, 121
2-Chloroaniline	127	65, 92
Hexachlorobutadiene	225	223, 227
Caprolactam	113	55, 56
Diethyl Aniline[3]	134	106, 77
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141, 115
1-Methylnaphthalene[3]	142	141, 115

Analyte	Primary Ion	Secondary Ion(s)
Group 3 - Acenaphthene-d10 (IS)	164	160, 162
Hexachlorocyclopentadiene	237	235, 272
1,2,4,5-Tetrachlorobenzene	216	179, 108
2,3-Dichloroaniline[3]	161	163, 90
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Fluorobiphenyl (S)	172	171
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
1,3-Dinitrobenzene	168	76, 50
2,6-Dinitrotoluene	165	63, 89
Acenaphthylene	152	150, 153
3-Nitroaniline	138	108, 92
Biphenyl (Diphenyl)	154	153, 152
Acenaphthene	153	154, 152
2,4-Dinitrophenol	184	154, 63
4-Nitrophenol	109	139, 65
2,4-Dinitrotoluene	165	63, 89
Dibenzofuran	168	139, 169
2,3,4,6-Tetrachlorophenol	232	131, 230
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluroene	166	165, 139
4-Nitroaniline	138	108, 65
Group 4 - Phenanthrene-d10 (IS)	188	80,94
4,6-Dinitro-2-methylphenol	198	51,105
N-Nitrosodiphenylamine	169	168, 167
Azobenzene[3]	77	182, 105
1,2-Diphenylhydrazine	77	105, 182
2,4,6-Tribromophenol (S)	330	332, 141
4-Bromophenyl-phenyl ether	248	250, 141
Hexachlorobenzene	284	142, 249
Atrazine	200	215, 202
Pentachlorophenol	266	264, 268
n-Octadecane[3]	57	43, 71
Phenanthrene	178	179, 176
Anthracene	178	176, 179
Carbazole[3]	167	166, 139
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 203
Benzidine	184	92, 185
Group 5 - Chysene-d12 (IS)	240	120, 236
Pyrene	202	101, 203
p-Terphenyl-d14 (S)	244	122, 212
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Bis(2-Ethylhexyl) phthalate	149	167, 279
Benzo(a)athracene	228	229, 226
Chrysene	228	226, 229

Analyte	Primary Ion	Secondary Ion(s)
Group 6 - Perylene-d12 (IS)	264	260, 265
Di-n-ocytlphthalate	149	279, 43
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluroanthene	252	253, 125
Benzo(a)pyrene	252	253,125
Indeno (1,2,3-cd)pyrene	276	138, 277
Dibenz (a,h) athracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277

[2] Target Compounds are subject to change.
[3] Compound is not listed in Method 8270C.

ATTACHMENT C-20

DETERMINATION OF ANIONS BY ION CHROMATOGRAPHY
PACE, INDIANAPOLIS

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STANDARD OPERATING PROCEDURE

DETERMINATION OF ANIONS BY ION CHROMATOGRAPHY

REFERENCE METHODS: EPA METHOD 300.0, REV. 2.1 AND EPA SW-846 METHOD 9056A

SOP NUMBER:	S-IN-O-170-rev.03
EFFECTIVE DATE:	February 12, 2018
SUPERSEDES:	S-IN-O-170-rev.02

APPROVAL

General Manager — February 8, 2018 Date

Quality Manager — February 7, 2018 Date

Department Manager — February 7, 2018 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of anions in aqueous and solid samples while meeting the requirements specified in EPA Method 300.0, Rev. 2.1 and SW-846 Method 9056A.

2. Summary of Method

2.1. Aqueous samples or solid sample extracts are introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

3. Scope and Application

3.1. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ion chromatograph systems and interpretation of associated data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This procedure is applicable to most drinking water, groundwater, surface water, wastewater, and solids.

5. Limits of Detection and Quantitation

5.1. The table below summarizes the anions that are routinely reported by this method and the current default reporting limits. Refer to the LIMS for method detection limits.

Anion	Reporting Limits-water, (mg/L)	Reporting Limits-soil, (mg/kg)
Bromide	0.05	0.5
Chloride	0.25	2.5
Fluoride	0.1	1
Iodide	0.5	5
Nitrate	0.05	0.5
Nitrite	0.05	0.5
Nitrate-Nitrite	0.1	1
Sulfate	0.25	2.5

6. Interferences

6.1. Interferences can be caused by substances with retention times that are similar to and that overlap those of the anions of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention time.

6.2. The water dip or negative peak that elutes near and can interfere with the fluoride peak can usually be eliminated by the addition of the equivalent of 1 mL of concentrated eluent to 100 mL of each standard and sample.

6.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

6.4. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow system.

6.5. The acetate, formate, and other monovalent organic acid anions elute early in the chromatographic run and can interfere with fluoride. The retention times of anions may differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples where acetate is used for pH adjustment.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	One 125mL plastic or glass bottle	No preservative	Cool to ≤6°C	**Nitrate or Nitrite:** Analysis must be completed within 48 hours of collection date/time. **Other Anions:** Analysis must be completed within 28 days of collection date.
Aqueous NO3+NO2	One 125mL plastic or glass bottle	pH<2 with H_2SO_4	Cool to ≤6°C	Analysis must be completed within 28 days of collection date.
Solid	One 4 oz. wide mouth plastic or glass jar	No preservative	Cool to ≤6°C	Analysis must be completed within 28 days of collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation/Equipment

Equipment	Description/Comments
Ion Chromatograph System	Dionex ICS-3000 system or equivalent system that includes autosampler and data system.

9.2. Chromatography Supplies

Item	Description
Guard Column	Dionex AG14 IonPac 4x50mm, or equivalent
Analytical Column	Dionex AS14 IonPac 4x250mm, or equivalent

9.3. General Supplies

Item	Description
Gas tight syringes	Various sizes
Pipets	Class A or calibration-checked variable volume
Volumetric flasks	Class A, various sizes
Graduated cylinders	Class A, various sizes
Beakers	Glass or plastic disposable
Syringe filters	0.45um for filtering samples when required
Filtration apparatus	With 0.45um filter disks for filtration of eluent
Sample vials	120mL plastic with screw top caps
Autosampler vials	1.5mL with screw top septum caps
Balance, Analytical/Top Load	Able to measure to nearest 0.1g
Agitation apparatus	Shaker table, ultrasonic bath or equivalent apparatus for preparation of solids

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Sodium Bicarbonate	A.C.S grade powder, Fisher S233 or equivalent
Sodium Carbonate, Anhydrous	A.C.S. grade powder, Fisher S263 or equivalent
Stock Eluent	Place 500mL of reagent water into a 1L volumetric flask, add 8.4g of Sodium Bicarbonate and 37.1g of Sodium Carbonate, dissolve and dilute to 1L with reagent water. This solution expires 6 months from the date prepared.
Working Eluent	Place 1000mL of reagent water into a 2L volumetric flask, add 20mL of the Stock Eluent and dilute to 2L with reagent water. This solution should be vacuum filtered through a 0.45um filter prior to use. This solution must be prepared fresh daily.
Simulated soil matrix	Teflon chips, glass beads, plastic beads or other suitable simulated soil matrix.

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration on an ongoing basis.	CCV
Spiking Standard	This solution contains the target analyte and is used to spike MS/MSD sets.	Same solution can be used for both the LCS and MS/MSD.

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
IC Stock Calibration Standard	AccuStandard; catalog #IS-17854-250ML; 100/250/500ug/mL, or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
IC Nitrite Stock Calibration Standard	AccuStandard, catalog #IC-NO2-N-10X-1; 1000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
IC Intermediate Calibration Standards	See prep directions below in Section 10.2.3.1.	Expires 30 days from date of preparation.	Refrigerate
IC Working Calibration Standards	See prep directions below in Section 10.2.3.2.	Expires one week from date of preparation.	Refrigerate
IC Stock ICV Standard	Inorganic Ventures; catalog # HES-8-REV1; 50/125/250ug/mL, or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions. Refrigerate after opening.
IC Working ICV Standard	See prep directions below in Section 10.2.3.3.	Expires one week from date of preparation.	Refrigerate
IC Iodide Stock Calibration Standard	Environmental Express, catalog #IC-II-M; 1000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
IC Iodide Intermediate Calibration Standard	See prep directions below in Section 10.2.3.5.	Expires six months from date of preparation.	Refrigerate
IC Iodide Working Calibration Standards	See prep directions below in Section 10.2.3.6.	Expires six months from date of preparation.	Refrigerate
IC Iodide Stock ICV Standard	O2Si, catalog #062013-01-01; 1000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions. Refrigerate after opening.
IC Iodide Working ICV Standard	See prep directions below in Section 10.2.3.7.	Expires six months from date of preparation.	Refrigerate

10.2.3. Standard Preparation Procedures

10.2.3.1. IC Intermediate Calibration Standard Preparation

Dilute 50mL of the IC Stock Calibration Standard (100/250/500ug/mL) and 5mL of the IC Nitrite Stock Calibration Standard (1000ug/mL) to 100mL with reagent water. Final concentration of anions:

Anion	Int. Std. Conc., ug/mL
Bromide	50
Chloride	125
Fluoride	50
Nitrate as N	50
Nitrite as N	50
Sulfate	250

10.2.3.2. IC Working Calibration Standards Preparation

Working Calibration Standards are prepared in reagent water from the IC Intermediate Calibration Standard (50/125/250ug/mL). Actual calibration concentrations may vary.

IC Working Calibration Standards (examples only)

Standard	IC Int. Cal. Std volume	Final Volume in reagent water
Calibration Std 0	0mL	100mL
Calibration Std 1	0.1mL	100mL
Calibration Std 2	0.4mL	100mL
Calibration Std 3 (CCV)	1mL	100mL
Calibration Std 4	2mL	100mL
Calibration Std 5	4mL	100mL
Calibration Std 6	10mL	100mL

Final concentrations in each working calibration standard as prepared above:

mg/L	CAL 0	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6
Bromide	0	0.05	0.2	0.5	1.0	2.0	5.0
Chloride	0	0.125	0.5	1.25	2.5	5.0	12.5
Fluoride	0	0.05	0.2	0.5	1.0	2.0	5.0
Nitrate as N	0	0.05	0.2	0.5	1.0	2.0	5.0
Nitrite as N	0	0.05	0.2	0.5	1.0	2.0	5.0
Sulfate	0	0.25	1.0	2.5	5.0	10	25

10.2.3.3. IC Working ICV Standard Preparation

Dilute 0.1mL of the IC Stock ICV Standard (50/125/250ug/mL) to 10mL with reagent water. Final concentration of anions:

Anion	ICV Std. Conc., mg/L
Bromide	0.5
Chloride	1.25
Fluoride	0.5
Nitrate as N	0.5
Nitrite as N	0.5
Sulfate	2.5

10.2.3.4. Method Blank, Laboratory Control Sample (LCS) and Matrix Spike (MS)

10.2.3.4.1. Aqueous Method Blank: consists of reagent water.

10.2.3.4.2. Aqueous LCS: Dilute 0.1mL of the IC Stock ICV Standard (50/125/250ug/mL) to 10mL with reagent water. Spike concentrations are the same as the Working ICV.

10.2.3.4.3. Aqueous MS: Dilute 0.1mL of the IC Stock ICV Standard (50/125/250ug/mL) to 10mL with sample. Spike concentrations are the same as Working ICV.

10.2.3.4.4. Soil Method Blank: place 10 +/-0.5g of simulated soil matrix and 100mL reagent water into a 120mL sample vial, secure the cap. Agitate for 10 minutes then allow the slurry to settle.

10.2.3.4.5. Soil LCS: place 10 +/-0.5g of simulated soil matrix, 1.0mL of the IC Stock ICV Standard (50/125/250ug/mL) and dilute to100mL with reagent water in a 120mL sample vial, secure the cap. Agitate for 10 minutes then allow the slurry to settle. Spike concentrations are the same as the Working ICV.

10.2.3.4.6. Soil MS: place 10 +/-0.5g of sample, 1.0mL of the IC Stock ICV Standard (50/125/250ug/mL) and dilute to100mL with reagent water in a 120mL sample vial, secure the cap. Agitate for 10 minutes then allow the slurry to settle. Spike concentrations are the same as the Working ICV. If filtration is necessary, Method Blank and LCS must also be filtered.

10.2.3.5. IC Iodide Intermediate Calibration Standard Preparation

Dilute 10mL of the IC Iodide Stock Calibration Standard (1000ug/mL) to 100mL with reagent water for a final concentration of 100ug/mL.

10.2.3.6. IC Iodide Working Calibration Standards Preparation

Working Iodide Calibration Standards are prepared in reagent water from the IC Iodide Intermediate Calibration Standard (100ug/mL). Actual calibration concentrations may vary.

IC Iodide Working Calibration Standards (examples only)

Standard	Iodide Stock Standard volume	Final Volume in reagent water	Final Iodide Conc., mgL
Calibration Std 0	0mL	100mL	0
Calibration Std 1	0.5mL	100mL	0.5
Calibration Std 2 (CCV)	1.0mL	100mL	1
Calibration Std 3	5mL	100mL	5
Calibration Std 4	10mL	100mL	10
Calibration Std 5	25mL	100mL	25

10.2.3.7. IC Iodide Working ICV Standard

Dilute 0.05mL of the IC Iodide Stock ICV Standard (1000ug/mL) to 10mL with reagent water for a final concentration of 5mg/L.

10.2.3.8. IC Iodide Method Blank, Laboratory Control Sample (LCS) and Matrix Spike (MS)

10.2.3.8.1. Aqueous Method Blank: consists of reagent water.

10.2.3.8.2. Aqueous LCS: Dilute 0.05mL of the IC Iodide Stock ICV Standard (1000ug/mL) to 10mL with reagent water for a final concentration of 5mg/L.

10.2.3.8.3. Aqueous MS: Dilute 0.05mL of the IC Iodide Stock ICV Standard (1000ug/mL) to 10mL with sample for a final concentration of 5mg/L.

10.2.3.8.4. Soil Method Blank: place 10 +/-0.5g of simulated soil matrix and 100mL reagent water into a 120mL sample vial, secure the cap. Agitate for 10 minutes then allow the slurry to settle.

10.2.3.8.5. Soil LCS: place 10 +/-0.5g of simulated soil matrix, 0.5mL of the IC Iodide Stock ICV Standard (1000ug/mL) and dilute to100mL with reagent water in a 120mL sample vial, secure the cap. Agitate for 10 minutes then allow the slurry to settle. Spike concentration is 50mg/kg.

10.2.3.8.6. Soil MS: place 10 +/-0.5g of sample, 0.5mL of the IC Iodide Stock ICV Standard (1000ug/mL) and dilute to100mL with reagent water in a 120mL sample vial, secure the cap. Agitate for 10 minutes then allow the slurry to settle. Spike concentration is 50mg/kg. If filtration is necessary to remove suspended particles, Method Blank and LCS must also be filtered.

11. Calibration

11.1. Initial Calibration

11.1.1. Set up and warm up the ion chromatograph to establish a stable baseline per manufacturer's instructions.

11.1.2. For initial calibration, analyze a blank and a minimum of three concentrations of calibration standard for each analyte of interest. The lowest calibration standard must be at or below the required reporting limit. Analyze calibration standards in order of increasing concentration. Document the peak area response and retention time for each analyte. A new initial calibration must be performed every six months, at a minimum.

11.1.3. Using the manufacturer's data system software, establish the individual analyte calibration curves by plotting the peak area response against the corresponding concentrations. Use a least squares linear regression to calculate the calibration curve formula. A weighted least squares regression may also be performed using 1/concentration or $1/(\text{concentration})^2$ as the weighting factor. In either case, the correlation coefficient must be 0.995 or greater to be used for quantitation. In situations where the analyst knows the instrument response does not follow a linear model over a sufficiently wide working range, or when other approaches have not met the acceptance criteria, a non-linear or quadratic calibration model may be employed. In order to use a quadratic calibration for quantitation of sample results, a minimum of six calibration standards must be used and the coefficient of determination (COD) or r^2 must be greater than or equal to 0.99. Refer to Method 8000C for additional guidance on calibration procedures.

11.1.4. **Initial Calibration Corrective Action:** If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.1.5. Each day that analysis is performed, the calibration standard(s) should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Are the retention times consistent over the range of calibration for each compound? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably.

11.1.6. **Initial Calibration Verification (ICV)**: The initial calibration must be verified through the analysis of an Initial Calibration Verification (ICV) standard. The ICV is prepared from an independent source at or near the mid-range of the calibration curve and analyzed immediately following the initial calibration curve. Acceptable recovery range for the ICV is +/-10% of its true value or 90-110% recovery.

11.1.7. **ICV Corrective Action:** If the ICV fails the criteria, another ICV may be analyzed. If the second ICV fails, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV fails and is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.2. Calibration Verification

11.2.1. The initial calibration must be verified daily with the analysis of a Continuing Calibration Verification Standard (CCV) at or near mid-range concentration at the beginning of the analytical sequence, after every 10 injections, and at the end of the analytical sequence, as a minimum

requirement. If a quadratic curve fit is used, the calibration must be verified at two concentration levels using at the beginning, after every 10 injections and at the end of each sequence. Acceptable recovery range for the CCV is +/-10% of its true value or 90-110% recovery. The retention time for each analyte must not vary by more than +/-10% from its expected value.

11.2.2. CCV Corrective Action*:* If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

12. Procedure

12.1. Set up and warm up the ion chromatography system to establish a stable baseline per manufacturer's instructions and equivalent to the conditions used for initial calibration. All samples must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

12.2. Sample Preparation and Handling

12.2.1. Aqueous Samples

Filter any samples that contain particles larger than 0.45um using a syringe filter. Fill a labeled autosampler vial with a minimum of 1mL of sample. An initial rinse of the vial with sample is recommended. Screw on the septum cap and the sample is ready for analysis. If filtration is necessary, the associated Method Blank, LCS and MS/MSD (if associated with filtered sample) must also be filtered. **NOTE:** Samples that are received acid preserved for NO3+NO2 must be diluted prior to analysis due to the sulfate interference presented by the sulfuric acid preservative.

12.2.2. Soil Samples

Weigh 10 +/-0.5g of sample into a120mL sample vial and add 100mL of reagent water. Agitate for 10 minutes then allow the slurry to settle. Fill a labeled autosampler vial with a minimum of 1mL of the supernatant. If filtration is necessary, the associated Method Blank, LCS and MS/MSD (if associated with filtered sample) must also be filtered. An initial rinse of the autosampler vial with the filtrate is recommended. Screw on the septum cap and the sample is ready for analysis.

12.3. Inject a suitable volume of sample or QC standard into the IC instrument per manufacturer's instructions. The volume of sample injected must be consistent with the volume used for initial calibration standards. Record the resulting analyte peak areas as well as the peak retention times. A typical run sequence may be as follows:

Instrument Blank
ICAL Standards
ICV
ICB
(If ICAL not run, CCV would replace the ICAL and the ICV in the sequence)
CCV
CCB
Method blank
LCS
Client samples
CCV
CCB

Client samples
CCV
CCB

12.4. Sample concentrations are calculated by comparing response data with the initial calibration. The width of the retention time window used to identify analytes in samples should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window for each analyte. The experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.5. If sample response exceeds the limits of the initial calibration range, dilute the sample with reagent water and reanalyze or the over range result must be qualified as estimated.

12.6. Refer to the Manual Integrations SOP for guidance on performing and documenting manual integrations.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	**300.0:** Target analytes should be less than the MDL. **9056A:** Target analytes should be <10% of the reporting limit or <10% of the lowest sample conc., whichever is greater.	Reanalyze associated samples if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Target analytes	One per preparation batch of up to 20 samples, per matrix.	**300.0:** 90-110% recovery **9056A:** 80-120% recovery	Reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified. 3) An associated matrix spike that passes LCS acceptance criteria can be used in place of a failing LCS.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Target analytes	**300.0:** One MS per a minimum of 10% of samples. **9056A:** One MS/MSD set per batch of up to 20 samples, per matrix.	80-120% recovery ≤15% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Sample Duplicate (DUP)	Sample	One duplicate sample analysis per batch if no MS/MSD performed	≤15% RPD	No corrective actions necessary. RPD outside acceptance criteria must be qualified appropriately.

14. Data Analysis and Calculations

14.1. Calculate the final concentration in the sample as follows**:**

Aqueous Sample (mg/L) = $(X_s)(D)$ Solid Sample (mg/kg) = $\frac{(X_s)(V_f)(D)}{(W_s)}$

Where: X_s = Concentration of the analyte in the sample from the curve in mg/L
D = Dilution factor of aqueous sample or solid extract
V_f = Final volume of solid extract in Liters
W_s = Weight of solid sample purged or extracted in kilograms

Moisture corrected concentration = $\frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$

14.2. LCS equation:

R = (C/S) * 100

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.3. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.4. RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. An MDL and/or LOD/LOQ verification study must be conducted every six months for each matrix per instrument.

18.2. Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Method modified for the determination of Iodide.

19.2. Columns and chromatographic conditions may differ from those recommended and are based on instrument manufacturer's specifications.

19.3. Eluent is filtered through 0.45um filter disks instead of 0.2um filter disks.

19.4. Soil extraction procedure found in Method 300.0, revision 2.1, Section 11.7 is also used for Method 9056A. Shaker table or ultrasonic bath is used for agitation of soils instead of magnetic stir bars.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. The use of gloves, lab coats and safety glasses is required. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Pollution Prevention and Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 9056A and 8000C.

25.2. Environmental Protection Agency, USEPA Method 300.0, Revision 2.1, August 1993.

25.3. Pace Analytical Quality Manual; latest revision.

25.4. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-O-170-rev.00	1. Converted to Pace SOP format. 2. Revised calibration to be a single curve with a more limited linear range. 3. Updated standard solutions final volumes to minimize waste. 4. Added requirement for use of simulated soil matrix to be used for soil batch QC.	04Feb2015
S-IN-O-170-rev.01	1. Table 9.3: added Iodide standards 2. Section 9.2.3: added preparation of Iodide standards. 3. Section 10.2.1: corrected CCV control limits to 90-110% recovery 4. Table 12.1: corrected LCS control limits for Method 300.0 to 90-110% recovery.	20Jul2015
S-IN-O-170-rev.02	1. Converted to 27-section format. 2. Table 7.1: revised storage temperature format and clarified holding time for Nitrate or Nitrite. 3. Section 10.1: updated filter used for eluent preparation. 4. Table 10.3: revised storage conditions. 5. Section 10.2.3: clarified preparation/final volume of batch QC for solids. 6. Section 14.1: corrected units in final concentration equations. 7. Section 25.4: added years 2003 and 2009 to TNI reference.	19Jul2017
S-IN-O-170-rev.03	1. Section 2.1: added analytical column. 2. Section 12.3: removed ICVA/CCVA and language about quadratic. 3. Table 13.1: updated acceptance criteria for method blank.	06Feb2018

ATTACHMENT C-21

WASTE HANDLING AND MANAGEMENT
PACE, INDIANAPOLIS

Pace Analytical Services
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Indianapolis, IN 46268
Phone: 317.228.3100
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STANDARD OPERATING PROCEDURE

WASTE HANDLING AND MANAGEMENT

Reference Methods: N/A

SOP Number:	S-IN-W-002-Rev.03
Effective Date:	April 10, 2017
Supersedes:	S-IN-S-002-Rev.02
SOP Template Number:	SOT-ALL-W-002-rev.07

APPROVALS

Laboratory General Manager — March 28, 2017 Date

Laboratory Quality Manager — March 24, 2017 Date

Laboratory Waste Coordinator — March 27, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose/Identification of Procedure

1.1. Pace Analytical Services (Pace) acknowledges its obligation to the responsible management of the environment and its resources. Pace senior management is committed to operating in such a way that meets or exceeds the state and federal laws governing waste management and encourages the use of best practices to reduce, reuse and recycle waste material where possible. This Standard Operating Procedure (SOP) documents the systems, processes and procedures that this location uses to manage generated wastes.

1.2. It is Pace's policy to minimize the amount of hazardous waste it produces and to reduce the hazardous properties of those wastes whenever practical within regulatory compliance. This can be achieved by periodic auditing of all processes producing hazardous waste; reduction of sample volume delivered by the client; return of excess sample material to clients whenever practical and economical; investigation of new technologies that might require smaller volumes of sample, or produce fewer or less hazardous by-products; implementation of lab cleaning procedures that reduce the volume of cleaning residue; recycling of hazardous materials; and investigation of new treatment technologies that are comprehensively destructive or are effective in reducing the volume or hazardous qualities of the wastes produced.

2. Summary of Method

2.1. Pace facilities that generate waste must initially contact the EPA to obtain an ID number. Each unique type of generated waste is classified and characterized into waste streams according to procedures in 40 CFR 261. The amount of waste the facility generates determines the Generator Status of a lab, which in turn determines how long and how much waste can accumulate. Pace is ultimately responsible for the waste it generates, and is required to obey any and all regulations during the process of creating, accumulating, disposing, and releasing waste to a TSDF for final disposal. Documentation is kept to prove all regulations have been obeyed.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel responsible for all aspects of waste handling and management.

3.2. This SOP is applicable to all processes that involve generated waste, and is designed to assist its operations in adhering to regulations set forth in the following federal statutes: Resource Conservation and Recovery Act (RCRA), Clean Water Act (CWA), Toxic Substances Control Act (TSCA), and DOT Title 49, and Transportation (parts 100-199). Particular attention is given to local pretreatment standards covering discharges to publicly owned treatment works (POTW) when performing elementary neutralization on acidic and basic waste. The local standards are based in part upon provisions in the National Pretreatment Standards and Prohibited Discharge Standards.

3.3. The degree to which RCRA regulations apply to Pace facilities is dependent upon the generator status of the operation. Under the federal rules (state requirements may be more stringent or give the classes a slightly different name) there are three different classes of hazardous waste generators based upon the amount of waste generated in a month to month time frame.

3.4. **Waste Generator Class Limits (federal categories, some locations may have different titles):**

Hazardous Waste Generator Class	Quantity of Hazardous Waste Generated per Month	Generated Monthly Acute Hazardous Waste	Maximum Allowable Hazardous Waste Quantity on-site	Maximum Permitted Waste Accumulation Time
Cond. Exempt Small Quantity	<100kg	<1 kg	<1000kg	Unlimited
Small Quantity	100-1000kg	<1 kg	<6000kg	180 days (270 days if the waste must be sent >200 miles to TSDF)
Large Quantity	>1000kg	>1kg	Unlimited	90 days

3.5. **Parameters**: Not applicable to this SOP.

4. Definitions

4.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

4.2. **Acutely Hazardous Waste** - A waste which is hazardous as identified with an (H) Hazard Code in the lists of Hazardous Waste in 40 CFR Part 261, Subpart D, Sections 261.30, 261.31 and 261.33.

4.3. **Animal and Plant Health Inspection Service (APHIS)** - an agency of the USDA responsible for protecting animal health, animal welfare, and plant health. APHIS is the lead agency for collaboration with other agencies to protect U.S. agriculture from invasive pests and diseases.

4.4. **Clean Air Act** - The Federal Clean Air Act, 42 U.S.C. 7401, and amendments thereto amending 42 U.S.C. 1857 et.seq.

4.5. **Conditionally Exempt Small Quantity Generator** - A generator who produces no more than 100 kilograms of hazardous waste or one kilogram of acutely hazardous waste (or a total of 100 kilograms of any residue or contaminated soil, waste or other debris resulting from the cleanup of a spill, into or on any land or water, or any acute hazardous waste) in a calendar month. The total amount of hazardous waste which may be accumulated on-site is 1000 kilograms.

4.6. **Confined Space** – A space that is large enough and so configured that an employee can bodily enter and perform assigned work; and has limited or restricted means for entry or exit (for example, tanks, vessels, silos, storage bins, hoppers, vaults, and pits are spaces that may have limited means of entry); and is not designed for continuous employee occupancy.

4.7. **Container** - Any device material is stored, transported, treated, disposed of, or otherwise handled.

4.8. **Contingency Plan** - A document setting out an organized, planned, and coordinated course of action to be followed in case of fire, explosion, or release of hazardous waste or hazardous waste constituents which could threaten human health or the environment.

4.9. **Designated Hazardous Waste Storage Area** - Area used to hold hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere. This is the storage area into which hazardous waste from the laboratory (e.g., satellite waste) is moved.

4.10. **DOT** - The United States Department of Transportation.

4.11. **DTSC** – Department of Toxic Substances Control.

4.12. **Elementary Neutralization Unit** – A device which: (1) is used for neutralizing wastes which are hazardous only because they exhibit the corrosivity characteristic defined in 40 CFR 261.22 or are listed in Subpart D of Part 261; and (2) meets the definition of tank, container, transport vehicle, or vessel in 40 CFR 260.10.

4.13. **EPA** - The United States Environmental Protection Agency.

4.14. **EPA Hazardous Waste Number** - The EPA number assigned to each EPA hazardous waste identified in 40 CFR Part 260, Subpart D - Lists of Hazardous Wastes.

4.15. **EPA Identification Number** - The site-specific number assigned to each generator, transporter, and TSDF upon approval of a notification form.

4.16. **Federal Clean Water Act** - 33 U.S.C. 1251, et. Seq.

4.17. **Foreseeable Emergency** - Any fire, explosion, or sudden or non-sudden release of hazardous waste or hazardous waste constituents to the air, soil, or surface water, which could threaten human health or the environment.

4.18. **Generator** - Any person, by site who owns or operates a facility where hazardous waste is generated, i.e. Pace.

4.19. **Hazardous Waste Coordinator** - The Pace employee responsible for creating, guiding, and implementing all hazardous waste management operations.

4.20. **Hazardous Waste** - As defined in 40 CFR Part 261, Subparts B and C, a solid, semi-solid, liquid or contained gaseous waste, or any combination of these wastes.

4.20.1. Which, because of either quantity, concentration, physical, chemical, or infectious characteristics may:

4.20.2. Cause or contribute to an increase in mortality or an increase in irreversible or incapacitating reversible illness; or

4.20.3. Pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed of or otherwise mismanaged.

4.20.4. Or which has been identified as having a characteristic of hazardous waste by the EPA using the criteria established under 40 CFR Part 261, Subpart C, or as listed under Sections 261.31, 261.32, 261.33, and 261.34. Such wastes include, but are not limited to, those which are reactive, toxic, corrosive, ignitable, irritants, strong sensitizers or which generate pressure through decomposition, heat or other means. Such wastes do not include radioactive substances that are regulated by the Atomic Energy Act of 1954, as amended. A waste is considered hazardous if it is listed or it fits into one of four categories. These categories are as follows:

4.20.4.1. Ignitable – (40 CFR 261.21, Waste Code D001) A flash point of less than 60^{o}C/140^{o}F.

4.20.4.2. Corrosive – (40 CFR 261.22, Waste Code D002) A pH of less than 2.0 or greater than 12.5.

4.20.4.3. Reactive – (40 CFR 261.23, Waste Code D003) Reactive wastes exhibit one or more of the following characteristics:

4.20.4.3.1. It is unstable and can undergo a violent change without detonating.

4.20.4.3.2. It can react violently with water.

4.20.4.3.3. When mixed with water it can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

4.20.4.3.4. It is cyanide or sulfide bearing waste that, when exposed to pH conditions between 2.0 and 12.5, can generate gases, vapors, or fumes that can present a danger to human health or the environment.

4.20.4.3.5. It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

4.20.4.3.6. It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

4.20.4.3.7. It is a forbidden explosive as defined in 49 CFR 173.51, or a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.88.

4.20.4.4. Toxic – (40 CFR 261.24, Waste Codes D004-D043) A solid waste that contains a toxic concentration of a contaminant listed in 40 CFR 261.24, Table 1. A toxic waste is given any and all D-codes that apply to the particular material.

4.21. **Hazardous Waste Constituent** - A substance, compound, or element listed as hazardous waste in EPA 40 CFR 261.

4.22. **Lab Pack Material** - A hazardous waste that does not match a listed Pace waste stream category.

4.23. **Large Quantity Generator (LQG)** - Any generator who generates at a rate greater than 1000 kilograms of hazardous waste per month.

4.24. **Manifest** - As defined in 40 CFR Part 262, Subpart B, namely "the form used for identifying the origin, quantity composition, routing and destination of hazardous waste".

4.25. **Plant Protection and Quarantine (PPQ)** – A program within APHIS which attempts to safeguard agriculture and natural resources in the U.S. against the entry, establishment, and spread of animal and plant pests and noxious weeds.

4.26. **Regulated Soil** – Soil from foreign countries, U.S. territories and areas within states that are under Federal quarantine that can be moved into or through continental U.S. only if conditions and safeguards prescribed by the USDA and APHIS are met.

4.27. **Sample** - Except as provided below in 4.27.2.2.3, any solid waste, water, soil, or air that is collected for the sole purpose of being tested to determine its characteristics or composition.

4.27.1. Samples are not subject to any requirements of 40 CFR Part 261.5 or Parts 262 through 267 or Part 270 or Part 124 or to the notification requirements of Section 3010 of RCRA, when:

4.27.1.1. The sample is being transported to a laboratory for the purpose of testing; or

4.27.1.2. The sample is being transported back to the sample collector after testing; or

4.27.1.3. The sample is being stored by the sample collector before transport to a laboratory for testing; or

4.27.1.4. The sample is being stored in a laboratory before testing; or

4.27.1.5. The sample is being stored in a laboratory after testing but before it is returned to the sample collector; or

4.27.1.6. The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary).

4.27.2. In order to qualify for the exemption in 4.27.1.1 and 4.27.1.2 above, a sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must:

4.27.2.1. Comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements; or

4.27.2.2. Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:

4.27.2.2.1. Assure that the following information accompanies the sample:

4.27.2.2.1.1. The sample collector's name, mailing address, and phone number;

4.27.2.2.1.2. The laboratory's name, mailing address, and phone number;

4.27.2.2.1.3. The quantity of the sample;

4.27.2.2.1.4. The date of shipment; and

4.27.2.2.1.5. A description of the sample.

4.27.2.2.2. Package the sample so that it does not leak, spill, or vaporize from its packaging.

4.27.2.2.3. This exemption does not apply if the laboratory determines that the waste is hazardous but the laboratory is no longer meeting any of the conditions stated in 4.27.1 above.

4.28. **Satellite Waste or Laboratory Satellite Waste** - Hazardous waste generated by Pace that is at or near any point of generation and under the control of the operator. Satellite accumulation provisions allow generators to accumulate up to 55 gallons of hazardous waste (or 1 quart of acute hazardous waste) in containers without starting the storage clock as described in Section 3.4.

4.29. **Satellite Waste Container** - Any portable device used to accumulate laboratory generated waste prior to transfer to the hazardous waste storage area.

4.30. **Small Quantity Generator(SQG)** - A generator who produces no more than 1000 kilograms of hazardous waste (or a total of 1000 kilograms of any residue or contaminated soil, waste or other debris resulting from the cleanup of a spill, into or on any land or water, or any acute hazardous waste) in a calendar month. The total amount of hazardous waste which may be accumulated on-site is 6000 kilograms.

4.31. **TSDF** - A Treatment/Storage/Disposal Facility.

4.32. **Universal Waste** – Commonly used items that are hazardous but can be recycled. These include fluorescent lights, computer monitors, etc.

4.33. **Waste Stream** - The generic profile of chemical and physical properties that satellite wastes exhibit.

5. Procedure

5.1. All Pace facilities that generate hazardous waste must have a Generator's US EPA Identification Number. The ID number is obtained through the applicable EPA region's office by completing EPA form 8700-12, and must be completed before generating any hazardous waste.

5.1.1. Pace only utilizes transporters and treatment, storage, or disposal facilities (TSDFs) that have EPA identification numbers for hazardous waste handling and meet the TSDF transfer requirements.

5.1.2. A new ID number is necessary when changing locations as the number is tied to the facility address.

5.1.3. This facility's US EPA Identification Number is IND984874206.

5.2. The laboratory generates wastes originating from several source types: materials and chemicals used to prepare and analyze samples (e.g., solvents, acids), unconsumed liquid and solid samples, certain types of batteries, mercury from lamps and broken thermometers and automobile waste. Unconsumed samples may include laboratory-contaminated sample residue (both liquid and soil) generated as part of digestion, extraction, etc., procedures used to prepare samples for analysis.

5.2.1. Based on table 3.4, this facility is classified as a Small Quantity Generator.

5.3. Hazardous waste classification is the most critical step in establishing an effective, compliant waste-handling program. Laboratory wastes are classified using the criteria set forth under RCRA for ascertaining non-hazardous versus hazardous status, and this criterion is listed in the definition of hazardous waste in 4.20.

5.4. The following are the waste streams resulting from materials and chemicals used in the laboratory operation. Applicable information for each is given pertaining to packing, labeling, or listing on a manifest. A description of how the wastes are created, and the preferred method of final disposal for each, is included. The overriding principle in hazardous waste classification is application of a conservative formula based on all known or suspected hazards related to a waste material. While this formula may result in some materials being disposed as hazardous when in fact, they are non-hazardous (e.g., false positive), the formula will not be compromised in the interest of reducing the amount of waste produced. This will minimize any risk of a material being disposed of erroneously as non-hazardous when it, by definition, is a hazardous waste.

5.4.1. **Corrosive waste** is generated in the majority of the departments in the laboratory. This waste stream consists primarily of spent or excess aqueous reagent solutions generated from preservatives, acid digestions of metals, impinger solutions or other corrosive solutions generated in the course of analysis. The predominant corrosives include hydrochloric acid, nitric acid and sulfuric acid, but corrosives also include bases. Varying concentrations of metals may be present dependent upon the composition of the reagents added. This waste stream only has the hazardous quality of being corrosive; therefore, if a waste has any additional hazardous waste quality (e.g., Toxic or Ignitable) it cannot be mixed with this stream. This stream is most commonly treated onsite.

Corrosive Waste	
DOT Shipping Name	RQ Waste, Corrosive Liquid, N.O.S (i.e. corrosive material)
EPA Waste #	D002
Container	LIST CONTAINER
Average pH	<2.0,>12.5
Disposal Method	Treatment by Neutralization onsite
Label	Corrosive

5.4.2. The **Chlorinated Waste Stream** consists primarily of methylene chloride with a very small amount of other organic solvents derived from extraction procedures performed on samples and from rinsing glassware. As a best practice, effort is made to have this waste stream as pure as possible in order to offer for recycling.

Chlorinated Solvents	
DOT Shipping Name	RQ Hazardous Waste, Toxic Liquid, N.O.S (i.e. dichloromethane, acetone, methanol)
EPA Hazard Codes	U080, F001/F002
Container	55 gallon drum
Average pH	7.0
Disposal Method	Removed by licensed waste handler
Label	Toxic, Chlorinated

5.4.3. **COD Waste** is specific waste that results from COD analysis. This waste comes from used and expired COD vials of samples and reagent. This stream has sulfuric acid, mercuric sulfate, potassium dichromate, and silver sulfate.

COD Waste	
DOT Shipping Name	NAME
EPA Waste #	D002, D007, D009, D011
Container	COD vial box
Average pH	<2
Disposal Method	Lab-packed by licensed waste handler
Label	Corrosive, Toxic

5.4.4. **PCB Waste** is specific waste that results from PCB analysis. This waste is generated from the preparation of standard solutions used for PCB analysis and may include pipets, vials and other disposable glassware that is contaminated with PCBs. Environmental samples containing 50ppm or more of PCBs are included in this waste stream for disposal by incineration.

PCB Waste	
DOT Shipping Name	UN3432, Polychlorinated Biphenyls
EPA Waste #	N/A
Container	55 gallon drum
Average pH	N/A
Disposal Method	Incineration by licensed waste handler
Label	PCB Waste

5.4.5. **Methanol Waste** is specific waste that results from the analysis of soils for VOCs. This waste comes from soil samples that are preserved in the vial with methanol and the waste stream will include glass vials with plastic lids along with the soil and methanol.

Methanol Waste	
DOT Shipping Name	UN1230, Waste Methanol
EPA Waste #	F003
Container	55 gallon drum
Average pH	N/A
Disposal Method	Removed by licensed waste handler
Label	Methanol Waste

5.4.6. **Miscellaneous Lab Waste** is generated as a result of expired reagents and chemicals, and hazardous samples that cannot be included in another waste stream. This would include waste such as heavy metals waste, solid and aqueous flammable waste, toxic waste and oils. This waste is lab-packed for disposal by a licensed waste handler.

5.5. Some waste can become complicated when attempting to classify as non-hazardous or hazardous due to the list of hazardous constituents contained in sections 40 CFR 261.30-261.35 including a majority of analytes of interest routinely analyzed in Pace laboratories. Definitions have been established for each of the F, K. P, and U lists covering hazardous waste originating from non-specific sources, specific sources and discarded commercial chemical products, off-specification species, container residues, and spill residues. The application of listed hazardous wastes and substances is intended for manufacturing processes involving pure products, by-products, wastes generated as part of the production process and cleanup of materials contaminated from a spill of the listed commercial chemical product or manufacturing chemical intermediate. See Attachment I for common F-listed wastes.

5.5.1. Hazardous waste classification of unconsumed samples by listed hazardous waste criteria is not commonly applied in laboratory operations. Examples of sample types which would be identified as listed hazardous wastes include the following:

5.5.1.1. Samples containing 5% or more (by volume) of halogenated and non-halogenated "spent solvents:" (e.g., drum sample with > 10% TCE);

5.5.1.2. Pure product and two phase solution samples containing a listed chemical product or manufacturing intermediate (e.g., drum sample);

5.5.1.3. Samples from specific sources listed in section 261.32 (e.g., bottom sediment sludge from the treatment of wastewaters from wood-preserving processes that use creosote and/or pentachlorophenol - K001);

5.5.1.4. Samples representing any residue or contaminated soil, water or other debris resulting from the cleanup of a spill into or on any land or water of any commercial chemical product or manufacturing chemical intermediate having a generic name listed in section 261.33, or any residue or contaminated soil, water or other debris resulting from the cleanup of a spill, into or on any land or water, of any off-specification chemical product and manufacturing chemical intermediate which, if it met specifications, would have the generic name listed in section 261.33.

5.5.2. For the wastes listed in 5.5.1.1 and 5.5.1.2, disposal can be achieved by individually lab packing them or combining with other compatible hazardous wastes.

5.5.3. The remaining two sample types in 5.5.1.3 and 5.5.1.4 would also require lab packing for disposal. However, it is important to note that in order for the laboratory to ascertain that the samples were derived from a specific listed source or from a spill of a listed chemical, they must be so informed by the industrial concern or lead agency (e.g., EPA, state regulators) submitting the sample for analysis. If a water or soil sample contains a listed hazardous waste substance whose origin is unknown or uncertain to the lead agency, then that sample is not classified as a listed hazardous waste. Rather in this case, determination of a hazardous waste classification can only be obtained by the waste exhibiting a characteristic of hazardous waste (e.g., hazardous contaminants, ignitability, corrosivity, reactivity).

5.5.4. Due to the fact that the majority of samples analyzed by Pace do not meet the well-defined criteria for identifying "listed" hazardous waste, disposal classification of unconsumed samples will be based upon characteristics of hazardous waste:

5.5.4.1. Non-Hazardous - Analysis results indicate an absence of contaminants; unless contaminants listed under the hazardous disposal categories are parts of the requested sample analysis.

5.5.4.2. Hazardous – Analysis results indicate presence of contaminants (Attachment III) or sample analysis requires hazardous materials and contaminants. Samples in this category are segregated from others and disposed of as hazardous according to laboratory procedures.

5.5.4.3. PCB Waste - Generated exclusively by samples contaminated with greater than trace levels of polychlorinated biphenyls ($\geq$ 50ppm). Samples containing 50ppm (total) or higher of PCBs must be segregated and disposed of as PCB waste.

5.5.4.4. Waste Oil/Paint - Samples which are predominantly of an oil matrix (e.g., highly viscous organic liquid) or paint (solvent and pigment blend) are segregated and disposed in a separate container. Though these samples are defined as nonhazardous, oil samples are a special case and never disposed as nonhazardous. Note: Bottle caps and liners do not typically contain sample residuals and can be disposed of directly through the non-hazardous building refuse.

5.5.5. USDA-APHIS-PPQ Regulated Soils (Regulated Soils) are a special case of sample strictly controlled under quarantine regulations 7 CFR 330 because they can readily provide a pathway for a variety of dangerous organisms throughout the United States. The movement of soil into the United States from foreign sources and from certain regulated areas within the continental U.S. is restricted unless permitted by APHIS under specific conditions and safeguards.

5.5.5.1. Any laboratory that handles Regulated Soils must have an approved Compliance Agreement from USDA-APHIS-PPQ, and labs that handle foreign soils must have an approved Permit to Receive Soil. See updated revision of *USDA Regulated Soil Handling and Disposal S-IN-C-007*for information regarding the handling of these materials.

5.5.6. Though Pace is obligated to ensure nonhazardous discharge complies with requirements set by applicable publicly owned treatment works (POTW) and local regulations, Pace is not obligated to run every available analysis on every sample for proper waste classification. Consequently, samples are characterized according to the preservatives added, the requested analytical testing data, and any knowledge of the sample provided by the client. When sample analysis is canceled/not completed, those untested samples are characterized by the preservatives added and any knowledge of the sample that is obtained by the client.

5.6. Consolidation of wastes from the laboratory proceeds via two distinct routes covering either laboratory-generated hazardous wastes or excess unconsumed samples.

5.6.1. Laboratory Accumulation and Satellite Waste Containers

5.6.1.1. Waste materials from routine lab procedures are collected in containers of appropriate construction, placed in convenient locations at the point of generation. Under RCRA guidelines, these are defined as satellite containers.

5.6.1.2. The amount of hazardous waste stored in the laboratory at the individual satellite areas cannot exceed 55 gallons (liquid) or 550 lbs (solid) per waste stream, for non-acute hazardous waste.

5.6.1.3. Satellite waste containers must be labeled in accordance with all regulations, including:

5.6.1.3.1. Designation of the contents to be hazardous waste with the words “Hazardous Waste” clearly legible.

5.6.1.3.2. The waste stream description (e.g., acid waste).

5.6.1.3.3. A hazard label (e.g., corrosive).

5.6.1.4. The satellite containers must be maintained such that evolution of chemical vapors is precluded. This requires that the container be closed at all times, except when adding or emptying hazardous waste to and from the container.

5.6.1.5. The most critical point in the waste handling system is when a person (e.g., analyst, technician) places a waste material into a satellite container. Here, the characteristics or listing of the waste and the waste stream must both be known to match. For this reason, only material from approved procedures should be placed in the compatible satellite containers. All materials from experimental procedures, unknown or out of the ordinary sources, or from spill cleanups must be characterized and described to the Hazardous Waste Coordinator, who determines the proper method of disposal.

5.6.1.6. Full satellite containers must be transferred to the proper accumulation drum within 3 calendar days. Lab collection containers must not be filled to the top of the opening. Space must be left to prevent splashing of hazardous material when containers are emptied and to allow for expansion and contraction within the drum during transport.

5.6.1.7. Satellite containers for liquid hazardous waste must have secondary containment made of material that could successfully contain the entire satellite container's contents.

5.7. Transferring Satellite Waste to the Waste Storage/Accumulation Area

5.7.1. All transfers of satellite waste to waste drums must be made by the Hazardous Waste Coordinator or designated, trained personnel. When a satellite waste container is full, the Hazardous Waste Coordinator, or designee must be notified. Regular disposal events may be scheduled to dispose satellite waste on a continuous basis.

5.7.2. Find the correct waste drum by referring to the Hazardous Waste placard and hazard label. Mixing solvents that are not compatible could result in a hazardous reaction.

5.7.3. Ensure there is enough capacity in the drum to hold all the content that will be dispensed.

5.7.4. Check to make sure there is a ground connection before opening a solvent waste drum.

5.7.5. Open and slowly pour the contents of the satellite container into the proper waste drum using an appropriate solvent resistant funnel.

5.7.6. Replace the cap on the bunghole and carefully screw the cap on but do not tighten the cap.

5.8. Disposal of Unconsumed Hazardous Samples

5.8.1. Client samples are stored on-site for a defined period of time after the final analytical report is generated and prior to sample disposal. The purpose of sample storage is to provide the client time to review the analytical report and determine if the samples require additional testing or need to be returned to the client. Samples are not considered a waste during this time according to 40 CFR 261.4(d)(1)(vi).

5.8.1.1. During sample storage, the process and sample status must be obvious to employees, customers and auditors. This transparency is imperative to ensure samples are considered active test specimens to be retained until they are categorized as a waste for disposal.

5.8.1.2. Samples which cannot be returned to the client for disposal are characterized according to section 5.4. Samples are characterized by one of three methods:

5.8.1.2.1. Analytical results are evaluated against characterization criteria established for the sample waste stream. The samples which exhibit waste characteristics as previously outlined are segregated and denoted per laboratory/facility policies. A LabTrack ticket is created and then forwarded to the Hazardous Waste Coordinator, who in turn uses the information to coordinate removal of unconsumed samples from active sample storage by the receiving staff.

5.8.1.2.2. The Hazardous Waste Coordinator prints the LabTrack ticket and assigns a client services technician in receiving the task of labeling the sample containers. The number, type and storage locations for the containers are determined form Epic Pro or the COC. An orange dot sticker indicating the hazard (e.g. Lead) is affixed to the top of each container. Non-aqueous liquid samples that are not determined to be hazardous are labeled as Oil/Liquid Non-Hazardous.

5.8.1.2.3. When sample storage areas and/or walk-in coolers are cleared of samples ready for disposal, any samples with orange dot stickers or Oil/Liquid Non-Hazardous labels are placed on a shelf in the disposal area intended for lab-packing. Lab-pack is performed periodically throughout each year by a licensed waste handler.

5.8.1.2.4. PCB and USDA regulated soils are segregated on shelves in the disposal area. The samples are then moved to the PCB hazard drum located in the organic prep lab after they have been retained for greater than 45 days.

5.9. Disposal of Unconsumed Non-Hazardous Samples

5.9.1. Non-hazardous soil/solid samples are placed into the trash compactor destined for incineration.

5.10. Elementary Neutralization

5.10.1. Dilute corrosive solutions (e.g., preserved metals samples) which do not exhibit any hazardous characteristics other than being corrosive, may be neutralized. Elementary neutralization is exempt from RCRA permitting requirements for on-site hazardous waste treatment. While exempt under RCRA guidelines, before utilizing this practice to reduce off-site treatment or disposal of wastes, local pretreatment and discharge standards must be met for publicly owned treatment works (POTW).

5.10.2. The discharges listed below are prohibited under the National Pretreatment Standards and Prohibited Discharge Standards:

5.10.2.1. Pollutants causing fire or explosion (waste with a flashpoint < 60^{o}C);

5.10.2.2. Corrosive wastes with pH less than 2 or greater than 12.5;

5.10.2.3. Solid or viscous pollutants that could potentially block the system;

5.10.2.4. Oxygen-demanding pollutants;

5.10.2.5. Wastes which generate toxic gases.

5.10.3. Dilute corrosive/acidic samples are neutralized in the following procedure:

5.10.3.1. Aqueous samples in glass containers are passed through the glass crusher. The water is pumped from the drum to a neutralization tank/vessel. Refer to Section 6.3 for instructions in the operation of waste disposal equipment.

5.10.3.2. Aqueous samples in plastic containers are poured from the container into a neutralization tank/vessel.

5.10.3.3. As needed, Sodium Hydroxide solution is added to adjust pH to between 6 and 8.

5.10.3.4. Ensure the pH is in the proper range using a pH strip and open the discharge valve from the neutralization tank/vessel and allow neutralized liquid to flow down the drain while flushing with clean water.

5.10.4. Dilute caustic/basic samples are neutralized in the following procedure:

5.10.4.1. Aqueous samples in plastic containers are poured from the container into a neutralization tank/vessel.

5.10.4.2. As needed, Hydrochloric Acid solution is added to adjust pH to between 6 and 8.

5.10.4.3. Ensure the pH is in the proper range using a pH strip and open the discharge valve from the neutralization tank/vessel and allow neutralized liquid to flow down the drain while flushing with clean water.

5.11. Waste Storage Container Requirements

5.11.1. Drums in the hazardous waste storage area are labeled consistent with both DOT and EPA regulations concerning hazardous materials and wastes (see Attachment IV for example of label).

5.11.2. Closure instructions must be available for all containers used to transport hazardous materials. If a container in the accumulation area is the same one the waste will be shipped away in, the Waste Coordinator must obtain the closure instructions from the provider of the containers.

5.11.3. Labels must be easily visible and legible (e.g., a drum must not be labeled and then placed in such a way that the label cannot be seen).

5.11.4. The Accumulation Start Date must be recorded on the drum. The date should reflect the first time waste was added to the drum and not the date when the waste was generated in the laboratory.

5.11.4.1. Once a waste is removed from the point of generation to a hazardous waste staging area, the clock is started for storage time prior to disposal.

5.11.4.2. Drums must be picked up by TSDF for disposal before accumulation time exceeds RCRA requirement for lab's generator status (see section 3.4).

5.11.5. The hazardous waste staging room must be arranged in such a fashion to assure direct access pathways in the event of foreseeable emergency and for safe waste transfer. A minimum aisle space of three feet must be maintained at all times to access hazardous waste containers.

5.11.6. All hazardous waste drums and containers must be securely closed when not in use. All volatile and flammable hazardous waste liquid containers must be securely grounded at all times. Drums containing these liquids should also be manipulated with non-sparking tools and fitted with a drum venting bung, to assure that excess pressure build-ups are safely released.

5.11.7. All liquid waste stream containers must be provided with secondary containment devices. Such containment devices must be made of materials compatible with each waste, and they must be free of leaks. The waste storage room may act as secondary containment as long as the room has been constructed to safely and effectively contain a hazardous waste spill.

5.11.7.1. Secondary containers must exceed the total volume of the largest container stored in each containment device for indoor storage.

5.11.8. Compatibility of wastes must be considered in arranging storage areas. For example, acid waste should never be stored adjacent to basic waste, particularly cyanide wastes. Further examples are outlined in 40 CFR 264, Appendix V.

5.11.9. The hazardous waste staging area is controlled so unauthorized personnel are not able to access the room or contents.

5.11.10. The maximum volume of acutely hazardous waste (e.g., P-listed wastes) that can be accumulated in the laboratory is one quart. The volumetric measurement of one quart is based upon container size in which the waste is stored and not the actual amount (volume) of waste present. An example of how this one quart limit can inadvertently be exceeded involves the disposal of a neat standard of 2,4-dinitrophenol into a one gallon bottle. While the neat standard itself may only constitute 1-2mL, the volume as defined under RCRA would be one gallon, thus the laboratory would be out of compliance.

5.12. Waste Documentation and Reporting

5.12.1. All drums containing hazardous waste are recorded in a database. The information contained in this database is useful when filling out EPA biennial reports and for retaining an accurate description of how much waste has been accumulated. The following information is entered into the logbook/database;

5.12.1.1. The drum number or waste stream identification;

5.12.1.2. The drum capacity (e.g., 55-gallon, etc.);

5.12.1.3. The manifest number associated with the drum's disposal.

5.12.2. The following hazardous waste records must be maintained for a minimum of five years:

5.12.2.1. Drum tracking logs;

5.12.2.2. Sample Reports;

5.12.2.3. Sample disposal information and waste records on computer disc;

5.12.2.4. Analytical records relating to sample waste stream profiling and characterization;

5.12.2.5. Labpack inventory logs;

5.12.2.6. Biennial Reports, Exception Reports, or other reports filed for compliance reasons;

5.12.2.7. Records related to unresolved enforcement action must be retained indefinitely until such a time that the matter is resolved;

5.12.2.8. Facility Certificates of Destruction or Recycling.

5.12.3. A Waste Manifest is the documentation form that must accompany all shipments of hazardous waste while in transit.

5.12.3.1. The manifest for hazardous waste must be signed and dated by a DOT-trained Pace employee responsible for the shipment and by the transporter. The transporter will leavea "two-signature page" copy of the manifest.

5.12.3.2. Within 35 days you will receive a three-signature page (generator, transporter, facility) showing the waste reached its intended destination. Alternatively, the three-signature page will be made available by the waste disposal company through the online account.

5.12.3.3. All manifests must be kept for a minimum of three years.

5.12.4. The central accumulation staging room must have a documented inspection weekly and satellite waste containers must have documented inspection as part of the monthly laboratory inspection. The inspections should ensure all regulations are obeyed.

5.12.4.1. A record of the inspections must be kept in an inspection log or summary.

5.12.4.2. Records must be maintained for at least three years from the date of inspection. At a minimum, the records must indicate:

5.12.4.2.1. The date and time of the inspection;

5.12.4.2.2. The name and signature of the inspector (typically will be Hazardous Waste Coordinator);

5.12.4.2.3. A notation of the observations made (can be in a check-off format, e.g., fire extinguisher: charged _X_ requires recharging __);

5.12.4.2.4. The date and nature of any repairs or other remedial actions.

5.12.5. Annual Generation Reports are required to be filed with the State of Indiana.

6. Training, Expectations, and Supplemental Information

6.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

6.2. **Equipment and Supplies -** The following equipment is mandatory under RCRA guidelines unless otherwise denoted. Periodic review (not to exceed monthly) of availability of equipment and supplies below should be conducted to maintain an adequate and viable supply.

6.2.1. **Chemical Spill Control Neutralizers**: The waste room stores three types of bulk dry spill neutralizers: solvent, acid and base. They may be utilized by placing the dry neutralizer onto a liquid chemical spill. Neutralization is indicated by a prevalent color change.

6.2.2. **Communication Device**: Required for emergency notification of spill, fire, etc.

6.2.3. **Drums**: Common types of waste drums used for storing and shipping hazardous wastes are polyethylene, steel-polyethylene lined, and steel. Sizes are typically 5gal, 15gal, 30gal, and 55gal. Drums used for liquids typically are closed top with an opening to pour the solvent through a funnel, while drums used for solids or lab packs are open-top. The UN rating for all containers must be suitable if the waste is to be transported under DOT regulations.

6.2.4. **Emergency Drench Shower**: Shower should deliver water approximately twenty gallons per minute with a non-interruptible flow. It may be turned on by pulling the shower handle down. It may be turned off by pushing the handle back to the 'off' position.

6.2.5. **Emergency Lighting** (as needed): The waste room is outfitted with emergency lighting that goes on if power fails.

6.2.6. **Exit Signs** (as needed): Exit signs are provided on all waste room doors. These signs are self-illuminating.

6.2.7. **Fire Alarm Pull Station/Evacuation Alarm**: A fire alarm pull station must be in close proximity to the hazardous waste room. The alarm may be activated by pulling the switch. Other alarm systems may be utilized as long as all personnel are trained on the procedures and the process can effectively notify facility employees of an emergency.

6.2.8. **Fire Extinguisher**: An extinguisher with a rating appropriate to the waste being stored in the area must be in close proximity to the hazardous waste room.

6.2.9. **Labels**: A multitude of labels are provided to ensure compliant labeling. They may be purchased or prepared manually.

6.2.10. **Liquid Chemical Neutralizers**: Liquid chemical neutralizers (base and acid) may be used to neutralize a contained hazardous liquid. This may be done by slowly adding the neutralizer to the liquid.

6.2.11. **Spill Control Pads**: Spill pads are used to soak up hazardous liquids. They do not neutralize spills. They are especially effective for cleaning up oily materials. Various pads are available for aqueous and petroleum based liquids.

6.2.12. **Spill Control Pillows**: Spill pillows may be used to soak up large amounts of liquid chemical spills. No neutralization occurs.

6.2.13. **Spill Dikes**: vary depending on the size and type of room: Their purpose is to encircle a spill, barring the spread of a hazardous chemical. They will also absorb liquids, but do not neutralize spills.

6.3. Operation of Waste Disposal Equipment

6.3.1. Specific Safety Warnings

6.3.1.1. **Failure to connect the proper voltages to the equipment may result in personal injury or equipment damage.**

6.3.1.2. **Failure to follow installation instructions may result in personal injury or equipment damage.**

6.3.1.3. **Do not place solid foreign articles such as wood or metal into the glass crusher as they could cause it to malfunction.**

6.3.1.4. **Eye protection and hearing protection must be worn at all times when operating waste disposal equipment.**

6.3.1.5. **Never wear loose fitting clothing, such as neckties, necklaces or scarves when operating waste disposal equipment. Gloves should be worn at all times when operating waste disposal equipment.**

6.3.1.6. **Always keep work area clean. Spills and/or debris on the floor may cause someone to fall against equipment causing personal injury or equipment damage.**

6.3.2. Glass Crusher Operation

6.3.2.1. When placing the glass crusher onto the 55-gallon drum, make sure the drum is stable and level. The drum should be free of defects or holes to insure proper fitting of the crusher. The area chosen for the location of the crusher must allow for airflow around the equipment as well as clearance for the feeding of containers into the machine.

6.3.2.2. The crusher is located in the waste disposal area under a ventilation hood.The unit requires a 115 volt AC receptacle. No lubrication of the equipment is required.

6.3.2.3. The chamber of the glass crusher can be cleaned with water and soap but care must be taken not to put water directly on the electrical controls and the machine should be unplugged before cleaning.

6.3.2.4. To operate, attach the crusher to the drum and plug in the cord. Turn on the motor's power switch and feed waste material into the feeder. If rubber flaps become worn or torn, they should be replaced.

6.3.3. **Trash Compactor Operation**

6.3.3.1. **NOTE: Always stay clear of all moving parts of the compactor.**

6.3.3.2. Carefully raise the overhead door.

6.3.3.3. Open the safety gate and carefully place waste into the receiving compartment of the compactor. Lighter waste items can be added over the top of the safety gate without opening it.

6.3.3.4. Do not overfill the receiving compartment.

6.3.3.5. Ensure that the safety gate is closed and securely latched prior to activating the compactor ram.

6.3.3.6. Press the green Start button to activate the compactor. The unit will make one complete cycle and then stop. Repeat the cycle if necessary.

6.3.3.7. Press the red Stop button to stop the compactor ram in mid-cycle. Press the black Reverse button to reverse the ram.

6.3.3.8. Close the overhead door when compactor is not in use.

6.3.3.9. Contact Republic Services for container removal/disposal when the compactor pressure gauge reads 1100-1300 psi.

6.4. **Attachments**

6.4.1. Attachment I: RCRA Requirements for Labs as a Function of Generator Status.

6.4.2. Attachment II: Hazard Codes for Common F-List Wastes (solvents).

6.4.3. Attachment III: TCLP Contaminant List with Concentration Limits.

6.4.4. Attachment IV: Hazardous Waste Label for Accumulation Drum (example).

6.4.5. Attachment V: Satellite Container Inspection Form (example).

6.4.6. Attachment VI: Waste Accumulation Room Inspection Form (example).

7. References

7.1. Pace Chemical Hygiene/Safety Manual-most current version.

7.2. Pace Quality Assurance Manual- most current version.

7.3. National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.

7.4. The NELAC Institute (TNI) Standard- most current version applicable to each lab.

7.5. Department of Defense (DoD) Quality Systems Manual- most current version.

8. Revisions

Document Number	Reason for Change	Date
S-IN-S-002-rev.01	1. Section 8: added sharps containers and pH strips. 2. Section 9: added acid and base for neutralization 3. Section 11: added PCB, non-biohazardous sharps, and miscellaneous lab waste streams. Added neutralization procedure for dilute caustic samples. Added instructions for the operation of waste disposal equipment.	11Jul2011
S-IN-S-002-rev.02	1. Converted to Corporate SOT format. 2. Cover page: added lab header, revised effective date format and revised document control format. 3. Section 9: added dust masks, hearing protection and sharps container. 4. Section 12: added waste stream information for Methanol Water 5. Section 26: removed manifest cover sheet for local version of SOP.	01Jan2016
S-IN-W-002-rev.03	1. Adapted from SOT-ALL-W-002-rev.07. 2. Section 5.1.3: added lab's EPA ID number. 3. Section 5.2.1: added lab's status as a SQG. 4. Section 5.4: added waste stream info for PCBs, Methanol and Miscellaneous. 5. Section 5.8.1.2: added lab's process for identifying hazardous samples. 6. Section 5.9: added lab's process for disposal of non-hazardous solids. 7. Section 5.10.3: described lab's process for neutralizing acidic waste. 8. Section 5.10.4: described lab's process for neutralizing basic waste. 9. Section 5.12.3.2.: added alternative to receiving 3-signature page by mail. 10. Section 6.3: added section to describe operation of waste disposal equipment. 11. Section 6.4: removed Hazardous Waste Manifest Cover Sheet.	23Mar2017

Attachment I: RCRA Requirements for Labs as a Function of Generator Status

Requirement (40CFR)	CESQG	SQG	LQG
Waste Determination (262.11)	Applicable	Applicable	Applicable
Generation Rate Limits (261.5 and 262.34)	<100 kg/mo	100-1,000 kg/mo	1,000 kg/mo or greater
Accumulation Quantity Limit w/o Permit (261.5 and 262.34)	Not to exceed 1,000 kg at any time. Not to exceed 1 kg acute at any time	not to exceed 6,000 kg at any time	No limit
Accumulation Time (261.5 and 262.34)	No limit	180 days or 270 if waste is to be transported over 200 miles.	90 days
EPA ID Number (262.12)	Not required***; possible state requirement	Required	Required
Mark Containers with Start Date (262.34)	Not applicable	Applicable	Applicable
Mark Containers "Hazardous Waste" (262.34(a))	Not applicable	Applicable	Applicable
Air Emission Standards 40 CFR 265 Subpart CC	Not applicable	Not applicable	Applicable
Satellite Accumulation (262.34(c))	Not applicable	Applicable	Applicable
Use Manifests (262, Subpart B)	Not required; possible state requirement	Required	Required
Exception Reporting (262.42)	Not required	Required after 60 days. No TSDF notification requirement.	Required after 45 days. Notification of TSDF within 35 days.
Biennial Report (262.41)	Not required	Not required; possible state requirement	Required
Contingency Plan (265, Subpart D)	Not required, but OSHA (29 CFR 1910.38) requires emergency planning	Basic planning required in accordance with the standards in 262.34(d)(4) and (5) and 265, Subpart C as well as OSHA regulations	Full written plan in accordance with 265 Subpart D, is required by 262.34(a)(4) and OSHA regulations
RCRA Personnel Training (262.34 and 265.16)	Not required, but recommended	Basic training required by 262.34(d)(5)(iii)	Full compliance with the training requirements in 265.16 is required by 262.34(a)(4)
Storage Requirements (without permit) (262.34 and 265)	None, but OSHA regulations under 29 CFR 1910, Subparts H and N, apply, particularly 29 CFR 1910.106	Compliance with technical standards in Part 265, Subparts I and J; for containers and tanks is required by 262.34(d)(2) and (3) and OSHA regulations	Compliance with technical standards in Part 265, Subparts I, J, W, and DD, is required by 262.34(a)(1) and OSHA regulations
Recordkeeping Requirements (262.40)	Waste determinations and generation log required (notification of regulated waste activity, training records, manifests, and land disposal restriction notifications recommended)	Notification of regulated waste activity, waste determinations, generation log, manifests, land disposal restriction notifications, exception reports, and correspondence with local emergency responders (written contingency plan, weekly container inspection & periodic equipment maintenance logs, and RCRA training records recommended)	Notification of regulated waste activity, waste determinations, generation log, manifests, land disposal restriction notifications, exception reports, biennial reports, correspondence with local emergency responders, RCRA training records, and written contingency plan required (weekly container inspection is required & periodic equipment maintenance logs is recommended)
Waste "Designated Facility"	State-approved or RCRA permitted facility or legitimate recycler	RCRA-permitted facility or legitimate recycler	RCRA-permitted facility or legitimate recycler
Land Disposal Restrictions (268.7)	Possible state requirement	Applicable	Applicable

Attachment II: Common F-Listed Solvents

Waste Name	Hazardous Waste Code(s)		Waste Name	Hazardous Waste Code(s)
Acetone	F003		Methylene Chloride	F001, F002
Benzene	F005		Methyl ethyl ketone (MEK)	F005
iso-Butanol	F005		Methyl isobutyl ketone	F003
n-Butyl alcohol	F003		Nitrobenzene	F004
Carbon Disulfide	F005		2-Nitropropane	F005
Carbon Tetrachloride	F001		Orthodichlorobenzene	F002
Chlorobenzene	F002		Pyridine	F005
Chlorinated fluorocarbons (CFC)s	F001		Tetrachloroethylene	F001, F002
Cresols	F004		Toluene	F005
Cresylic acid	F004		1,1,1-Trichloroethane	F001, F002
Cyclohexanone	F003		1,1,2-Trichloeoethane	F002
2-Ethoxyethanol	F005		1,1,2-Trichloro-1,2,2-trifluoroethane	F002
Ethyl acetate	F003		Trichloroethylene	F001, F002
Ethyl benzene	F003		Trichloroflourormethane	F002
Ethyl ether	F003		Xylene	F003
Methanol	F003			

Attachment III: TCLP Contaminant List

Waste ID #	Contaminant	Conc (mg/L)
D004	Arsenic	5.0
D005	Barium	100.0
D006	Cadmium	1.0
D007	Chromium	5.0
D008	Lead	5.0
D009	Mercury	0.2
D010	Selenium	1.0
D011	Silver	5.0
D012	Endrin	0.02
D013	Lindane	0.4
D014	Methoxychlor	10.0
D015	Toxaphene	0.5
D016	2,4-D	10.0
D017	2,4,5-TP Silvex	1.0
D018	Benzene	0.5
D019	Carbon Tetrachloride	0.5
D020	Chlordane	0.03
D021	Chlorobenzene	100.0
D022	Chloroform	6.0
D023	o-Cresol	200.0
D024	m-Cresol	200.0
D025	p-Cresol	200.0
D026	Cresol	200.0
D027	1,4-Dichlorobenzene	7.5
D028	1,2-Dichloroethane	0.5
D029	1,1-Dichloroethylene	0.7
D030	2,4-Dinitrotoluene	0.13
D031	Heptachlor	0.008
D032	Hexachlorobenzene	0.13
D033	Hexachlorobutadiene	0.5
D034	Hexachloroethane	3.0
D035	Methyl ethyl ketone	200.0
D036	Nitrobenzene	2.0
D037	Pentachlorophenol	100.0
D038	Pyridine	5.0
D039	Tetrachloroethylene	0.7
D040	Trichlorethylene	0.5
D041	2,4,5-Trichlorophenol	400.0
D042	2,4,6-Trichlorophenol	2.0
D043	Vinyl Chloride	0.2

Attachment IV: Hazardous Waste Label for Accumulation Drum (Example)

Attachment V: SatelliteContainer Inspection Form

Waste Container ID	Clearly Labeled as "Hazardous Waste" with Waste Stream	Liquid Waste has Secondary Containment	Closed when not in use
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No

If any of the above fields are a "NO", please document how the container was brought back into compliance.

Comments: ____

____

____

Inspector Signature ______________________ **Date:** ____________

Reviewer Signature ______________________ **Date:** ____________

ATTACHMENT VI: WASTE ACCUMULATION ROOM INSPECTION FORM

Containers closed when not in use	**Labels Easily Seen and Legible**	**Drums have Accumulation Start Date**	**Storage Amounts and Limits Obeyed**[1]	**Secondary Containment**for Liquid Waste	**Adequate Aisle Space**	**Available Emergency Equip. and Materials**	**Signature and Date of Inspection**	**Corrective Action for NO Answers**
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		

1: Accumulation limits are 90 days for LQG, and 180 days for SQG. SQG may have no more than 6000kg waste at any time.

ATTACHMENT C-22

WASTE MANAGEMENT TRAINING REQUIREMENTS
PACE, INDIANAPOLIS

Pace Analytical Services
7726 Moller Road
Indianapolis, IN 46268
Phone: 317.228.3100
Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

WASTE MANAGEMENT TRAINING REQUIREMENTS

Reference Methods: N/A

SOP Number:	S-IN-W-003-rev.03
Effective Date:	April 10, 2017
Supersedes:	S-IN-S-003-rev.02
SOP Template Number:	SOT-ALL-W-003-rev.05

APPROVALS

Laboratory General Manager — March 28, 2017 Date

Laboratory Quality Manager — March 24, 2017 Date

Laboratory Waste Coordinator — March 27, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1. The purpose of this Standard Operating Procedure (SOP) is to detail the procedures for training all employees in waste management. This SOP outlines the training requirements for each job title with regards to waste management.

2. Summary of Method

2.1. This SOP is intended to provide compliance assistance regarding waste management training. The SOP categorizes the waste management roles into different levels. The level classification of an employee determines the amount of training required.

2.2. Pace Analytical Services designates a Hazardous Waste Coordinator at each location as the qualified person to oversee the waste program of the laboratory. The waste program not only involves the act of correctly classifying and managing waste, but also adequately training all employees involved in waste management. The Hazardous Waste Coordinator must ensure that all applicable county, state, or other local requirements are obeyed.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP apply to all Hazardous Waste Coordinators.

3.2. This SOP relates only to training required for waste management, and obeying this SOP does not guarantee compliance in other areas, such as safety, quality, etc.

3.3. **Parameters**: Not applicable to this SOP.

4. Definitions

4.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

4.2. **Hazardous Waste Coordinator (HWC) –** The Pace employee responsible for creating, guiding, and implementing all hazardous waste management operations at their location.

4.3. **Emergency Coordinator** – The Pace employee responsible for updating and directing the local emergency/contingency plan.

5. Procedure

5.1. Determine waste management training needed by job title and responsibilities.

5.1.1. The job titles and roles for consideration are as follows (these titles include all qualifiers of that position, for example I, II, III, IV, Senior, Intern, Assistant, Lead, etc.):

Laboratory Technician	Sales/Marketing
Laboratory Analyst	Administrative
Supervisor/Managers	Corporate Directors/Officers
Client Services Technicians/Supervisors	Human Resources
Safety Officer	Project Managers/Coordinators
Field Analyst/Field Tech	Information Technology
Hazardous Waste Coordinator	Emergency Coordinator
Environmental Technician	Instrument Services Specialist

5.1.2. **Client Services Technician/Supervisor job description** (with regards to waste management):

5.1.2.1. Waste handling duties to include: labeling containers, understanding storage time limits, proper satellite accumulation, ensure adequate space between waste containers, ensure all waste containers are closed and incompatible materials are stored separately, understand secondary containment, and may include documenting weekly inspections. Other duties may include sample disposal which involves neutralizing and sewering waste, drumming up remaining RCRA and non-RCRA solids, and drumming RCRA Liquids for transport to final disposal facility.

5.1.2.2. Department supervisors and managers related to personnel in sample receiving have the same job description and are responsible for the same job duties.

5.1.3. **Laboratory Technician and Laboratory Analyst job description** (with regards to waste management):

5.1.3.1. Waste handling duties to include: labeling containers, understanding storage time limits, proper satellite accumulation, ensure adequate space between waste containers, ensure all waste containers are closed and incompatible materials are stored separately, understand secondary containment, and may include documenting weekly inspections.

5.1.3.2. Department supervisors and managers related to personnel in each area of the laboratory have the same job description and are responsible for the same duties.

5.1.4. **Emergency Coordinator job description** (with regards to waste management):

5.1.4.1. Duties include maintaining the contingency plan; activating internal alarms and oversee evacuation of entire facility; contact the fire department and/or police and/or ambulance service; notify proper local, state and federal agencies related to emergencies; direct all personnel performing emergency and clean up functions; assemble and direct any on-site emergency responders; maintain all emergency supplies on site; update emergency maps; and other duties outlined in Pace policies and SOP related to safety and other job related duties.

5.1.5. **Hazardous Waste Coordinator job description** (with regards to waste management):

5.1.5.1. Duties include evaluating waste streams; understanding licensing requirements; understanding proper labeling and storage of waste containers; select appropriate transportation and disposal companies; maintain complete and thorough records including manifests and training; implement waste minimization and Pollution Prevention policies; train personnel on the job duties outlined in 5.1.2 and 5.1.3; follow all Pace policies and SOPs related to safety and waste in addition to other responsibilities related to any additional roles held at Pace.

5.2. Determine the level of training required for employee. If an employee has multiple roles or a title that falls between different levels, always take the highest level training requirements. If an employee has a title in one level, but has responsibilities at another level, always assign the highest level training requirements.

5.2.1. Level One – positions that do not require any chemical or hazardous waste contact and do not have a job description related to hazardous waste management:

5.2.1.1. Administrative Positions (including Quality Managers and Senior Quality Managers);

5.2.1.2. Sales/Marketing Positions;

5.2.1.3. Human Resources Positions;

5.2.1.4. Corporate Directors/Officers;

5.2.1.5. Information Technology;

5.2.1.6. Project Managers/Coordinators.

5.2.1.7. Lab General Manager or Assistant General Manager.

5.2.2. Level Two – positions whose jobs require the transferring of wastes from satellite accumulation areas to final accumulation areas, access to a satellite accumulation area, or operating a solvent still, an evaporator or wastewater treatment unit:

5.2.2.1. Laboratory/Environmental Technician;

5.2.2.2. All Specialists, Analysts, and Chemists;

5.2.2.3. Field Analyst/Field Tech;

5.2.2.4. Sample Receiving;

5.2.2.5. Safety Officer;

5.2.2.6. Supervisors/Managers of employees listed in Level Two.

5.2.3. Level Three – positions whose jobs require inspecting accumulation areas/emergency equipment, clean-up to spills, response to emergencies, waste management training of new or relocated employees positions, filling out waste manifests and maintaining waste compliance of the facility:

5.2.3.1. Hazardous Waste Coordinator;

5.2.3.2. Emergency Coordinator.

5.3. Determine the Training Requirements – employees must receive documented training of the following topics:

5.3.1. Level One Training:

5.3.1.1. Read and understand local Pace Analytical Chemical Hygiene/Safety Plan;

5.3.1.2. Receive introductory orientation of facility and familiarization with all safety equipment and procedures for local emergency/contingency plans.

5.3.2. Level Two Training:

5.3.2.1. Everything required in Level One Training (5.3.1);

5.3.2.2. Hazardous Waste Management Training (for general employees). Training must be given within the first 90 days of employment, and annually thereafter. The following topics must be covered at minimum: License requirements; hazardous waste definitions and determination; satellite accumulation requirements and time limits; waste container labeling, inspections, and storage requirements; tank inspections and labeling; general awareness of manifest completion, copy distribution and land disposal restriction notices; record keeping regarding inspections, training, annual reports; waste reduction; and emergency response.

5.3.3. Level Three Training:

5.3.3.1. Everything required in Level Two Training (5.3.2);

5.3.3.2. DOT-HAZMAT Training, with refresher training no more than 3 years after prior training;

5.3.3.3. Hazardous Waste Management Training for Hazardous Waste Coordinators.

6. Training, Expectations, and Supplemental Information

6.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

7. References

7.1. Pace Chemical Hygiene/Safety Manual-most current version.

7.2. Pace Quality Assurance Manual- most current version.

7.3. National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.

7.4. The NELAC Institute (TNI) Standard- most current version applicable to each lab.

7.5. Department of Defense (DoD) Quality Systems Manual- most current version.

8. Revisions

Document Number	Reason for Change	Date
S-IN-S-003-rev.01	1. Section 11.1.4: added Safety Officer as optional Emergency Coordinator 2. Section 14: added Method Modifications section per TNI requirements.	01Feb2013
S-IN-S-003-rev.02	1. Re-formatted to Corporate SOT format. 2. Cover page: changed phone number, revised effective date format and revised document control format.	22Dec2015
S-IN-W-003-rev.03	1. Adapted from SOT-ALL-W-003-rev.05. 2. Changed SOP name from a “Safety” SOP to a “Waste” SOP. 3. Cover page: changed phone number and document control format. 4. Added Table of Contents	15Mar2017

ATTACHMENT C-23

ANALYSIS OF SAMPLES FOR ALPHA EMITTING ACTINIDES AND PU-241
PACE, PITTSBURGH

Document Information

Document Number: ENV-SOP-GBUR-0068 **Revision:** 00

Document Title: Analysis of Samples for Alpha Emitting Actinides and Pu-241

Department(s): Rad Chem

Previous Document Number: S-PGH-R-008-rev.13

Date Information

Effective Date: 08 Feb 2018

Next Review Date: 08 Feb 2020 **Last Review Date:**

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Pace Analytical™

STANDARD OPERATING PROCEDURE

Analysis of Samples for Alpha Emitting Actinides and Plutonium-241

Methods: ASTM Method D-3972-90 and HASL 300 Method U-02

SOP NUMBER:	S-PGH-R-008-rev.13
REVIEW:	R. Kinney
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	PGH-R-008-12
REVIEW DATE:	Upon Procedural Change

APPROVALS

Department Manager/Supervisor — 02/08/18 Date

Senior Quality Manager — 02/08/18 Date

PERIODIC REVIEW
SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

This is **COPY#**_____ distributed on _____ by _____and is ___**CONTROLLED or** __**UNCONTROLLED.**

TABLE OF CONTENTS

1. Purpose

1.1 This SOP describes the procedure to be used for the determination of micro-quantities of Americium, Curium, Thorium, Plutonium (including Pu-241), Neptunium and Uranium in various sample matrices. It also addresses routinely difficult matrices such as filters, solids in quantities over two grams, and aqueous samples where precipitation as described in the procedure may not be successful.

1.2 Samples not meeting the criteria outlined in this procedure should be brought to the attention of the Department Manager/Supervisor or designee for further direction.

1.3 This procedure is applicable for determining compliance for isotopic uranium in drinking water and is substantially compliant with ASTM Method D-3972-90 and HASL 300 Method U-02. Deviations from these methods are addressed in Section 19 of this procedure.

1.4 This procedure is designed for the determination of the following radio-nuclides:

Isotope	Half-life	Alpha Energy MeV (Abnd.)
Americium 241	*458 yrs*	*5.49 (86%) 5.44 (13%)*
Americium 243	*7370 yrs*	*5.28 (88%) 5.23 (11%)*
Curium 242	*162.79 days*	*6.069 (25%) 6.112 (74%)*
Curium 244	*18 yrs*	*5.80 (76%) 5.76 (23%)*
Plutonium 238	*87.8 yrs*	*5.50 (72%) 5.46 (28%)*
Plutonium 239	*24131 yrs*	*5.16 (73%) 5.14 (15%)*
Plutonium 240	*6569 yrs*	*5.17 (74%) 5.12 (26%)*
Plutonium 241	*14.35 yrs*	*Beta 20.81Kev (5.23 Ave)*
Plutonium 242	*375850 yrs*	*4.90 (78%) 4.86 (22%)*
Neptunium 237	*2140000 yrs*	*4.79 (47%) 4.77 (25%)*
Thorium 228	*1.9132 yrs*	*5.42 (73%) 5.34 (27%)*
Thorium 230	*77000 yrs*	*4.69 (76%) 4.62 (23%)*
Thorium 232	*14050000000 yrs*	*4.01 (77%) 3.95 (23%)*
Thorium 229	*7340 yrs*	*4.85 (56%) 4.90 (10%)*
Thorium 227	*18.718 days*	*6.03 (24%), 5.98 (23%), 5.76 (20%)*
Uranium 232	*72 yrs*	*5.32 (69%) 5.26 (31%)*
Uranium 238	*4468000000 yrs*	*4.19 (77%) 4.15 (23%)*
Uranium 235	*703800000 yrs*	*4.39 (55%) 4.36 (11%)*
Uranium 236	*23415000 yrs*	*4.49 (74%) 4.45 (26%)*
Uranium 234	*244500 yrs*	*4.78 (72%) 4.72 (27%)*
Uranium 233	*159200 yrs*	*4.82 (84%) 4.78 (13%)*

2. Scope and Application

2.1 This procedure covers the measurement of various isotopic alpha emitters in various matrices including drinking water. Since all plutonium isotopes behave the same, this procedure also addresses the isolation of beta emitting Pu-241. With the exception of drinking water sources, most other matrices, especially soils, contain elements, which may complex some actinides, making it necessary to aggressively treat such samples.

2.2 This method is a laboratory promulgated method based on multiple accredited methods and the behavior of individual alpha emitters.

2.3 Sample results are decay corrected to the client supplied collection date and time for all analytes reported using this SOP.

2.4 Alpha count sources generated as the product of this SOP are analyzed as documented in the current revision of Pace SOP PGH-R-020, "Alpha Spectroscopy Instrument Operations."

2.5 Plutonium-241 count sources generated as the product of this SOP are analyzed as documented in the current revision of Pace SOP PGH-R-022, "Liquid Scintillation Counter Operations."

2.6 Beta-emitting tracer sources produced from application of this SOP are analyzed for yield determination as documented in the current revision of Pace SOP PGH-R-002, "Gas Flow Proportional Counter Operation."

3. Summary of Method

3.1 The nuclides listed in Section 1 are first separated from the interfering substances by iron hydroxide precipitation, and appropriately dissolved in either a hydrochloric acid or nitric acid solution. Uranium and Plutonium separation can be accomplished using an anion exchange column. Subsequent separation and purification of Americium and Curium are accomplished using Tru-Resin™ columns. For Thorium and Neptunium, separation can be accomplished using anion exchange columns. Additionally, Americium/Curium spectral resolution can be improved by using TEVA-Resin™ columns.

3.2 Following separation, the individual isotopes are micro-precipitated onto a filter and the corresponding alpha activity is determined using an alpha spectrometer.

3.3 Counting efficiency is determined by micro-precipitating a solution composed of three different energy range alpha standards (such as Cm-244, Pu-239, and Th-230) and counting the sources in each detector of an alpha spectrometry system or using a commercially prepared source with at least 3 different energy range nuclides.

4. Interferences

4.1 Any nuclide with an alpha emission similar in energy to the isotopes in question that cannot be separated from the target nuclide will interfere with this analysis.

4.1.1 It is not always necessary to re-prep samples where both sufficient tracer counts and an interfering nuclide are present.

4.1.2 A clean-up may be performed on the micro-precipitated source to remove the interfering nuclides.

4.1.3 Analysts should consult with the Department Manager/Supervisor or the specified designee on how to proceed.

4.2 Samples that contain isotopes employed as working tracers will lead to an overestimate of yield and low bias in measured results. If the sample activity is known, or can be determined, and the tracer activity corrected to reflect the true concentration of the tracer nuclide present in the sample, accurate results can be obtained.

4.3 Daughters of Th-229 will interfere with Cm-244 analysis. Thorium and americium should be run separately in order to exclude such interferences.

4.3.1 If sample volume limits running these analyses separately, Method 4 (Purification of Americium Using Teva-Resin™) must be performed, subsequent to Method 3 (Purification of Americium Using Tru-Resin™), to remove any thorium decay daughters from the americium/curium fraction.

4.4 Unless isolated separately, Neptunium-237 will interfere with the Pu-242 yield calculation, therefore Pu-236 should be used as a tracer when it is desirable to extract and count plutonium with neptunium on the same counting source.

4.5 Plutonium-236 decays to U-232, which in turn decays to Th-228, therefore it is important to know when the last U-232 separation was performed on the Pu-236 working tracer.

4.5.1 Analysts should be mindful of the potential for a bias in the uranium tracer recovery depending on the length of time since the separation of U-232 from the working tracer.

4.5.2 Plutonium and thorium can be run sequentially if Th-228 is not desired or thorium decay daughters were previously separated from the Pu-236 working tracer.

4.6 Excess fluoride during sample digestions may produce insoluble actinide complexes leading to low yields. Addition of saturated boric acid solution must be performed at the completion of sample digestion to avoid this.

4.7 Silica in solution may cause problems with uranium and thorium separations and analyte retention on columns.

4.8 Excess carbonate in solution will prevent the iron hydroxide precipitation of the uranium isotopes. To prevent this, it is extremely important to thoroughly boil water samples during the initial precipitation steps of uranium analysis.

4.9 The analysis volume for samples known to contain elevated concentrations of uranium should be controlled so as to limit the quantity of uranium carried through to the final source preparation. Excessive mass

contained in the final micro-precipitated count source may negatively affect peak resolution (FWHM) for which there are control limits required by this SOP.

5. Safety

5.1 Procedures must be carried out in a manner that protects the health and safety of all personnel. Since this analysis is for a radioactive constituent, the sample must be treated as radioactive.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3 When mixing or diluting acids, always add the acid slowly to water and swirl. Dilution of acids must always be done in a hood. Appropriate eye-protection, gloves, and a lab coat must be worn.

5.4 Exposure to radioactivity and chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous and/or non-radioactive, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 In order to minimize the potential for cross contamination of high and low levels of radioactive samples, good housekeeping and good laboratory practices are essential and must be strictly adhered to.

5.6 Organic samples of unknown content must be handled with extreme caution and under the direct instruction of the Department Manager or Manager-specified designee. Direct treatment of organic matrices with strong oxidizing chemicals such as nitric acid and/or hydrogen peroxide is strictly prohibited.

5.7 Hydrofluoric acid is particularly hazardous because a serious skin exposure may cause no immediate sensation of pain. The acid penetrates the skin and spreads internally, causing tissue damage deep under the skin. The resulting burn is painful, difficult to treat, and easily infected. Gloves must be checked for pinhole leaks before use. They must be rinsed before they are removed and must be discarded after use. HF burn gel shall be put on suspected HF burns after flushing (except the eyes) until medical help can be obtained. Medical attention shall be sought even if suspicions arise after working hours. Contact your group leader immediately for further information if a HF burn is suspected.

5.8 In addition, HF vapors are also hazardous. Exposure can cause permanent damage. Breathing HF vapors even for a short time and at a low temperature can be injurious to the respiratory system and even fatal. All such direct contact must be avoided.

5.9 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety

information can be obtained from the MSDS files maintained in the laboratory.

6. Definitions

6.1 Batch: For all analysis, an analytical batch contains 20 or fewer samples of a similar matrix, prepared at the same time, by the same analyst, using the same reagents.

6.2 SRM: Standard Reference Material.

6.3 Throughout this procedure, approximate weights and measures will be designated by the use of whole numbers when referring to masses exceeding 1g or volumes in excess of 1mL. Measurements of masses and volumes so designated can be made with top loading balances, graduated cylinders, etc. For approximate measures below 1g or 1mL, the word "approximately" must be used prior to the described mass or volume.

6.4 Throughout this procedure, exact or critical masses and volumes will be designated by the use of one or more decimal places. Measurements of weights and volumes so designated should be made with accurate analytical instruments such as analytical balances, calibrated pipettes, etc.

6.5 When aliquotting samples on a balance, the observed mass on the balance must be recorded in preparation logbooks to the lowest weight indicated on the balance. Sample aliquot masses must not be targeted. Once sample is removed from the sample container and transferred to a beaker, it must not be removed from the beaker.

6.6 The method utilized for obtaining the sample aliquot, whether on a balance, in a graduated cylinder, or by pipette, must be clearly annotated in the preparation logbook.

7. Responsibilities and Distribution

7.1 General Manager/Assistant General Manager (GM/AGM)

7.1.1 The GM/AGM has the overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the laboratory involving the collection and reporting of analytical data.

7.1.2 The GM/AGM and Senior Quality Manager/Quality Manager have final review and approval authority for all SOPs prepared within the laboratory.

7.2 Senior Quality Manager/Quality Manager (SQM/QM)

7.2.1 The SQM/QM will maintain a master file of all SOPs applicable to the operations departments.

7.2.2 The SQM/QM will assign a unique number to each SOP prepared prior to approval and distribution.

7.2.3 The SQM/QM will distribute SOPs to applicable personnel and maintain an accurate accounting of such distribution to ensure that the SOPs, in the hands of the users, are current and complete.

7.3 Department Manager/Supervisor

7.3.1 The Department Manager/Supervisor is responsible for ensuring all staff members read, follow, and are adequately trained in the use of the SOPs

7.3.2 The Department Manager/Supervisor coordinates the preparation and revision of all SOPs within the department whenever a procedure changes.

7.3.3 The Department Manager/Supervisor provides initial approval of all SOPs within the department.

7.3.4 The Department Manager/Supervisor makes recommendations for SOP revision to the SQM/QM via written memo.

7.4 Individual Staff

7.4.1 Individual staff members are responsible for adherence to the specific policies and procedures contained in the applicable SOPs.

7.4.2 Individual staff members will only use a signed, controlled copy of the SOP. Each person may make recommendations to the Department Manager/Supervisor for revising SOPs as the need arises.

7.4.3 Personnel are responsible for ensuring that any deviations from this SOP are reported to the Department Manager/Supervisor.

8. Sample Collection, Preservation, and Handling

8.1 Aqueous Samples

8.1.1 Containers used for sample collection must never be re-used. Either plastic or glass containers may be used for sample collection.

8.1.1.1 Aqueous samples must be preserved at the time of collection by adding enough concentrated (16N) HNO_3 to the sample to make the sample pH <2. Typically, 2mL of 16N HNO_3 per liter of sample is sufficient to obtain the desired pH. Samples must be preserved within five days of collection. If samples are collected without preservation, they must be received by the laboratory and preserved within five days of collection. Following preservation with acid, samples must be held in the original container for a minimum of 24 hours before analysis or transfer of sample. For samples preserved at the time of receipt, the pH must be re-checked by laboratory personnel prior to removing sample for analysis. The pH re-check date and time, the initials of the analyst verifying the pH, as well as any adjustments or notes regarding the preservation must be recorded in the pH Verification Logbook.

8.1.1.2 For dissolved analysis, samples must be filtered through a 0.45µm membrane filter and then preserved to a pH <2.

8.1.1.3 For total analysis, the sample is not filtered, but is preserved to a pH<2.

8.1.2 Refrigeration is not required for aqueous or solid samples, but is recommended for all biological samples.

8.1.4 The maximum hold time for samples analyzed by this procedure is 180 days between sample collection and sample analysis.

9. Equipment and Supplies

9.1 Multi-channel Analyzer:

9.2 Alpha Spectrometer: Refer to SOP PGH-R-020, current revision "Alpha Spectroscopy Instrument Operation" for instructions on alpha spectroscopy system operation.

9.3 Electric hot plate or griddle.

9.4 Ion exchange columns, 2mL, disposable from Eichrom or equivalent.

9.5 TRU and TEVA cartridges from Eichrom.

9.6 Vacuum box apparatus and applicable parts, disposable yellow and white tips, 10-50mL syringe barrels, etc from Eichrom or equivalent.

9.7 Polypropylene filters, 25mm, 0.1µm pore size from Environmental Express, or equivalent.

9.8 PTFE FEP Beakers, 100mL size and other assorted sizes, or equivalent.

9.9 Multi-port vacuum filtering apparatus for 25mm filters (referred to as the filter rig).

9.10 Miscellaneous glassware: beakers, watch glass covers, and stir rods.

9.11 Membrane Filters, 5.5cm diameter, 0.45µm pore size.

9.12 Analytical Balance: Sensitivity to 0.1mg, capacity 0 - 160g.

9.13 Top loader balance, sensitivity to 0.1g, capacity 0-2000g.

9.14 Vacuum filtration apparatus for 5.5cm diameter membrane filters.

9.15 General-purpose centrifuge and disposable centrifuge tubes, 50mL made of high-density polyethylene or equivalent.

9.16 Vortex mixer.

9.17 Liquid scintillation vials, glass.

9.18 Muffle oven capable of 105ºC to 550ºC, with or without ramping capabilities.

9.19 Software supplied with the instrument to control instrument operation. Refer to SOP PGH-R-020, current revision "Alpha Spectroscopy Instrument Operation" for applicable software details.

9.20 Computer capable of running the Alpha spectrometer Counter System software, monitor, mouse, keyboard, and printer. Refer to SOP PGH-R-020, current revision "Alpha Spectroscopy Instrument Operation" for computer hardware specifications.

10. Reagents and Standards

10.1 Reagents must be prepared from reagent grade chemicals, unless specified otherwise. Distilled or deionized (DI) water. ASTM Type II as produced using the specifications documented in SOP PGH-C-027, current revision. Consult the Safety Data Sheets for the properties of these reagents, and how to work with them.

10.2 Anion exchange resin, Bio-Rad AG 1x8 (100 - 200 mesh, Cl^- form) or equivalent. Slurry the resin in a squirt bottle with ASTM Type II DI water.

10.3 Ammonium Hydroxide, 15N, concentrated, sp. gr. 0.90, 56.6%.

10.4 Ammonium Thiocyanate, 6M: Dissolve 476g ammonium thiocyanate in 300mL of ASTM Type II DI water and dilute to 1.0L with ASTM Type II DI water.

10.4.1 CAUTION! The reaction is extremely exothermic and the bottle may become very slippery.

10.5 Ammonium Thiocyanate, 3M/ 0.1N Formic acid: Dilute 250mL 6M ammonium thiocyanate and 50mL 1.0N formic acid to 500mL with ASTM Type II DI water. Prepare fresh daily.

10.6 Ammonium Thiocyanate, 1M/ 0.1N Formic acid: Dilute 100mL 6M ammonium thiocyanate and 60mL 1.0N formic acid to 600mL with ASTM Type II DI water. Prepare fresh daily.

10.7 Ascorbic Acid, 1.0M: Dissolve 17.6g ascorbic acid into 50mL ASTM Type II DI water and dilute to 100mL with ASTM Type II DI water. Prepare weekly.

10.8 Boric Acid, 5% Saturated Solution: Add 50g granulated boric acid to 500mL ASTM Type II DI water and dilute to 1L with ASTM Type II DI water. The boric acid should not completely dissolve.

10.9 Distilled or deionized DI water. Resistance value between 0.5 and 2.0 Mmhos (2.0 to 0.5µohms/cm specific conductivity) at 25˚C.

10.10 Deionized Water adjusted to a pH of 10.0 by the addition of 6-8 drops of ammonium hydroxide to each 500mL of DI.

10.10.1 The pH should be checked prior to each addition of ammonium hydroxide, since it is not always necessary to add the full 6-8 drops each time pH 10 DI is made up.

10.11 Ethanol, 80%. 800mL of reagent grade alcohol mixed with 200mL of ASTM Type II DI water.

10.12 Formic acid, concentrated, 88%.

10.13 Formic acid, 1.0N: Dilute 42.5mL concentrated formic acid to 1.0L with ASTM Type II DI water.

10.14 Hydrochloric acid, 12N, concentrated, sp. gr. 1.19, 37%.

10.15 Hydrochloric acid, 9N / 0.10% Hydrogen Peroxide: For every 100mL of 9N HCl, add 0.1mL of 30% H_2O_2. Shake well and allow to sit for 5 minutes prior to use. Prepare daily.

10.16 Hydrochloric acid, 9N: Dilute 750mL concentrated 12N HCl to 1L with ASTM Type II DI water.

10.17 Hydrochloric acid, 6N: Dilute 500mL concentrated 12N HCl to 1L with ASTM Type II DI water.

10.18 Hydrochloric acid, 4N: Dilute 332mL concentrated 12N HCl to 1L with ASTM Type II DI water.

10.19 Hydrochloric acid, 0.1N: Dilute 8.3mL concentrated 12N HCl to 1L with ASTM Type II DI water.

10.20 Hydrochloric acid, 6N / 0.52N Hydrofluoric acid Solution: Dilute 500mL of concentrated 12N HCl and 18mL of concentrated HF to 1L with ASTM Type II DI water.

10.21 Hydrochloric acid, 9N / 0.05M Ammonium Iodide Solution: For every 100mL of 9N HCl, add 0.724g of NH_4I solid. Shake well and let stand for 5 minutes prior to use. Prepare daily.

10.22 Hydrazine Dihydrochloride, 25% Solution: 25g hydrazine dihydrochloride dissolved in 75mL of ASTM Type II DI water.

10.23 Hydrogen Peroxide (30%) H_2O_2.

10.24 Hydrofluoric acid, 29N, concentrated, sp. gr. 1.18, 49%. Must be stored in a plastic container.

10.25 0.1% Hydrofluoric acid: Dilute 1.0mL concentrated HF to 1L with DI.

10.26 Iron Carrier: 10.0mg Fe/ml: Dissolve 72.4 g $[Fe(NO_3)_3]\bullet 9\ H_2O$ in 500mL of ASTM Type II DI water and dilute to 1L with ASTM Type II DI water.

10.27 Neodymium carrier (0.5mg/mL): Dilute 5.0mL of 10mg/mL of neodymium carrier to 100mL of ASTM Type II DI water.

10.28 Nitric acid, 16N, concentrated, sp. gr. 1.42, 70%.

10.29 Nitric acid, 2N: Dilute 125mL of conc. 16N HNO_3 to 1L with DI water.

10.30 Nitric acid, 8N: Dilute 500mL of conc. 16N HNO_3 to 1L with DI water.

10.31 Nitric acid, 2N / 0.5M $Al(NO_3)_3$: Dissolve 188g aluminum nitrate nonahydrate in 500mL of ASTM Type II DI water. Add 125mL conc. 16N HNO_3 to the solution and then dilute to 1L with ASTM Type II DI water.

10.32 Nitromethane, ACS reagent.

10.33 Potassium Thiocyanate Solution, 0.1N: purchased ready made from Fisher Scientific, or equivalent.

10.34 Tru-Resin™: Eichrom Inc., Mix 100g (s-grade) of Tru-Resin with 428mL ASTM Type II DI water and 2mL of conc. 16N HNO_3.

10.35 TEVA-Resin™: Eichrom Inc., Mix 100g (s-grade) of TEVA-Resin with 428mL of ASTM Type II DI water and 2mL conc. 16N HNO_3.

10.36 Titanium (III) Chloride: 10% wt. Solution.

10.37 Standards

10.37.1 Tracer: A solution containing Am-243, Np-239 (prepared from Am-243), Pu-242, Pu-236, Th-229, Th-234 (prepared from U-238), or U-232 prepared from a NIST-traceable and certified solution, or equivalent. Tracer spike aliquots shall have DPM values consistent with the activity of the samples. Check the certificate for the expiration date.

10.37.2 Laboratory Control Sample (LCS)

10.37.2.1 Liquid: A solution of Am-241, Cm-244, Pu-239, Pu-241, Np-237, Th-230, or U-238 prepared from a NIST-traceable and certified source, or equivalent. Check the calibration certificate for the expiration date.

10.37.2.2 Solid: A soil containing Am-241, Cm-244, Pu-239, Pu-241, Np-237, Th-230, or U-238 prepared from a NIST-traceable and certified source, or equivalent. Check the certificate for the expiration date.

Note: Equivalent is defined as being traceable to any international source that provides a certificate of calibration. As the project requires, a solution may be used where the isotopic activity has been confirmed by multiple laboratory analyses.

11. Calibration

11.1 Plated sources can be purchased from a NIST supplier, but they must have the same size effective area and they must be positionable so the effective area is the same distance from the detector as those sources prepared for sample counting.

11.2 If plated sources cannot be purchased, sources can be prepared using NIST traceable standards as follows.

11.3 Sources for alpha spectroscopy system calibration must be prepared in a fashion that will ensure >99% recovery of each calibration source analyte.

11.4 Concentrated standards or SRMs that limit the volume of the standard to less than 0.5mL per radioisotope must be used in order to ensure that the acid concentration is kept as dilute as practical. Quantitative precipitation of many actinides from acid solutions with concentrations greater than 0.5N is impossible and will lead to erroneous results.

11.5 Transfer an appropriate volume or mass of analytical standard to a labeled, disposable centrifuge tube. Record the standard numbers utilized and mass or volume information in the appropriate standard dilution logbook. Dilute the combination of standards to 20mL with 0.1N HCl.

11.6 Add the following to the solution in the centrifuge tube:

11.6.1 0.1mL of neodymnium carrier solution (0.5mg/mL)

11.6.2 Four drops of the iron carrier solution.

11.6.3 Seven drops of the 1M ascorbic acid solution

11.6.3.1 This reduces the iron to the +2 state.

11.6.3.2 Proper reduction of iron will result in a clear, colorless solution.

11.7 Cap each tube and swirl the solution to mix the contents. Place tubes in a warm water bath to ensure proper equilibration between the carrier and the radioisotopes.

11.8 After five minutes, add 3mL concentrated HF acid to each source and swirl the source to mix the contents. Let the sources sit in the warm water bath for 15 minutes, then remove them, and allow them to cool for 15 minutes.

11.9 Prepare a filter apparatus by placing a filter funnel into an HDPE vacuum flask. Turn on the vacuum and rinse the apparatus with several mLs of ASTM Type II DI water.

11.10 Carefully wash and rinse a filter funnel that is dedicated to, and set aside for, the preparation of calibration sources.

11.11 Place a polypropylene filter onto the filtering apparatus and ensure it is centered.

11.12 Place the clean filter funnel on the rig and secure it, being careful not to rip the filter in the process.

11.13 Rinse the inside surface of each funnel with the 80% ethanol solution then rinse the inside surface of each funnel with approximately 10mL of ASTM Type II DI water.

11.14 Transfer the calibration solution to the filter funnel and allow it to pass through the filter. After all of the calibration solution has passed through the filter, rinse the centrifuge tube with approximately 5mL of the 80% ethanol solution and transfer it to the filter funnel as a rinse.

11.15 Rinse the inside of the funnel with an additional 2-3mL of the 80% ethanol solution.

11.16 Label the metal backside of a one inch counting disk with the appropriate standard number as determined from the standard preparation logbook, and remove the paper backing from the pre-taped side of the disk.

11.17 Remove the filter funnel and carefully remove the calibration filter from the filtration rig and affix it to the disk.

11.18 Allow the disk to air dry for a minimum of 15 minutes.

11.19 When the calibration source has dried, place it in a labeled petri dish and submit it to the count room for counting.

11.20 Perform calibration of alpha spectrometry detectors as specified in the current version of Pace SOP PGH-R-020, “Alpha Spectroscopy Instrument Operations”. Count sources long enough to obtain a minimum of 10000 net counts in each of the three nuclides regions of interest.

11.21 Transfer the contents of the vacuum flask to a 600mL PTFE beaker.

11.21.1 Rinse the flask three times with 50mL of ASTM Type II DI water and add the rinses to the beaker.

11.22 Evaporate the solution to dryness on a hotplate on a low to medium heat setting.

11.23 Carefully add 10mL of concentrated nitric acid and 1mL of 30% hydrogen peroxide solution to the beaker and evaporate the solution to dryness.

11.24 Carefully add 10mL of concentrated nitric acid and 1mL of 5% boric acid solution to the beaker and evaporate the solution to dryness.

11.25 Dissolve the residue into 20mL of 8N nitric acid solution by heating the solution.

11.26 Allow the solution to cool, then transfer it to a 100mL volumetric flask.

11.26.1 Rinse the beaker three times with 10mL of ASTM Type II DI water and add the rinses to the volumetric flask.

11.26.2 Dilute the solution to the mark with ASTM Type II DI water.

11.26.3 Transfer the diluted calibration effluent to a labeled HDPE bottle.

11.27 Analyze 10mL of each calibration effluent sample for gross alpha content using the most recent revision of SOP PGH-R-001.

11.27.1 Calculate the total alpha content for the entire source effluent.

11.27.2 Calculate the total alpha activity of each calibration source.

11.27.3 Compare the total effluent activity to the theoretical activity to ensure that the effluent contains less than 1% of the total activity. Calibration sources must contain >99% of each analyte in order to be accepted for calibration purposes.

11.27.4 Consult with the Department Manager/Supervisor or specified designee if the sources are not acceptable.

11.28 The Plutonium-241 calibration is performed separately on a liquid scintillation counter. Add 1000 to 2000 dpm of Pu-241 by mass to ten, labeled, glass scintillation vials. Add 1.0mL of concentrated HCl to each vial and heat the sources to dryness on a hotplate.

11.29 Add 2.0mL of 0.1N HCl to each vial to dissolve the residue. Then add 15mL of Ultima Gold AB liquid scintillation cocktail to each vial. Cap each vial and shake it vigorously to mix the contents.

11.30 Establish variations in quench by carefully opening the vials and adding nitro methane to each vial in increasing increments. Nitro methane is not added to one of the vials and not to the background vial. For example, add 10 µL to one vial, 20 µL to another, 40 µL, 70 µL, 100 µL and so on, documenting the amount of nitro methane added to each vial for future reference.

11.31 Count calibration sources according to the Pace SOP, PGH-R-022, “Liquid Scintillation Counter Operations”. Count the samples long enough to obtain 10,000 net counts in the Pu-241 window. Count times will vary from source to source based on the quench factor. Adjust the amount of nitro

methane in each vial and recount the set as necessary to achieve the most representative range of values for the quench curve.

11.32 Calculate the Pu-241 efficiency based on the quench values using Microsoft Excel or equivalent curve plotting software.

11.33 Calibrations must be verified initially prior to counting samples and annually thereafter by counting a separate source in the detector and processing the source as a sample. The resulting activity of the source must be within 10% of the known target activity of the source for the calibration to be deemed acceptable. Additionally, all criteria with regards to FWHM and spectral resolution must be satisfied. Verifications which do not meet these criteria require a new calibration to be performed. In some instances this may involve replacing the actual detector prior to attempting a re-calibration. A new calibration is required whenever a detector is replaced or when calibration verification does not meet the defined acceptance criteria. Calibrations related to the analysis of Drinking Waters must be verified initially using a source that is un-related to the initial calibration source. Subsequent annual calibration verifications may be performed using the initial calibration source or a source that is un-related to the initial calibration source.

12. Procedure

Unless specified otherwise, the documented analysis process must be followed, as written, including the order of analytical process and the addition of chemicals.

12.1 Waters and Liquids, Including Drinking Water

12.1.1 Shake each sample and weigh 300g of aqueous sample into an appropriately sized beaker. Record the observed measured mass of sample to the lowest decimal on the balance. Do not remove sample from the beaker once it has been added. The actual quantity of sample used may be less than 300g if matrix interferences are expected or there is limited sample quantity available to the laboratory. If less than 300g of sample is used, dilute the sample with ASTM Type II DI water to the 300mL mark on the beaker. Fortify the pH of diluted samples by adding 2mL of HNO_3.

12.1.2 Prepare appropriate batch Quality Control samples including a Method Blank, Laboratory Control Sample (LCS), and LCS Duplicate (LCSD) by weighing an appropriate quantity of ASTM type II DI water to an appropriate size beaker. To each QC sample add 5mL of concentrated 16N HNO_3.

12.1.3 Add the applicable spikes to the appropriate QC samples in the amounts specified in Section 14 of this SOP. Add a pre-selected amount of the applicable working tracers to each of the samples and QC samples.

12.1.4 Add 1mL of iron carrier to each sample.

12.1.5 If there is a significant amount of residue present in the sample and total analysis is requested,

12.1.5.1 Filter the sample through a 0.45µm Metricel™ filter.

12.1.5.1.1 Transfer filtered sample to a labeled glass beaker

12.1.5.1.2 Transfer the Metricel™ filter and the residue to a labeled PTFE beaker.

12.1.5.2 To digest the filter and solids, add 10mL conc. 16N HNO_3, 10mL conc. 12N HCl, and 10mL conc. HF to the PTFE beaker. Cover the PTFE beaker with a cover and reflux on a hotplate for one hour.

12.1.5.3 Remove the cover and continue to heat the sample to dryness.

12.1.5.3.1 Repeat Step 12.1.5.2 as necessary based on the amount of residue remaining.

12.1.5.4 Dissolve the residue in 10mL conc. 12N HCl and 1mL saturated boric acid. Place the sample back on the hot plate and heat it to dryness again.

12.1.5.5 Dissolve the digested filter in 10mL of conc. 16N HNO_3. Place the sample back on the hotplate and heat to dryness.

12.1.5.6 Dissolve the digested filter in 10mL conc. 16N HNO_3. Use 8N HNO_3 to transfer the digested filter into the corresponding glass beaker (12.1.5.1.1) that contains the filtered aqueous sample fraction.

12.1.6 Place a watch glass on the glass beaker to cover the sample and heat it to boiling on a hotplate for a minimum of one hour.

12.1.7 Carefully add several mLs of conc. 15N NH_4OH to the sample while stirring until the pH of the sample is adjusted to 10 and a visible precipitate forms within the sample.

12.1.8 Heat the sample to boiling for another thirty minutes or longer, until an iron hydroxide precipitate breaks up into small particles.

12.1.9 Remove the sample from the hot plate and allow it to cool and the precipitate to settle.

12.1.10 Decant the excess supernate from the sample and discard it in the appropriate waste stream. Transfer the precipitate to a labeled disposable centrifuge tube with pH 10 ASTM Type II DI water.

12.1.11 Centrifuge the sample, and discard the supernate in the appropriate waste stream.

12.1.12 Rinse the iron precipitate with at least double its volume in pH 10 ASTM Type II DI water.

12.1.12.1 Vortex and centrifuge the sample. Discard the rinse in the appropriate waste stream. This rinse will eliminate excess ammonium hydroxide.

12.1.13 Proceed to the appropriate separation method beginning in Section 12.5. For Uranium in drinking water analysis, perform the uranium-specific method detailed in section 12.5.

12.2 Filter Samples

12.2.1 If the filter is cellulose:

12.2.1.1 Place a representative portion depending on the client requirements or radioactivity levels of the sample (usually one half or one quarter) of the filter into a clean ceramic crucible and pipette a selected amount of the working tracers onto it.

12.2.1.2 Cover the sample with aluminum foil and heat sample in a muffle furnace using the RAMP feature to ash the filter.

12.2.1.2.1 Use the temperature ramping guidelines in the current revision of the sample preparation SOP.

12.2.1.2.2 NOTE: Mark the PACE sample ID on each crucible with a high temperature wax pencil.

12.2.2 If the filter is glass fiber or otherwise non-organic:

12.2.2.1 Place a suitable portion of the filter into a PTFE beaker and pipette a selected amount of the working tracers onto the sample.

12.2.3 Proceed to step 12.3.3 for the sample digestion.

12.3 Soil and Vegetation Samples

12.3.1 Weigh a suitable amount of the dried sample into a ceramic crucible and pipette a selected amount of the working tracers onto the sample. Prepare a crucible designated as the LCS and LCSD if applicable and spike with the appropriate spike solutions. Prepare a crucible designated as the MB, and add 0.5mL iron carrier to the samples and all QC samples.

12.3.2 Cover the crucibles with a ceramic lid and place the crucible into a cold muffle furnace. Set the final temperature at 550ºC. Once this temperature has been reached, maintain for at least 4 hours or longer depending on the amount of material in the crucible. (For samples which are obviously highly organic, use the muffle oven's RAMP feature to prevent flashover as the sample organic components burn off.)

12.3.2.1 At the end of 4 hours, turn off the oven, allow the samples to cool to room temperature, and remove the samples from the furnace.

12.3.3 Remove the crucibles from the oven and re-label the crucibles. Add 10mL of conc. HNO_3 and 10mL of conc. HCl to the sample and heat it to dislodge the solids.

12.3.4 If the soil/solid sample does not require uranium or thorium then proceed to 12.4. Otherwise transfer the sample to a clean, labeled PTFE beaker.

12.3.4.1 Use a PTFE scraper to remove any residue that is stuck to the bottom of the crucible and rinse it into the PTFE beaker with 9N HCl.

12.3.5 Add 10mL of conc. HF to each sample in the PTFE beaker and then cover with a PTFE watch glass.

12.3.6 Reflux on an electric griddle or hotplate set at 250ºC for a minimum of thirty minutes. After 30 minutes, remove the watch glass cover and allow the sample to evaporate to dryness.

12.3.7 Add 10mLconc. HCl, 10mL conc. HNO_3, and 10mL conc. HF to the sample. Return the sample to the griddle, and allow it to evaporate dryness. This process may be repeated as often as necessary to ensure complete dissolution of the sample. In some matrices, high temperature oxides have been formed which severely limit analyte recovery. These highly insoluble oxides must be treated with repeated and prolonged contact with mineral acid. Acids must cover the samples for multiple days at low heat in order to properly dissolve the sample.

12.3.8 Add 10mL conc. HNO_3 and 10mL conc. HCl to the sample. Return the sample to the griddle and allow it to evaporate to dryness.

12.3.9 Add 10mL 12N HCl or 16N HNO_3 depending on the acid used for the load solution in order to minimize complexants. (For example, if the load solution for the column work will be 15mL 8N HNO_3, add 10mL 16N HNO_3 to the sample for the final cookdown to remove as much chloride as possible.) Add 1mL saturated boric acid to the sample and return it to the griddle and allow it to evaporate to dryness.

12.3.10 Dissolve the sample in the appropriate load solution depending on the column method to be utilized, for example 8N HNO_3 for uranium and thorium isolation using nitric conditioned anion columns, or 9N HCl/H_2O_2 for americium and plutonium isolation using chloride conditioned anion columns.

12.3.11 Heat to aid in dissolution of the sample solids. Scrape the bottom of the beaker to dislodge any solids. Centrifuge the sample as necessary prior to loading the sample onto columns.

12.3.12 Proceed to the appropriate separation method beginning in section 12.5.

12.4 Soil leach for samples not requiring Uranium or Thorium

12.4.1 Transfer the muffled and traced sample from the crucible to a labeled 100mL glass beaker. A larger beaker may be used if necessary.

12.4.2 Add 15mL HNO_3 and 5mL of HCl to each sample and cover with a watch glass. More acid may be necessary for larger aliquots but the ration of 3:1 should remain the same.

12.4.3 Reflux on a hotplate or griddle for a minimum of thirty minutes at medium to low heat. Samples must be kept from overheating, boiling or bumping.

12.4.4 Remove the samples from the hotplate and allow them to cool. Slurry the samples in the beaker and transfer the leachate and solids to a clean, labeled centrifuge tube with the aid of ASTM Type II DI water.

12.4.5 Centrifuge the samples for 15 minutes at maximum speed.

12.4.6 Decant the leachate to a 250mL labeled glass beaker. Place this beaker on a hotplate at medium and evaporate to dryness.

12.4.7 To the solids in the centrifuge tube repeat the steps from 12.4.2 to 12.4.6 using the same labeled glass beaker until the solids appear light gray or tan.

12.4.8 After evaporating to dryness, solids must be converted to the appropriate acid type by treatment with a quantity of the appropriate concentrated acid. If the chemistry load solution is to be a nitrate column, add 10mL of concentrated nitric acid and evaporate to dryness. Likewise, if the chemical load solution is chloride-based, add 10mL of concentrated HCl and evaporate to dryness. This step is not necessary for the isotopic plutonium/neptunium analysis.

12.4.9 Proceed to the appropriate separation method beginning in Section 12.5.

12.5 Method 1: Separation of Actinides on Anion Resin Using Hydrochloric Acid Solution

12.5.1 This method uses an anion exchange resin and the parameters dissolved in a hydrochloric acid solution to separate and purify any of the following groups of actinides.

- Americium, Plutonium (Neptunium), and Uranium
- Thorium, Plutonium (Neptunium), and Uranium
- Plutonium, Neptunium, and Uranium
- Uranium in drinking water samples.

12.5.1.1 This is the default method for waters and soils (less than 2 grams) where little or no interferences are expected to be present.

12.5.1.2 It is not easy to separate americium/curium from thorium using this method, so if sequential analysis requires both analyses be performed on a single aliquot, start with Method 2 to isolate the thorium fraction first from all of the other nuclides.

12.5.2 Dissolve the sample precipitate in 15mL of 9N HCl / 0.10% H_2O_2. The hydrogen peroxide need only be used if plutonium will be isolated.

12.5.3 Prepare a 2mL resin column using Bio-Rad AG 1x8.

12.5.3.1 Slurry the resin with ASTM Type II DI water to give a resin bed of about 3cm. The final resin volume must be consistent for all columns.

12.5.3.2 Once the ASTM Type II DI water has drained, condition the column with 10mL of 9N HCl.

12.5.4 If heated, allow the sample to cool, then transfer it to the prepared resin column.

12.5.5 Collect the effluent from the column in a clean labeled centrifuge tube if either americium/curium or thorium analysis is desired.

12.5.5.1 Rinse the sample beaker or centrifuge tube with an additional 10mL 9N HCl / 0.10% H2O2 and collect.

12.5.5.2 Rinse the column with 20mL of 9N HCl and collect.

12.5.5.3 Transfer the contents of the centrifuge tube to a clean labeled glass beaker.

12.5.5.4 If the contents are to be analyzed for americium/curium, proceed to Method 3: Americium/Curium Purification on TRU-Resin™ for americium/curium analysis.

12.5.6 If it is desirable or necessary to isolate isotopic Plutonium from Neptunium, elute the plutonium from the column into a clean, labeled glass beaker with 20mL 9N HCl / 0.05N NH_4I.

12.5.6.1 If analysis for plutonium is not required, do not perform this step and proceed to step 12.5.7.

12.5.6.2 Add 10mL conc. HNO3 and 3-4 drops of the iron carrier to the plutonium fraction.

12.5.6.3 Allow enough time for the nitric acid and the hydrochloric acid to react.

12.5.6.4 With a disposable pipette, and in a drop wise fashion, slowly add approximately 1mL of 30% H_2O_2 to the solution.

12.5.6.5 Place the beaker on a hotplate and allow the plutonium fraction to evaporate to dryness.

12.5.6.6 Add 5mL concentrated HCl to each beaker and evaporate the contents to dryness on a hotplate.

12.5.6.7 Dissolve the residue in the beaker with 10mL 9N HCl.

12.5.6.8 Transfer the plutonium fraction to a labeled centrifuge tube with ASTM Type II DI water and dilute to 20mL with ASTM Type II DI water.

12.5.6.9 Proceed to Step 12.9.2, Micro-Precipitation of Plutonium.

12.5.7 Rinse the column with 15mL of 6N HCl / 0.52N HF, which will elute any neptunium from the sample (Use 20 mL if step 12.5.6 was not utilized and Plutonium is being eluted with Neptunium.

12.5.7.1 Proceed to Step 12.9.5 for the Micro-Precipitation of Neptunium. (Step 12.9.2 if analyzing for Plutonium and Neptunium simultaneously.)

12.5.7.2 If analysis for neptunium is not required, discard the effluent.

12.5.8 Rinse the column with 10mL of 9N HCl and discard the effluent.

12.5.9 Elute the uranium fraction from the column into a clean, labeled centrifuge tube with 20mL of 0.1N HCl.

12.5.9.1 Proceed to Step 12.9.3 for the Micro-Precipitation of Uranium.

12.6 **Method 2: Separation of Actinides on Anion Resin using Nitric Acid Solution**

12.6.1 This method uses an anion exchange resin and the parameters dissolved in a nitric acid solution to separate and purify the following group of actinides:

- Americium, Plutonium and Uranium from Thorium

12.6.1.1 This method is best used for solids (any amount), waters, or filters (organic based, cellulose), and when it is desirable to reuse the resin column after isolating Thorium.

12.6.2 Dissolve the sample into 15mL of 8N HNO_3

12.6.2.1 Heat the solution if necessary to aid in the dissolution of the precipitate.

12.6.3 Prepare a 2mL resin column with Bio-Rad AG 1x8.

12.6.3.1 Slurry the resin with ASTM Type II DI water to give a resin bed of about 3cm.

12.6.3.2 When the ASTM Type II DI water has drained, condition the column with 20mL 8N HNO_3.

12.6.4 Cool the sample and transfer it to the prepared resin column.

12.6.4.1 Collect the effluent from the column in a clean glass beaker if uranium/plutonium analysis is required. This fraction will also contain any Americium/Curium or Neptunium if present in the sample.

12.6.4.2 Rinse the column with an additional 15mL 8N HNO_3 and collect. The rinse will contain any residual americium, plutonium, and uranium from the sample.

12.6.4.3 Repeat step 12.6.4.2. Collect this rinse.

12.6.4.4 Place the americium, plutonium, uranium fraction on a hotplate and allow it to evaporate to dryness.

12.6.4.5 Continue with the separation of analytes in the solution in step 12.6.4.4 by proceeding to Step 12.5.2 after adding 15mL conc. HCl, and evaporating the samples to dryness.

12.6.5 Elute the thorium from the column with 25mL of 9N HCl into a labeled centrifuge tube.

12.6.5.1 Transfer the solution containing the thorium from the centrifuge tube to a clean, labeled plastic cup and add 0.8mL of 10mg/mL iron carrier.

12.6.5.2 Dilute the samples to approximately 220mL with ASTM Type II DI water.

12.6.5.3 Precipitate thorium with iron as a hydroxide by adding 22-25mL of concentrated ammonium hydroxide to each sample dilution. Stir the solution vigorously with a stir rod for several seconds until a distinct iron hydroxide precipitate forms.

12.6.5.4 Allow the samples to settle for about 15 minutes, and stir vigorously once more to further break up the iron hydroxide precipitate.

12.6.5.5 Remove the stir rod and allow the samples to completely settle for a minimum of one hour.

12.6.5.6 Decant the excess supernate and transfer the iron hydroxide precipitate to the original labeled centrifuge tubes from Step 12.6.5.1.

12.6.5.7 Centrifuge the samples and discard the supernate. Dissolve the precipitate in 3mL 9N HCl. Dilute the samples to 25mL with ASTM Type II DI water.

12.6.5.8 Proceed to Step 12.9.4 for the Micro-Precipitation of Thorium.

12.6.5.9 If the column is to be reused for the uranium purification, rinse the column with 25mL of ASTM Type II DI water.

12.6.6 Perform all of steps listed in Method 1 to purify the americium, plutonium, and uranium as required.

12.7 **Method 3: Purification of Americium/Curium Using Tru-Resin™**

12.7.1 This method may be used as the initial separation step for liquids or waters only if americium/curium is requested. It must be used subsequent to Method 1 in all other instances to purify americium/curium and separate them from thorium.

12.7.2 Add 3-4 drops of the iron carrier to the americium fraction collected in Step 12.5.5.4. Place the sample on a hotplate and evaporate the sample to dryness.

12.7.3 Add 5mL of concentrated nitric acid to each sample and evaporate to dryness to remove residual chlorides.

12.7.4 Dissolve the sample residue into 10mL of 2N HNO_3 /0.5M $Al(NO_3)_3$. Additional 2N HNO_3/0.5M $Al(NO_3)_3$ may be used to dissolve the sample fraction, but the total load volume must not exceed 15mL.

12.7.4.1 Heat the sample gently as necessary to aid in the dissolution.

12.7.5 Add one drop of 0.5M potassium thiocyanate to the sample and swirl it, then add 6-8 drops of 1.0M ascorbic acid to the sample and swirl it.

12.7.5.1 The color of the sample should go from clear to red and then back to clear again, unless there is no significant iron present in the sample.

12.7.6 The sample should be centrifuged prior to loading it on the column if there are any solids present.

12.7.7 Prepare a resin column with 5 drops of pre-resin filter followed by 3.5mL (2 grams) of Tru-Resin™.

12.7.7.1 Allow any excess water to drain.

12.7.7.2 Condition the columns by passing 5mL of 2N HNO_3 through them and discarding the effluent.

12.7.8 Load the sample onto the column.

12.7.8.1 A change in resin color should be observed, usually pink, but it may be multi-colored.

12.7.8.2 Discard the effluent.

12.7.9 Rinse the column with three 5mL aliquots of 2N HNO_3 allowing each to pass through the column. Discard the effluents.

12.7.10 Place a clean, labeled centrifuge tube under the columns and elute the americium/curium from the resin with 2mL of 9N HCl.

12.7.11 Complete the elution of the americium/curium from the column by adding 10mL 4N HCl. Combine the effluent from the column

in the centrifuge tube. If interferences are expected to cause spectral resolution issues, proceed directly to step 12.8.3.

12.7.12 Proceed to Step 12.9.1 for the Micro-Precipitation of Americium.

12.8 **Method 4: Purification of Americium/Curium Using TEVA-Resin**

12.8.1 After Method 3 has been preformed, the following steps are available to further purify the americium/curium.

12.8.2 Transfer the americium/curium eluate from step 12.7.11 to a small glass beaker. Place the sample on a hotplate and allow it to evaporate to dryness

12.8.2.1 Do not allow the samples to bake.

12.8.3 Dissolve the residue in the beaker in 10mL conc. HCl and place it back on the hotplate allowing it to evaporate to dryness.

12.8.3.1 Do not allow the sample residue to bake.

12.8.4 Add 1mL conc. formic acid to each sample and allow them to evaporate to dryness.

12.8.5 Dissolve the sample in 15mL 3M ammonium thiocyanate/ 0.1N formic acid.

12.8.5.1 Heat the sample as necessary to aid in the dissolution.

12.8.5.2 The sample should take on a light pink color.

12.8.6 Prepare a 2cm column with 3.5mL prepared (2 grams) TEVA resin™.

12.8.6.1 Allow any excess water to drain through.

12.8.6.2 Condition the column with 5mL of the 3M ammonium thiocyanate/ 0.1N formic acid.

12.8.7 Ensure that the sample has cooled and load it onto the conditioned column.

12.8.7.1 Rinse the sample beaker with 2mL 1M ammonium thiocyanate/ 0.1N formic acid.

12.8.7.2 Pour the rinse onto the column when the sample has completely passed through.

12.8.8 Rinse the column first with 3mL and then with 5mL of the 1M ammonium thiocyanate/ 0.1N formic acid.

12.8.8.1 Discard all rinses to waste. (Note: Ammonium thiocyanate should be neutralized with dilute nitric acid in an open container prior to disposal to avoid violent reactions.)

12.8.9 Elute the americium/curium from the column with 15mL of 2N HCl into a clean, labeled centrifuge tube.

12.8.10 Proceed with Step 12.9.1 for Micro-Precipitation of Americium.

12.9 Micro-precipitation of the Actinides

12.9.1 Americium/Curium

12.9.1.1 Add 0.1mL of the 0.5 mg/mL neodymnium carrier to the sample. Swirl the sample to mix the contents.

12.9.1.2 Add 3mL conc. HF to the sample. Cap the centrifuge tube and shake the sample vigorously to mix the contents.

12.9.1.3 Allow the sample to sit for thirty minutes, then proceed with Step 12.9.6 for filtration.

12.9.2 Plutonium

12.9.2.1 Add 0.1mL 0.5mg/mL neodymnium carrier to the sample and swirl to mix.

12.9.2.2 Add 12 drops of 25% dihydrazine dihydrachloride solution to each sample.

12.9.2.3 Swirl the solution to mix the contents, then allow the sample to sit for 5 minutes.

12.9.2.4 Add 3mL of conc. HF to the sample. Cap the sample and shake it vigorously to mix the contents.

12.9.2.5 Allow the sample to sit for thirty minutes.

12.9.2.6 Proceed to Step 12.9.6 for filtration.

12.9.3 Uranium

12.9.3.1 Add 0.1mL 0.5mg/mL neodymnium carrier to the sample and swirl to mix.

12.9.3.2 Add enough of the titanium chloride solution to the sample to maintain a light purple color when swirled (approximately 1-2mL).

12.9.3.3 Swirl the solution to mix the contents, then allow the sample to sit for a few minutes.

12.9.3.4 Add 3.0mL conc. HF to the sample. Cap the sample and shake it vigorously to mix the contents.

12.9.3.5 Allow the sample to sit for thirty minutes.

12.9.3.6 Proceed with Step 12.9.6 for filtration.

12.9.4 Thorium

12.9.4.1 Add 0.1mL 0.5mg/mL neodymnium carrier to each sample. Swirl the contents to mix.

12.9.4.2 Add 3.0mL conc. HF to the sample. Cap the sample and shake it vigorously to mix the contents.

12.9.4.3 Allow the sample to sit for thirty minutes.

12.9.4.4 Proceed with Step 12.9.6 for filtration.

12.9.5 Neptunium

12.9.5.1 Add 0.1mL 0.5 mg/mL neodymnium carrier to each sample. Swirl the contents to mix.

12.9.5.2 Add 3.0mL conc. HF to the sample. Cap the sample and shake it vigorously to mix the contents.

12.9.5.3 Allow the sample to sit for thirty minutes.

12.9.5.4 Proceed with Step 12.9.6 for filtration.

12.9.6 Filtration

12.9.6.1 Prepare the filtration unit by turning on the vacuum and rinsing each apparatus with severalmLs of ASTM Type II DI water.

12.9.6.2 Carefully wash and rinse each of the filter funnels and set them aside.

12.9.6.3 Place a polypropylene filter onto each unit and ensure that it is centered.

12.9.6.4 (Carefully) Place the funnels on the filtration unit.

12.9.6.4.1 Do not rip the filters.

12.9.6.4.2 Rinse the sides of the funnels with 80% reagent grade alcohol.

12.9.6.5 When the sample has completely passed through the filter, rinse the sides of the funnel with a few mLs of the 0.1% HF solution.

12.9.6.6 Rinse the funnel and filter with a few mLs of 80% reagent grade alcohol.

12.9.6.7 When the rinses have completely passed through the filter, remove the filter funnels, turn off vacuum, and carefully remove the filter paper with tweezers.

12.9.6.8 Place the filter paper onto a labeled pre-taped metal disc for counting.

12.9.6.8.1 Allow the filter to completely dry prior to counting by placing it in a labeled petri dish, and storing it in the count room until ready to count.

12.9.6.9 Count the samples in an alpha spectrometry detector as instructed in the current revision of the instrument SOP, PGH-R-020.

12.9.6.10 Obtain instrument printouts and perform calculations as detailed in Attachment 1 of this SOP.

12.9.6.11 If plutonium-241 analysis is desired, the Pu counting source must be removed from the taped disk after alpha spectroscopy counting using a minimal amount of acetone.

12.9.6.12 Place the filter in a glass liquid scintillation vial. Cover the vial with aluminum foil and muffle it in an oven at 550°C for a minimum of 2 hours or until the filter has completely ashed away and there is no black residue left in the vial.

12.9.6.13 Remove the vial from the oven and discard the foil. Add 6 drops of saturated boric acid and 1mL of concentrated HCl to each sample and heat to dryness.

12.9.6.14 Add 2.0mL 0.1N HCl to each sample to dissolve the residue and 15mL of Ultima Gold AB liquid scintillation cocktail. Cap the vial and shake it vigorously.

12.9.6.14.1 The samples should be free of any color.

12.9.6.15 Clean the outside of each vial with acetone followed by ASTM Type II DI water to remove any finger prints or residue.

12.9.6.16 Dark-adapt the samples for one hour prior to counting in a calibrated, liquid scintillation counter in accordance with the liquid scintillation instrument operating SOP, PGH-R-022, current revision.

13. Calculations

13.1 Refer to Attachment I of this SOP for all actinide analysis associated calculations.

13.2 Any verified result for drinking water that exceeds the maximum contaminant level (MCL) established for Uranium must be reported to the appropriate personnel and agencies according to the specific requirements of the state where the water was sampled. The directions for reporting and results that exceed the MCL limits are documented in the State Drinking Water Emergency Reporting Requirements Binder and Pace SOP PGH-C-025, current revision.

13.2.1 Uranium MCL >= 20 pCi/L total uranium (U-238 + U-235 + U-234)

14. Quality Control

14.1 General guidelines for drinking water samples with results that exceed the Maximum Contaminant Level include the following: (All steps are to be conducted as soon as the exceedence has been identified.)

14.1.1 Verify the result(s) to ensure that there were no transcription or calculation errors and that all QC results are within the acceptable limits. Correct any problems and determine the new result. If there were no errors or the result still exceeds the MCL, continue with the reporting process.

14.1.2 Immediately notify the Department Manager/Supervisor, and QA Department that a reportable result has been identified. Use telephone notifications to inform the contact people if the variance is identified after hours along with an e-mail follow up to document the event.

14.1.3 Refer to the State Drinking Water Emergency Reporting Requirements Binder for the state specific information regarding the proper course of action to take. Time is of the essence during this process with some of the state reporting requirements as short as 1 hour from the verification of an exceedence

14.2 Each analyst who performs this test must satisfactorily complete a Demonstration of Capability Study as documented in Section 3.4 of the most recent revision of the Quality Assurance Manual.

14.2.1 The DOC study results are evaluated against the LCS acceptance limits.

14.3 Daily instrument Quality Control checks for the alpha spectrometer must be completed following the instructions detailed in the current revision of Pace SOP PGH-R-020, “Alpha Spectroscopy Instrument Operations.”

14.4 Daily instrument Quality Control checks for the liquid scintillation counter must be completed following the instructions detailed in the current revision of Pace SOP PGH-R-022, “Liquid Scintillation Instrument Operations.”

14.5 Daily instrument Quality Control checks for the gas flow proportional counters must be completed following the instructions detailed in the current revision of Pace SOP PGH-R-002, “Gas Flow Proportional Instrument Operations.”

14.6 See Appendix II for performance indicator evaluation calculations and criteria. Numerical performance indicators may be used to assess QC for non-drinking water samples when the default assessment indicates a QC failure. The numerical performance indicator must be within +/- 3 for all other matrices. The z-score for precision assessment may be used for drinking waters with the approval of the Department Manager/Supervisor using the +/- 2 specification.

14.7 Sample Tracer Recovery/Tracer Peak Energy

14.7.1 Sample tracer is added to each sample and used to calculate sample recovery. Tracer recovery is required to be within 30%-110% for samples analyzed under jurisdiction of the DOD QSM. Pace’s default acceptance criteria for tracer recovery requires recovery between 30%-110%; however, recoveries may be acceptable between 10% and as high as 130% with the documented permission of the Department Manager/Supervisor or specified designee.

14.7.2 Samples with tracer recoveries outside of this range should be re-prepped with direction from the Department Manager or Manager-specified designee after determining possible causes.

14.7.3 Sample tracer counts should be sufficient to minimize the expanded uncertainty of the count and any potential for tailing of the tracer counts into other regions of interest. A minimum of 400 tracer counts is recommended, however, in some instances, the sample matrices may prohibit achieving a minimum 400 tracer counts, and the sample data must be evaluated prior to extending or recounting samples solely to achieve a minimum 400 tracer counts.

14.7.4 Excluding samples analyzed for isotopic thorium content using Th-229 as a tracer, samples analyzed under jurisdiction of the DOD QSM, the tracer energy must be within 40 keV of the known tracer isotope peak energy.

14.7.5 For samples associated with the DOD QSM, if the tracer energy is not within 40 keV of the known peak energy, this may indicate an issue with the detector used for analysis. The sample may be reanalyzed using an alternate detector. If re-analysis results indicate an acceptable tracer energy peak location, results may be reported. If, following re-preparation and re-analysis, the tracer peak is not within 40 keV of the known peak energy, results may be reported with appropriate “J” flagging.

14.7.6 If the tracer is a non-Th-229 alpha emitter, its peak full width half maximum (FWHM) value must be evaluated and generally should be less than 100 keV. When the FWHM is greater than 100 keV the sample data must be inspected using professional judgment to determine if the detector was functioning properly and if the procedure was adequate. Data that has FWHM up to 125 keV may be reported with qualification, except for samples analyzed under the requirements of the DOD QSM. For DOD-associated samples, if the tracer FWHM exceeds 100 keV, the affected sample must either be re-prepared and re-analyzed or the alpha count source may be “re-purified” in accordance with the process outlined in this SOP. If count source re-purification is attempted but fails the FWHM criteria, the sample must be re-prepared and re-analyzed. If upon re-preparation and re-analysis the sample exhibits a tracer peak energy outside of established control criteria, analysis results may be reported with the appropriate “J” flag.

14.7.7 Unlike other radioactive tracers used for yield monitoring, the alpha peak energies associated with Th-229 are of lower abundance and greater energy distribution over the region of interest associated with Th-229. For this reason, there is a greater uncertainty in calculating peak centroid energies and FWHM values when using spectroscopy systems that perform alpha spectral analysis of. When it is observed that the Th-229 centroid energy is calculated to be greater than 40 keV from the average Th-229 peak energy or when the Th-229 peak resolution is determined to be greater than 100 keV, analytical spectra must be reviewed and approved by the Department Manager or a

Department Manager specified designee. For approval, analytical spectra must document clear de-markation between the Th-229 tracer and adjoining analyte regions of interest.

14.8 Method Blank (MB)

14.8.1 One MB must be prepared for each analytical batch. The purpose of the MB is to monitor for cross contamination during the analytical process. When available, the MB should be prepared from a similar matrix as samples contained in the analytical batch. If appropriate blank matrix material is not available, ASTM Type II DI water (Reagent Blank) must be carried through the procedure. A reagent blank may be used for sample correction purposes following approval of affected clients.

14.8.2 The MB result must be less than the MDC. If the method blank result is greater than MDC, individual sample results may still be reportable.

14.8.2.1 PASI's default criteria allows reporting of sample results less than the CRDL (contract required detection limit) or greater than 10 times the blank result. Relative sizes of the sample and blank aliquots must be factored when making this determination (raw counts).

14.8.2.2 For samples analyzed under the DoD QSM, the Method Blank result must be less than ½ the detection limit. Corrective action is necessary for any MB result greater than ½ the detection limit.

14.9 Sample Duplicate (DUP)

14.9.1 One Duplicate Sample (DUP) must be randomly assigned within each batch. The purpose of the sample DUP is to measure precision of the analytical process. Laboratory duplicates are not intended to assess precision related to the sample collection process. Sample collection precision can only be assessed through collection of duplicate samples at the time of sample collection. The sample DUP is a duplicate volume of sample processed identically as other samples in the analytical batch.

14.9.2 For batches with drinking water samples originating from the state of Arizona, Duplicate Samples (DUP) must be randomly assigned within each batch at a frequency of no less than 10%. A batch of ten samples or fewer must contain at least one duplicate sample. A batch of greater than 10 samples up to 20 samples must contain a minimum of two duplicate samples if the batch contains samples originating from Arizona.

14.9.3 Calculation

$$\%RPD = \frac{\left|\left(R_S - R_D\right)\right|}{\left(\left(R_S + R_D\right)/2\right)}$$ Where:

R_S = sample activity concentration
R_D = duplicate activity concentration

14.9.4 Duplicate sample performance is acceptable when the %RPD is <25%.

14.10 Sample Matrix Spikes (MS)

14.10.1 Because this analytical method requires the use of radiotracers for yield determination, PASI's default QC policy is that a sample matrix spike (MS) is not required for alpha spectrometry analyses except for drinking water analysis.

14.10.2 A matrix spike is prepared by spiking a known amount of spike solution (Am-241, Cm-244, Pu-239, Th-232, U-238, Np-237, etc.) into a portion of one sample in the batch, and it must be processed identically as for other samples. The purpose of the MS is to assess the effect of sample components on the analytical process. The volume of sample used for the MS must be equivalent to the volume used for sample analysis. The spike amount should be approximately 10 times the detection limit and not less than 20% of the anticipated sample concentration.

14.10.3 Matrix Spike Recovery Calculation

$$\%REC = \frac{(x - x_0)}{c} x100$$ Where:

x = measured concentration of the spiked sample
x_0 = measured concentration of the unspiked sample
c = spike concentration added

14.10.4 MS performance is acceptable when agreement of the measured value and the expected value is within ±25% of the true value.

14.11 Sample Matrix Spike Duplicates (MSD)

14.11.1 A sample Matrix Spike Duplicate (MSD) is not required for this analysis. When required by the customer/contract, a MSD must be prepared for each analytical batch. The MSD must be prepared as a duplicate of the MS.

14.11.2 For all matrices the MSD must pass the criteria established for the MS. Additionally, the MS and MSD must pass the criteria established for duplicate precision.

14.12 Laboratory Control Sample (LCS)

14.12.1 One LCS must be prepared for each analytical batch and is a reference material that contains a known concentration of spike (Am-241, Cm-244, Pu-239, Th-232, U-238, Np-237, etc.) in a matrix that is similar to the samples within the batch. If this material is not available, a well-characterized material (WCM) may be used. If neither of these is available, DI may be spiked with a spike solution greater than 2 times the detection limit.

14.12.2 LCS Recovery calculations

$$\%REC = \frac{x}{c} x100$$ Where:

x = Analytical result of the LCS
c = Known concentration of the LCS

14.12.3 LCS performance is acceptable when the %REC is within ±25% of the known value.

14.13 Laboratory Control Sample Duplicate (LCSD)

14.13.1 A LCSD must be analyzed to measure batch precision whenever adequate sample volume is not available for sample DUP analysis. The LCSD must be prepared in an identical fashion as the LCS and processed identically as for other samples.

14.13.2 The LCSD must pass the criteria established for the LCS.

14.13.3 Additionally, the LCS and LCSD must pass the criteria established for duplicate precision.

14.14 Summary of QC related Activities:

Method Blank	One per Batch
Reagent Blank	One per Batch (as required by client)
Duplicate Sample	One per Batch (frequency of 1 per 10 samples for batches containing DW originating from AZ)
Matrix Spike	One per Batch (for drinking water analysis or as required by client)
Matrix Spike Duplicate	One per Batch (frequency of 1 per 10 samples for batches containing DW originating from AZ or as required by client)
Laboratory Control Sample	One per Batch
Laboratory Control Sample Dup	One per Batch for samples in the absence of Duplicate sample.

14.15 Corrective Actions for Out-Of-Control Data

14.15.1 Method Blank (Reagent Blank) (MB/RB) – Individual samples that do not meet the acceptance criteria must be reanalyzed. If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

14.15.2 Duplicate (DUP) – DUP analysis that fails the replicate test must be reanalyzed to determine if analytical failure or sample heterogeneity was the cause of the problem.

14.15.3 Matrix Spike Recovery (MS) – MS recoveries that fail high and outside of control criteria with a sample result that is less than the reporting limit may be reported with narration. Additionally, MS recoveries that fail low and outside of control criteria for

Drinking Water samples with a sample result that is greater than the MCL must be reported with comment as potentially biased low due to matrix interference. Otherwise, MS recoveries that do not meet the acceptance criteria must have that sample reanalyzed. If a Matrix Spike Duplicate is also analyzed and the recovery is comparable to the MS, the results are reported and noted in the final report. Matrix effect must be determined by re-analysis of the MS/Sample pair or demonstration of acceptable precision between a MS/MSD pair.

14.15.4 The analyst must evaluate the MS results to attempt to determine the cause of the failure and the appropriate action to take based on that evaluation. All decisions made must be documented.

14.15.5 Matrix Spike Duplicate (MSD) - If an MSD is analyzed and the recovery is comparable to the MS, the results are reported with qualification in the final report.

14.15.6 Laboratory Control Sample (LCS) – If an LCS analysis does not meet the acceptance criteria, the entire analytical batch must be re-prepped and reanalyzed.

The results of the batch may be reported, with qualification in the final report, if the LCS recoveries are high and the sample results within the batch are less than the reporting limit.

14.15.7 Laboratory Control Sample Duplicate (LCSD) – If an LCSD does not meet the recovery acceptance criteria, the entire analytical batch must be reanalyzed.

The results of the batch may be reported, with qualification, if the LCS recoveries are high and the sample results within the batch are less than their the reporting limit, and duplicate precision meets the acceptance criteria.

14.15.8 If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

14.16 Contingencies for handling Out-of-Control or Unacceptable Data

14.16.1 Method Blank (Reagent Blank): If the sample is exhausted, evaluate the usefulness of the data in the final report.

14.16.2 Duplicates: If the sample is exhausted, evaluate the usefulness of the data in the final report.

14.16.3 Matrix Spike Recovery: If a Matrix Spike is analyzed and the spike recoveries are not comparable, and the sample is exhausted, evaluate the data usefulness in the final report.

14.16.4 Matrix Spike Duplicate: If a Matrix Spike Duplicate is analyzed and the spike recovery is not comparable to the Matrix Spike and the sample is exhausted, evaluate the data usefulness in the final report.

14.16.5 Tracer recovery: If the tracer recovery is less than 30% but greater than 10% with more than 400 tracer counts, the sample may be reported with supervisor permission. Tracer recovery above 110% but below 130% may be reported but must be narrated. Samples with tracer recovery below 10% or above 130% must be re-analyzed. If the sample is exhausted, evaluate the usefulness of the data in the final report.

15. Method Performance

15.1 Each analyst must read and understand this procedure with written documentation maintained in their training file on the Learning Management System (LMS).

15.2 An initial demonstration of capability (IDOC) study must be performed. A record of the IDOC will be maintained on file in each analysts training file in the LMS.

15.3 On an annual basis, each analyst will complete a continuing demonstration of capability (CDOC).

15.4 Laboratory Control Samples are analyzed with each batch, the results are charted to monitor control limits and trending.

16. Pollution Prevention and Waste Management

16.1 Place radioactive waste into the appropriate receptacles.

16.2 Discard acidified samples and unusable standards into the proper waste drains.

16.3 Dispose of waste materials in accordance to type: (Non-hazardous, hazardous, non-radioactive, radioactive or mixed).

17. References

17.1 Bishop, C. T., et.al. “Radiometric Method for the Determination of Uranium in Water,” EPA 600/7-79-093, EMSL-LV, April 1979.

17.2 Edwards, K. W. “Isotopic Analysis of Uranium in Natural Waters by Alpha Spectrometry,” Radiochemical Analysis of Water, Geological Survey Water – Supply Paper 1696-F, U.S. Government Printing Office, Washington, D.C., 1968.

17.3 Krieger, H. L. and Whittaker, E. L., Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, “Uranium-Radiochemical Method,” Method 908.0, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH, August, 1980.

17.4 ASTM E181-93, Standard Test Methods for Detector Calibration and Analysis of Radionuclides, ASTM Standards, Vol. 12.02.

17.5 ASTM D-3972-90, Test Method for Isotopic Uranium in Water by Radiochemistry, ASTM Standards, Vol. 12.04.

17.6 Table of Radioactive Isotopes, Brown and Firestone, Shirley editor, John Wiley & Sons, 1986.

17.7 Currie, L., Limits for Quantitative Detection and Quantitative Determination, Analytical Chemistry, Vol. 40. No. 3, Pg 586-593, 1968.

17.8 Currie, L., Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements, NUREG/CR - 4007, USNRC, 1984.

17.9 "American National Standard Calibration and Usage of Alpha/Beta Proportional Counters", ANSI N42.25-1997.

17.10 "Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)", July 2004, Final.

17.11 Eichrom Industries, Various Actinide Procedures. Darien, Illinois, 1995

17.12 EML Procedures Manual, HASL-300 28th Edition.

17.13 Pace SOP PGH-R-001, current revision (Analysis of Samples for Gross Alpha and Gross Beta content).

17.14 Pace SOP PGH-R-002, current revision (Gas Flow Proportional Counter Operation).

17.15 Pace SOP PGH-R-020, current revision (Alpha Spectroscopy Instrument Operations).

17.16 Pace SOP PGH-R-022, current revision (Liquid Scintillation Counter Operations).

17.17 Pace SOP PGH-R-024, current revision (Radiochem Sample Preparation).

17.18 Pace SOP PGH-C-027, current revision (Deionized Water Quality and Suitability).

17.19 Pace SOP PGH-C-025, current revision (Reporting SDWA MCL Violations).

17.20 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (most recently approved revision).

17.21 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.

17.22 Pace Analytical Services, LLC. - Pittsburgh Laboratory Quality Assurance Manual, current version.

17.23 Department of Defense (DOD), Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories; DOD QSM version 5.1, DOE Quality Systems for Analytical Services Version 3.1, 2017.

18. Tables, Diagrams, Flowcharts, Appendices, etc.

18.1 Attachment No 1: Concentration Calculation & Counting Uncertainty

18.2 Attachment No 2: Evaluation of QC using Numerical Indicators.

18.3 Figure No 1: Analysis Flowchart for Sequential Analysis of Am, Pu, U, and Th

18.4 Figure No 2: Analysis Flowchart for Sequential Analysis for Am, Pu, U (Regular Water or Solids)

18.5 Figure No 3: Analysis Flowchart for Sequential Analysis of Am and Pu in Solids >1 Gram

18.6 Figure No 4: Analysis Flowchart for Sequential Analysis of U and Th in solid or water

19. Method Modifications

19.1 This method for uranium in water is substantially compliant with ASTM Method D-3972-90 and HASL 300 Method U-02 for uranium with the following exceptions:

19.2 ASTM D3972-90 Modifications:

19.2.1 Ammonium iodide or HNO_3/H_2O_2 rinses have been substituted for the HI rinse to strip plutonium from the column prior to eluting uranium.

19.3 HASL U-02 Modifications:

19.3.1 U-02 foresees initial pre-concentration by simple evaporation. This can often lead to analyte loss due to poorly soluble residues. The method has been modified to employ a ferric hydroxide precipitation which both avoids formation of insoluble residues while providing additional decontamination.

19.3.2 Method HASL 300 U-02 specifies the use of AG 1X4 anion exchange resin. PASI utilizes AG 1X8 anion exchange resin due to increased loading capacity as well as enhanced selectivity in the separation of uranium from competing/interfering elements.

19.3.3 Method HASL 300 U-02 indicates the use of 7N HCL solution in the loading of uranium onto the anion exchange column with 1 N HCL as the eventual eluant for uranium. PASI utilizes a 9N HCL for loading and 0.1N HCL for elution to enhance analyte recovery.

19.3.4 Additionally, PASI utilizes intermediate stripping reagents to selectively remove plutonium (9N HCl / 0.05N NH_4I) and neptunium (6N HCl / 0.52N HF), if present in the samples.

19.3.5 Additional rinses have been added to HASL 300 U-02 to provide efficient decontamination from alpha emitting interferences.

19.3.6 Quality Control requirements have been modified to conform to PACE Analytical Services, LLC. QAP requirements and procedures.

19.4 This method for plutonium, americium and curium isotopes in all matrices and uranium in non-drinking water matrices is a PACE Analytical Services, LLC. proprietary method and is based in parts on EPA or other promulgated methods.

20. Revisions

Document No.	Reason for Change	Date
PGH-R-008-6	1. Updated cover sheet to include Periodic Review. Updated Cover Page, Headers and Footers for this revision. Added periodic review signature lines to the cover page. 2. Updated Table of Contents section to include Attachments	31Dec2012

Document No.	Reason for Change	Date
	and Flowcharts. Made TOC updateable and linked to all the primary sections. 3. Updated Flow charts for the current thorium procedure after column separation. 4. Updated flowcharts with "Count to meet MDC and obtain 400 tracer counts", removing any suggested time requirement. 5. Section 12.7.6.6 – added to include addition of HCl to match flowchart directions. 6. Corrected section 12.8.7 to direct to the correct section for Pu analysis. 7. Section 14.4.3– expanded suggested minimum tracer counts requirements. 8. Section 15 – Removed annual MDL study requirement. Not performed for radiochemical methods. 9. Section 17 – Added TNI reference 10. Removed all references allowing approval of a "senior analyst" and replaced with "approval of Department Manager or Manager-specified designee."	
PGH-R-008-7	1. Updated cover page for this revision and to update the copy right footnote. Also updated to include the Methods Referenced 2. Section 1.3 corrected location of Method Deviations section. 3. Section 6: Updated to specify not targeting weights, recording observed measurements, and not removing sample from beakers once transferred from bottle. 4. Section 10: DI reference to ASTM Type II, and SOP reference. 5. Section 12: Updated to specify spiking and tracing preceding all chemical additions beyond initial preservation. 6. Section 17: Updated to include ASTM D 3972-90 and PASI-PGH QAM. 7. Section 19: Updated deviations from HASL 300 U-02 method.	07Nov2013
PGH-R-008-8	1. Annual SOP review and update. 2. Section 2 – Added references to applicable instrument operation SOPs. Included these references where instrumentation is discussed in other areas of the document. 3. Section 8.1.1 – Included pH verification requirements and recording. 4. Section 8.1.4 – Added maximum hold time requirement. 5. Section 9 – Included references to Pace SOP for instrument operations and listed instrumentation. 6. Section 12.1 – Clarified aqueous sample analysis amounts and instructions for diluting samples. 7. Section 12.6 – Urine samples measured with graduated cylinders not weighed. 8. Section 14.5 – Changed to be consistent with other SOPs for discussion of numerical indicator application. 9. Section 14 – Included duplicate requirement for Arizona drinking water samples in applicable sections.	13Jul2014

Document No.	Reason for Change	Date
	10. Section 15 – added CDOC requirements 11. Section 17 – Added applicable instrument SOP references. 12. Reformatted document.	
PGH-R-008-9	1. Removed from section 5.1: Analysts must be trained as radiation workers and personal dosimeter worn. 2. Section 14 modified to add tracer peak quality control requirements for USDOD-related sample analysis. 3. Section 17 modified to add reference to the US DOD/DOE QSM version 5.0.	20Feb15
PGH-R-008-10	1. Annual review and update. 2. Updated section references throughout document. 3. Updated section 12.4.7 to discuss the process for treating highly oxidized solids to improve analyte recovery.	27Jul2015
PGH-R-008-11	1. Periodic review and update. 2. Section 2.3 – All analytes are decay corrected to the supplied collection date and time. 3. Section 3.2 – Removed reference to a Rapid Extraction Method, not performed. 4. Sections 12.6, 12.11, 12.12, 12.13, and 12.14 – Removed the instructions for Urine analysis, and all instructions referring to specialized methods which are not being performed. 5. Section 10 – Removed reagents associated with the removed methods/sections. 6. Section 12.10.3 and 12.10.4 removed since they are not used. 7. Methods 1 and 2 – Removed the “additional” volumes on each step, since various sized columns are not utilized.	15Mar2017
PGH-R-008-12	1. Periodic review and update. 2. Section 4.9- Inserted comment regarding MAPEP soil series interferences and enhanced dissolution process outlined in section 12.11 3. Section 9- Included additional apparatus for performing the enhanced dissolution procedure. 4. Sections 10- Included additional reagents needed for performing enhanced dissolution. 5. Section 12.1 – Removed precipitate rinse using pH 10 DI water. 6. Section 12.4.2.2 – Added due to section 12.11. 7. Section 12.6.6- Changed wording to reflect when it is necessary to perform this step. 8. Section 12.7.4.5, 12.7.5- Adjusted to reflect volumes used for these steps. 9. Section 12.11- Added to provide instruction for performing enhanced dissolution by fusion method for Uranium/plutonium analysis of MAPEP solid samples. 10. Section 17 – Included references for fusion method.	26Sep2017
S-PGH-R-008-rev.13	1. Section 8.1.1.1 samples must be held minimum of 24 hours. 2. Section 2.1 updated to remove biota as a matrix. 3. Sections 8.1.2 and 8.1.3 updated to remove urine as a matrix. 4. Section 11.33 updated to broaden instructions for	08Feb2018

Document No.	Reason for Change	Date
	calibration verifications. 5. Section 12.3 deleted to remove reference to biota samples which are not analyzed by the lab. All subsequent sections moved up in the SOP. 6. Line references updated to adjust for removal of section 12.3. 7. Numerous sections updated to remove reference to urine and biota. 8. Section 12.5.6 and 12.5.7, updated to current practice with explanation. 9. Section 13.2 and 14.1 added to discuss uranium MCL violation determination and actions. 10. Section 14.8.2 updated to include DoD QSM method blank requirements. 11. Section 14.14.3 modified to enhance assessment of failing MS/MSDs.	

Attachment I - Calculations

The **radioactivity concentration** of a sample is calculated according to the following equations:

Eq. 1 $$A = \frac{(S_S / T_s)}{Denom}$$

Eq. 2 $$E_T = R * E$$

Eq. 3 $$E_i = \frac{S_E}{T_E * C_E * F_E * D_E}$$

Eq. 4 *E = average of individual isotope efficiencies*

Eq. 5 $$D = e^{-\lambda t}$$

Eq. 6 $$\lambda = \frac{\ln 2}{T_{1/2}}$$

Eq. 7 $$Denom = E_T * V * 2.22 * D * F$$

Eq. 8 $$R = \frac{(S_T / T_S)}{(E * C)}$$

Where:

A = The radioactivity concentration for the radioisotope being measured in units of pCi per Liter, gram, filter, or sample. ***"Activity."***

S_S = Represents the background corrected net counts for the radioisotope being measured. ***"Peak Net Cts."***

B = Represents the Bkg Cts acquired in the applicable region of interest (ROI) referenced to the sample count time. "***Bkg Cts (ref to Sample ct time)".***

T_S = Represents the count time for the sample. ***"Sample time (min)."***

T_B = Represents the count time for the background. ***"Bkg Time (min)."***

E_T = Represents the total system efficiency for the counted sample. This represents the detector efficiency corrected for chemical recovery. ***"Total Eff."***

V = Represents the sample volume (in liters), mass (in grams), filter portion analyzed (fractional), or sample portion analyzed (fractional). ***"Aliquot."***

2.22 = Represents the factor to convert from disintegrations per minute (dpm) to picocuries (pCi). ***"Act. Conv. From dpm to pCi."***

D = Represents the fraction of analyte remaining after decay time T. ***"Fract. Remain."***

F = Represents the summed branching ratio for all alpha particles emitted in the region of interest. In most cases this is 100% (+/- 1%). If the branching ratio varies significantly (greater than associated uncertainty) from 100%, this correction should be applied. ***"abnd."***

R = Represents the analytical chemical recovery for the tracer analyte. ***"Chemical Yield."***

S_T = Represents the background corrected net tracer peak counts in the applicable region of interest (ROI). Located in the **"Peak Net Cts"** column for the tracer nuclide row.

B_T = Represents the background counts for the tracer analyte acquired in the tracer region of interest (ROI) referenced to the sample count time. Located in the **"Bkg Cts (ref to Sample Ct time)"** column for the tracer nuclide row.

E = Represents the efficiency for the detector used for sample counting. The efficiency is a fixed number for each detector representing the fractional percent of radioactive events that are measured by the counting system. ***"Det. Eff. (cpm/dpm)."***

C = Represents the dpm of tracer (decay corrected to the time of sample counting) added to the sample. ***"Spike dpm."***

$T_{1/2}$ = Represents the half-life of the radionuclide being measured. ***"T1/2 (y)."***

t = Represents the elapsed time between the reference and count dates in the same units as $T_{1/2}$.

The sample specific **counting uncertainty** is calculated as follows.

Eq. 9 $$\textit{Counting Uncertainty} = \frac{1.96 * \sqrt{((S_S/T_S)/(T_S)) + ((B/Ts)/(T_B))}}{\text{Denom}}$$

As summed background and analyte count rates approach zero, assumptions underlying the uncertainty calculation are violated and it will return an unrealistic value of zero (0) uncertainty when zero summed counts are observed. The following equation provides a more accurate estimate of count uncertainty at zero and near-zero count rates.

Eq. 10 $$\text{ZeroUnc} = \frac{1.96 * \sqrt{zaf / T_S / T_S + zaf / T_B / T_B}}{Denom}$$

Where:

zaf = zero activity factor

and T_S, T_B, and Denom were previously defined

Note 1: Depending on sample type and contract requirements the zero activity factor may be either 3.0 or 2.71. PASI's default is 2.71 consistent with the current version of ANSI N42.23. Bioassay samples must be calculated using 3.0 to be consistent with ANSI N13.30.

Note 2: The Zero Count Uncertainty is compared to the count uncertainty above. The larger of the two is used as the counting uncertainty in subsequent total error calculations.

The error term is further evaluated to provide an estimate of total error hereafter referred to as the *Combined Standard Uncertainty* (CSU a.k.a. TPU).

Eq. 11 $$\text{CSU (pCi/unit)} = \sqrt{(\text{CountingUncertainty})^2 + (UE1 * A)^2 + (UE2 * A)^2 + (UE3 * A)^2 + (UE4)^2}$$

UE1, UE2, UE3, and UE4 represent partial derivatives estimating the relative uncertainty at the ***95% confidence interval*** for various factors in the activity calculation as follows:

UE1 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with preparation (e.g., sample aliquot or transfers and splits prior to addition and equilibration of tracer).

UE2 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with analysis (e.g., peak integration, peak overlap, tracer contaminants).

UE3 represents combined factors estimating relative uncertainty (fractional) associated with yield correction (e.g., count uncertainty for tracer peak, SRM known value, tracer volume or mass aliquot, tracer equilibration efficiency).

UE4 represents the factor estimating additional uncertainty (activity) associated with an individual sample -- to be used in exceptional circumstances with approval of the Department Manager or Manager-specified designee and with appropriate documentation and narration only.

The Minimum Detectable Concentration (MDC), Decision Level (DL) activity and Critical Level are calculated per guidance of ANSI N42.23 and N13.30 as:

Eq. 12 $$MDC = \frac{4.65 * \sqrt{(B/Ts) * T_S} + ZeroActFact}{Ts * Denom}$$

The Critical Level (LC), is calculated per guidance of ANSI N42.23 as:

Eq. 13 $$LC = \frac{1.65 * \sqrt{(B/Ts) * (1/T_S + 1/T_B)}}{Denom}$$

Attachment II - (Numerical Performance Indicators)

1. **Method Blank (MB)**

1.1 The numerical performance indicator for the method blank is calculated by:

$$Z_{Blank} = \frac{x}{u(x)}$$

Where:
x = Measured blank activity
u(x) = Combined standard uncertainty (1 sigma) in the blank measurement

1.2 MB performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. MB performance indicator values should be recorded on a control chart.

2. **Laboratory Control Sample (LCS)**

2.1 The numerical performance indicator for a laboratory control sample is calculated by:

$$Z_{LCS} = \frac{x - c}{\sqrt{u^2(x) + u^2(c)}}$$

Where:

x = Analytical result of the LCS
c = Known concentration of the LCS
$u^2(x)$ = Combined standard uncertainty (1 sigma) of the result squared.
$u^2(c)$ = Combined standard uncertainty (1 sigma) of the LCS value squared.

2.2 LCS performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. Performance indicator values should be recorded on a control chart.

3. **Duplicates (DUP)**

3.1 These criteria are applicable for the evaluation of the Duplicate, Matrix Spike Duplicate and Laboratory Control Sample Duplicates.

3.2 The numerical performance indicator for laboratory duplicates is calculated by:

$$Z_{Dup} = \frac{x_1 - x_2}{\sqrt{u^2(x_1) + u^2(x_2)}}$$

Where:
x_1, x_2 = Two measured activity concentrations
$u^2(x_1)$, $u^2(x_2)$ = The combined standard uncertainty (1 sigma) of each measurement squared.

3.3 Duplicate sample performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. DUP performance indicator values should be recorded on a control chart for each QC sample type (Dup, MSD, LCSD)

4. **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

4.1 The numerical performance indicator for a matrix spike sample is calculated by:

$$Z_{MS} = \frac{x - x_0 - c}{\sqrt{u^2(x) + u^2(x_0) + u^2(c)}}$$

Where:
x = measured concentration of the spiked sample
x_0 = measured concentration of the unspiked sample
c = spike concentration added
$u^2(x)$, $u^2(x_0)$, $u^2(c)$ = the squares of the respective combined standard uncertainties (1 sigma) of these values.

4.2 MS performance for all matrices is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. MS performance indicator values should be recorded on a control chart.

Figure 1. Analysis Flowchart for Sequential Analysis of Am, Pu, U, and Th (Method 2)

BOX 1___Precondition a small anion column with 20mL 8N HNO_3 – discard
___Dissolve sample in 15mL 8N HNO_3 - heat to aid in dissolution as necessary – samples must be clear – centrifuge if suspended solids are present
___Load sample onto conditioned columns – collect in clean c-tubes labeled **ALL**
___Rinse sample c-tube with 15mL 8N HNO_3 and add to columns – collect for **ALL**
___Rinse column with 15mL 8N HNO_3 – collect for **ALL**
___Pour contents of **ALL** c-tubes into clean glass beakers and heat to dryness. **Proceed to Box 3.**
___Place new c-tubes under columns labeled **TH** – elute and collect thorium by adding 25mL 9N HCl to columns. **Proceed to Box 2.**
___Save columns for re-use

BOX 2___Transfer **TH** fraction to new labeled 250mL disposable cup.
___Add 0.80ml of iron carrier and dilute to the 220mL mark with DI water
___Precipitate the thorium as a hydroxide by adding 22mL of NH4OH and stirring with a disposable pipette.
___Stir the samples vigorously after 15 minutes.
___Allow the precipitate to settle for 30 minutes, decant the supernate, and transfer the rest to the original c-tube with pH 10 DI water.
___Centrifuge and discard the supernate.
___Dissolve **TH** residue in 3mL 9N HCl
___Dilute the dissolved **TH** with DI water to a final volume of 25mL
___Microprecip **TH** with Neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 3___Add 15mL conc HCl to beakers labeled **ALL** and heat to dryness
___Dissolve contents of beakers labeled **ALL** in 15mL 9N HCl / 0.1% H_2O_2 – heat to aid in dissolution
___Load samples onto columns previously used – collect in c-tubes labeled **AM**
___Rinse **ALL** beakers with 10mL 9N HCl / 0.1% H_2O_2 and pour on columns – collect for **AM**
___Rinse columns with 20mL 9N HCl – collect for **AM. Proceed to Box 4**
___Place new c-tubes under columns labeled **PU –** elute and collect plutonium by adding 20mL 9N HCl / 0.05N NH_4I. **Proceed to Box 5**
___Rinse columns with 15mL 6N HCl / 0.52N HF – discard to waste
___Rinse columns with 10mL 9N HCl – discard
___Elute uranium into c-tubes labeled **U** by adding 20mL 0.1N HCl
___Microprecip **U** with neodymnium, titanous chloride to persistent purple color, and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 4___Transfer contents of c-tubes labeled **AM** to new glass beakers, add 3 drops Fe carrier and heat to dryness
___Add 5mL conc HNO_3 and heat to dryness
___Prepare Tru resin columns with pre-filter resin on bottom
___Condition columns with 5mL 2N HNO_3 - discard
___Dissolve contents of beaker in 10ml 2N HNO_3 / 0.5N $AlNO_3$
___Add 1 drop KSCN indicator and 6-8 drops of fresh 1N ascorbic acid – samples should be clear
___Pour sample onto columns – discard
___Rinse sample beaker with 5mL 2N HNO_3 – add to column - discard
___Rinse with 5mL 2N HNO_3 - discard
___Rinse with 5mL 2N HNO_3 - discard
___Elute AM into c-tubes with 2mL 9N HCL followed by 10mL 4N HCl
___Transfer to beakers and heat to dryness
___Add 1.0mL formic acid – heat to dryness
___Condition TEVA columns with 5mL fresh 3N Ammonium Thiocyanate / 0.1N Formic acid
___Dissolve samples in 15mL 3N AThio/ 0.1N formic
___Load sample onto columns – discard
___Rinse columns with 2mL 1N AThio/ 0.1N formic – discard
___Rinse with 3mL 1N AThio/ 0.1N formic – discard
___Rinse with 5ml 1N AThio/ 0.1N formic – discard
___Elute **AM** into new c-tubes with 15mL 2N HCl
___Microprecip **AM** with neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 5___Transfer **PU** fraction to new glass beakers
___Add 10mL conc HNO_3 and 3 drops iron carrier
___Heat until reactions subside
___Add H_2O_2 dropwise to destroy NH_4I – about 15 drops
___Heat to dryness
___Add 5mL conc HCl
___Heat to dryness
___Dissolve **PU** residue in 10mL 9N HCl
___Transfer to new c-tubes with DI water to a final volume of 20mL
___Microprecip **PU** with neodymnium, 10-12 drops 25% dihydrazine dihydrochloride, and HF
___Count to meet MDC and obtain 400 tracer counts.

Figure 2: Analysis Flowchart for Sequential Analysis for Am, Pu, U (Water or Solids) Method 1

Box 1___Prepare samples in accordance with the applicable section based on the matrix (12.1 for aqueous samples, 12.4 for solid samples). Solids must undergo complete digestion with HF.
___Condition prepared anion columns with 10mL 9N HCl.
___**Proceed to Box 2.**

BOX 4___Transfer **PU** fraction to new glass beakers
___Add 10mL conc HNO_3 and 3 drops iron carrier
___Heat until reactions subside
___Add H_2O_2 dropwise to destroy NH_4I – about 15 drops
___Heat to dryness
___Add 5mL conc HCl
___Heat to dryness
___Dissolve **PU** residue in 10mL 9N HCl
___Transfer to new c-tubes with DI water to a final volume of 20mL
___Microprecip **PU** with neodymnium, 10-12 drops 25% dihydrazine dihydrochloride, and HF
___Count for to meet MDC and obtain 400 tracer counts.

BOX 2___Dissove sample residue/precipitate in 15mL 9N HCl / 0.1% H_2O_2 – heat to aid in dissolution
___Centrifuge samples if necessary
___Load samples onto columns collect in c-tubes labeled **AM**
___Rinse sample c-tube with 10mL 9N HCl / 0.1% H_2O_2 and pour on columns – collect for **AM**
___Rinse columns with 20mL 9N HCl – collect for **AM.**
Proceed to Box 3
___Place new c-tubes under columns labeled **PU –** elute and collect plutonium by adding 20mL 9N HCl / 0.05N NH_4I. **Proceed to Box 4**
___Rinse columns with 15mL 6N HCl / 0.52N HF – discard to waste
___Rinse columns with 10mL 9N HCl – discard
___Elute uranium into c-tubes labeled **U** by adding 20mL 0.1N HCl
___Microprecip **U** with neodymnium, titanous chloride to persistent purple color, and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 3___Transfer contents of c-tubes labeled **AM** to new glass beakers, add 3 drops Fe carrier and heat to dryness
___Add 5mL conc HNO_3 and heat to dryness
___Prepare Tru resin columns with pre-filter resin on bottom
___Condition columns with 5mL 2N HNO_3 - discard
___Dissolve contents of beaker in 10ml 2N HNO_3 / 0.5N $AlNO_3$
___Add 1 drop KSCN indicator and 6-8 drops of fresh 1N ascorbic acid – samples should be clear
___Pour sample onto columns – discard
___Rinse sample beaker with 5mL 2N HNO_3 – add to column - discard
___Rinse with 5mL 2N HNO_3 - discard
___Rinse with 5mL 2N HNO_3 - discard
___Elute AM into c-tubes with 2mL 9N HCL followed by 10mL 4N HCl
___Microprecip **AM** with neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

Figure 3: Analysis Flowchart for Sequential Analysis of Am and Pu in Solids >1g

Box 1___1. Aliquot 3-4 grams of dry pulverized solid into clean labeled ceramic crucible.
___2. Add 1.0mL of iron carrier. Add tracers to all samples, and spike solution to LCS and MS.
___3. Cover sample with crucible lid and place in oven at 550C overnight (minimum of 4 hours).
___4. Remove samples from oven, re-label, and carefully loosen solid with a disposable pipette.
___5. Transfer loosened solid to a labeled glass beaker with a minimal amount of 8N HNO_3.
___8. Add 5mL 16N HNO_3 and 5mL 12N HCl to the crucible and place on hotplate at medium heat to loosen/remove the remaining solid.
___9. Transfer this solution to the appropriate glass beaker with 9N HCl. Repeat the above step as necessary to remove solid from the crucible.
___10. __ __Add 15mL 16N HNO_3 and 15mL 12N HCl to each sample beaker.
___11. __ __Cover sample with a watch glass and leach for 30 minutes on hotplate at medium heat.
___12. __ __Cool sample and transfer to a centrifuge tube using DI water. Centrifuge the sample.
___13. __ __Transfer the supernate to a clean labeled glass beaker and heat to dryness.
___14. __ __Transfer the solid back to the original beaker with 9N HCl.
___15. Repeat steps 10-14 two additional times adding the supernate from each centrifuge cycle to the beaker in step 13. The solid should be lighter in color indicating complete leaching of iron metal from the sample.
___16. Heat the supernate to dryness.
___17. Add 10mL 12N HCl to each sample beaker and heat to dryness. **Proceed to Box 2.**

BOX 4___Transfer **PU** fraction to new glass beakers
___Add 10mL conc HNO_3 and 3 drops iron carrier
___Heat until reactions subside
___Add H_2O_2 dropwise to destroy NH_4I – about 15 drops
___Heat to dryness
___Add 5mL conc HCl
___Heat to dryness
___Dissolve **PU** residue in 10mL 9N HCl
___Transfer to new c-tubes with DI water to a final volume of 20mL
___Microprecip **PU** with neodymnium, 10-12 drops 25% dihydrazine dihydrochloride, and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 2___Precondition small anion column with 10mL 9N HCl.
___Dissolve contents of beakers in 15mL 9N HCl / 0.1% H_2O_2 – heat to aid in dissolution – centrifuge sample
___Load samples onto columns – collect in c-tubes labeled **AM**
___Rinse beakers/c-tubes with 10mL 9N HCl / 0.1% H_2O_2 and pour on columns – collect for **AM**
___Rinse columns with 20mL 9N HCl – collect for **AM.**
Proceed to Box 3
___Place new c-tubes under columns labeled **PU –** elute and collect plutonium by adding 20mL 9N HCl / 0.05N NH_4I.
Proceed to Box 4
___Rinse columns with 15mL 6N HCl / 0.52N HF – discard to waste
___Rinse columns with 10mL 9N HCl – discard
___Elute uranium into c-tubes labeled **U** by adding 20mL 0.1N HCl
___Microprecip **U** with neodymnium, titanous chloride to persistent purple color, and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 3___Transfer contents of c-tubes labeled **AM** to new glass beakers, add 3 drops Fe carrier and heat to dryness
___Add 5mL conc HNO_3 and heat to dryness
___Prepare Tru resin columns with pre-filter resin on bottom
___Condition columns with 5mL 2N HNO_3 - discard
___Dissolve contents of beaker in 10ml 2N HNO_3 / 0.5N $AlNO_3$
___Add 1 drop KSCN indicator and 6-8 drops of fresh 1N ascorbic acid – samples should be clear
___Pour sample onto columns – discard
___Rinse sample beaker with 5mL 2N HNO_3 – add to column - discard
___Rinse with 5mL 2N HNO_3 - discard
___Rinse with 5mL 2N HNO_3 - discard
___Elute AM into c-tubes with 2mL 9N HCL followed by 10mL 4N HCl
___Transfer to beakers and heat to dryness
___Add 10mL HCl and heat to dryness
___Add 1.0mL formic acid – heat to dryness
___Condition TEVA columns with 5mL fresh 3N Ammonium Thiocyanate / 0.1N Formic acid
___Dissolve samples in 15mL 3N AThio/ 0.1N formic
___Load sample onto columns – discard
___Rinse columns with 2mL 1N AThio/ 0.1N formic – discard
___Rinse with 3mL 1N AThio/ 0.1N formic – discard
___Rinse with 5ml 1N AThio/ 0.1N formic – discard
___Elute **AM** into new c-tubes with 15mL 2N HCl
___Microprecip **AM** with neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

Figure No 4: Analysis Flowchart for Sequential Analysis of U and Th in solid or water utilizing Anion Resin.

BOX 1___Precondition a small anion column with 20mL 8N HNO_3 – discard
___Dissolve sample in 15mL 8N HNO_3 - heat to aid in dissolution as necessary – samples must be clear – centrifuge if suspended solids are present. An extra 5mL of 8N HNO_3 may be used if transferring solids to a centrifuge tube.
___Load sample onto conditioned columns – collect in clean c-tubes labeled **U**
___Rinse sample c-tube with 15mL 8N HNO_3 and add to columns – collect for **U**
___Rinse column with 15mL 8N HNO_3 – collect for **U**
___Pour contents of **U** c-tubes into clean glass beakers and heat to dryness. **Proceed to Box 3.**
___Place new c-tubes under columns labeled **TH** – elute and collect thorium by adding 25mL 9N HCl to columns. **Proceed to Box 2.**
___Save columns for re-use

BOX 2___Transfer **TH** fraction to new labeled 250mL disposable cup.
___Add 0.80ml of iron carrier and dilute to the 220mL mark with DI water
___Precipitate the thorium as a hydroxide by adding 22mL of NH4OH and stirring with a disposable pipette.
___Stir the sample vigorously after 15 minutes.
___Allow the precipitate to settle for 30 minutes, decant the supernate, and transfer the rest to the original c-tube with pH 10 DI water.
___Centrifuge and discard the supernate.
___Dissolve **TH** residue in 3mL 9N HCl
___Dilute the dissolved **TH** with DI water to a final volume of 25mL
___Microprecip **TH** with Neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 3___Add 5mL conc HCl to beakers labeled **U** and heat to dryness
___Dissolve contents of beakers labeled **U** in 15mL 9N HCl– heat to aid in dissolution
___Load samples onto columns previously used
___Rinse **U** beakers with 10mL 9N HCl and pour on columns
___Rinse columns with 20mL 9N HCl
___Rinse columns with 20mL 6N HCl / 0.52N HF – discard to waste
___Rinse columns with 10mL 9N HCl – discard
___Elute uranium into c-tubes labeled **U** by adding 20mL 0.1N HCl
___Microprecip **U** with neodymnium, titanous chloride to persistent purple color, and HF
___Count to meet MDC and obtain 400 tracer counts.

ATTACHMENT C-24

ANALYSIS OF WATER SAMPLES FOR RA-226 CONTENT 903.1
PACE, PITTSBURGH

Document Information

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Document Title: Analysis of Water Samples for Ra-226 Content - 903.1

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STANDARD OPERATING PROCEDURE

Analysis of Water Samples for Radium-226

Methods: EPA 903.1, SM7500-Ra C

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APPROVALS

Maureen K. DeRubeis
Senior Quality Manager — 02/08/18 Date

Department Manager/Supervisor — 02/08/18 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date

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Table of Contents

1. Purpose

1.1 This SOP documents the analytical procedure to be used for analysis drinking water and other aqueous samples for radium-226.

2. Scope and Application

2.1 This procedure covers the measurement of radium-226 in drinking water samples and should be employed after the gross alpha or gross radium alpha screening technique had indicated possible non-compliance with the alpha radioactivity limits set forth in the Safe Drinking Water Act, PL 93-523. 40 FR 34324.

2.2 Additionally, this SOP is applicable to the analysis of other aqueous sample types without modification.

2.3 This procedure is specific for radium-226, and is based on the emanation of radon-222, a daughter product of radium-226. Radium-226 concentration is determined by scintillation counting of radon-222, polonium-218, and polonium-214 (daughters of radon-222).

2.4 The detection limit for this procedure assures measuring radium-226 concentrations lower than 1.0pCi/L and potentially as low as 0.1pCi/L.

2.5 Without qualification, this procedure, as written, is compliant with Method 903.1 of "*Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA-600/4-80-032". Deviations from the promulgated methods are discussed in Section 18 of this SOP.

2.6 Pace Analytical applies isotope decay correction only in instances where the total impact in the analysis result is 2% or greater. Assuming a maximum hold time of 180 days, a 2% isotope decay would occur only for radioisotopes with a half-life of 17.14 years or less. The parameters reported in this SOP are not affected by this policy. Decay correction has not been applied to the parameters measured by this SOP.

3. Summary of Method

3.1 The radium-226 in the drinking water sample is concentrated and separated by co-precipitation on barium sulfate. The precipitate is dissolved in EDTA reagent and placed in a sealed bubbler. Radon-222 is flushed from the sample using inert helium or nitrogen gas. The bubbler is stored for ingrowth of radon-222. After ingrowth, the gas is purged into a scintillation cell. When the short-lived radon-222 daughters are in equilibrium with the parent, the scintillation cell is counted for alpha activity.

3.2 The absolute measurement of radium-226 is ensured by calibrating the scintillation cell system with a standard solution of radium-226.

4. Interferences

4.1 There are no radioactive interferences in this method.

4.2 To prevent possible cross contamination during the emanation of multiple samples through the same apparatus, apply vacuum intermittently to the apparatus and attached drying tube. This will allow the drying tube and apparatus to be flushed with room air.

4.3 To prevent contamination of bubblers, samples with visible suspended material must be centrifuged prior to transfer to the bubbler for ingrowth.

5. Safety

5.1 Procedures must be carried out in a manner that protects the health and safety of all personnel. Since this analysis is for a radioactive constituent, the sample must be treated as radioactive.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves will be cleaned immediately.

5.3 When mixing or diluting acids, always add the acid slowly to water and swirl. Dilution of acids must always be done in a hood. Appropriate eye-protection, gloves, and lab coat must be worn.

5.4 Exposure to radioactivity and chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous and/or non-radioactive, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 In order to minimize the potential for cross contamination of high and low levels of radioactive samples, good housekeeping and good laboratory practices are essential and must be strictly adhered to.

5.6 Organic samples of unknown content must be handled with extreme caution and under the direct instruction of a Department Manager/Supervisor or Department Manager/Supervisor's specified designee. Direct treatment of organic matrices with strong oxidizing chemicals such as nitric acid and/or hydrogen peroxide is strictly prohibited.

5.7 Hydrofluoric acid is particularly hazardous because a serious skin exposure may cause no immediate sensation of pain. The acid penetrates the skin and spreads internally, causing tissue damage deep under the skin. The resulting burn is painful, difficult to treat, and easily infected. Gloves must be checked for pinhole leaks before use. They must be rinsed before they are removed and must be discarded after use. HF burn gel shall be put on suspected HF burns after flushing (except the eyes) until medical help can be obtained. Medical attention shall be sought even if suspicions arise after working hours. Contact your group leader immediately for further information if a HF burn is suspected.

5.8 In addition, HF vapors are also hazardous. Exposure can cause permanent damage. Breathing HF vapors even for a short time and at a low temperature can be injurious to the respiratory system and even fatal. All such direct contact must be avoided.

5.9 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory.

5.10 Refer to the Pace Analytical Services, LLC. – Pittsburgh Chemical Hygiene Plan/Safety Manual for the specific safety requirements to be followed when working in the laboratory.

5.11 The toxicity and carcinogenicity of each reagent used in this procedure has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. At a minimum, personal protective equipment must include a lab coat, gloves, and safety glasses.

5.12 Analysts must be familiar with the Safety Data Sheets (SDS) for all chemicals and reagents used in this procedure, and the location of the SDS within the laboratory.

6. Definitions

6.1 See the glossary section of the most recent revision of the Pace Analytical Services LLC. Quality Assurance Manual for commonly used laboratory terms.

6.2 Batch: For all matrices, an analytical batch contains 20 or fewer samples of a similar matrix, prepared at the same time, by the same analyst, using the same reagents.

6.3 Throughout this procedure, approximate weights and measures will be designated by the use of whole numbers when referring to masses exceeding 1g or volumes in excess of 1mL. Measurements of weights and volumes so designated can be made with top loading balances, graduated cylinders, etc. For approximate measures below 1g or 1mL, the word "approximately" must be used prior to the described mass or volume.

6.4 Throughout this procedure, exact or critical masses and volumes will be designated by the use of one or more decimal places. Measurements of masses and volumes so designated should be made with accurate analytical instruments such as analytical balances, calibrated pipettes, etc.

6.5 Observed masses must be recorded in logbooks to the lowest weight indicated on the balance. Sample aliquot masses cannot be targeted. Once sample is aliquotted, it cannot be removed from the beaker.

6.6 The method employed to measure the sample, whether it be by balance, pipette, or graduated cylinder, must be clearly documented in the preparation logbook.

7. Responsibilities and Distribution

7.1 General Manager/Assistant General Manager (GM/AGM)

7.1.1 The GM/AGM has the overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the laboratory involving the collection and reporting of analytical data.

7.1.2 The GM/AGM and Senior Quality Manager/Quality Manager have final review and approval authority for all SOPs prepared within the laboratory.

7.2 Senior Quality Manager/Quality Manager (SQM/QM)

7.2.1 The SQM/QM will maintain a master file of all SOPs applicable to the operations departments.

7.2.2 The SQM/QM will assign a unique number to each SOP prepared prior to approval and distribution.

7.2.3 The SQM/QM will distribute SOPs to applicable personnel and maintain an accurate accounting of such distribution to ensure that the SOPs, in the hands of the users, are current and complete.

7.3 Department Manager/Supervisor

7.3.1 The Department Manager/Supervisor is responsible for ensuring all staff members read, follow, and are adequately trained in the use of the SOPs

7.3.2 The Department Manager/Supervisor coordinates the preparation and revision of all SOPs within the department whenever a procedure changes.

7.3.3 The Department Manager/Supervisor provides initial approval of all SOPs within the department.

7.3.4 The Department Manager/Supervisor makes recommendations for SOP revision to the SQM/QM via written memo.

7.4 Individual Staff

7.4.1 Individual staff members are responsible for adherence to the specific policies and procedures contained in the applicable SOPs.

7.4.2 Individual staff members will only use a signed, controlled copy of the SOP. Each person may make recommendations to the Department Manager/Supervisor for revising SOPs as the need arises.

7.4.3 Personnel are responsible for ensuring that any deviations from this SOP are reported to the Department Manager/Supervisor.

8. Sample Collection, Preservation, and Handling

8.1 Plastic or glass containers may be used for sample collection.

8.1.1 Containers used for sample collection must never be re-used.

8.2 Aqueous samples must be preserved at the time of collection by adding enough concentrated (16N) HNO_3 to the sample to make the sample pH <2. Typically, 2mL of 16N HNO_3 per liter of sample is sufficient to obtain the desired pH. Samples must be preserved within five days of collection. If samples are collected without preservation, they must be received by the laboratory and preserved within five days of collection. Following preservation with acid, samples must be held in the original container for a minimum of 24 hours before analysis or transfer of sample. Samples preserved upon receipt at the laboratory, must be re-checked by laboratory personnel a minimum of 24 hours after preservation. The pH re-check date and time, the initials of the analyst verifying the pH, as well as any adjustments or notes regarding the preservation must be recorded in the pH Verification Logbook.

8.2.1 For dissolved analysis, samples must be filtered through a 0.45μm membrane filter and preserved to a pH <2.

8.2.2 For total analysis, the sample is not filtered, but is preserved.

8.3 Refrigeration is not required for aqueous samples.

The maximum hold time for samples analyzed by this SOP is 180 days from collection to analysis.

9. Equipment and Supplies

9.1 Scintillation Cell system.

9.2 Radon emanation apparatus consisting of 1) radon bubbler, 2) scintillation cell and 3) a drying tube.

9.3 Electric hot plate.

9.4 Centrifuge and disposable 50mL centrifuge tubes.

9.5 Membrane filters, 0.45μm, 47mm, Metricel® or equivalent.

9.6 Glassware, various sizes.

9.7 Analytical balance.

9.8 Vacuum manifold.

9.9 Vortex mixer

9.10 Hot water bath.

10. Reagents and Standards

10.1 Reagents should be prepared from reagent grade chemicals, unless otherwise specified below. NOTE: Consult the Safety Data Sheets for the properties of these reagents, and how to work with them.

10.2 Distilled or deionized (DI) water. ASTM Type II as produced using the specifications documented in SOP PGH-C-027, current revision.

10.3 Acetic acid, 17.4N: glacial CH_3COOH (conc.), sp. Gr. 1.05, 99.8%.

10.4 Ammonium hydroxide, 15N: NH_4OH (conc.), sp. gr. 0.90, 56.6%.

10.5 Ammonium sulfate, 200mg/mL: Dissolve 20 g $(NH_4)_2SO_4$ in water and dilute to 100 mL.

10.6 Anti-foaming agent, Anti-foam B, or equivalent.

10.7 Ascarite, drying reagent: 8-20 mesh.

10.8 Barium carrier, 16 mg/mL, standardized. Dissolve 28.46 g $BaCl_2 \bullet 2H_2O$ in water, add 5.0 mL 16N HNO_3, and dilute to 1.0L with ASTM Type II DI Water.

10.9 Citric acid, 1M: Dissolve 192 g of $C_6H_8O_7 \bullet H_2O$ in water and dilute to 1.0L with ASTM Type II DI water.

10.10 EDTA reagent, basic, (0.25M): Dissolve 20 g NaOH in 750mL water and slowly add 93g disodium ethylenedinitriloacetate dihydrate, $(Na_2C_{10}H_{14}O_8N_2 \bullet 2H_2O)$ while stirring. After the salt is in solution, dilute to 1L with ASTM Type II DI Water. The heat generated from the solid sodium hydroxide added to DI water should be sufficient so as to support complete dissolution of EDTA. If the EDTA does not readily dissolve, gradually heat the reagent until dissolved.

10.11 Helium Gas, ultra high purity.

10.12 Nitrogen gas, ultra high purity.

10.13 Lead carrier, 150mg/mL: Dissolve 239.7 g $Pb(NO_3)_2$ in ASTM Type II DI water, add 5.0mL 16N HNO_3 and dilute to 1.0L with ASTM Type II DI water.

10.14 Methyl Orange Indicator, 0.1%: Dissolve 0.1g methyl orange indicator in 100mL ASTM Type II DI water.

10.15 Nitric acid, 16N: HNO_3 (conc.), Sp. Gr. 1.42, 70.4%.

10.16 Magnesium perchlorate, $Mg(ClO_4)_2$: reagent grade.

10.17 Radioactivity standard solutions: radium-226 may be utilized for batch control spike samples or instrument calibrations and barium-133 may be used for radio-tracer yield determinations. All radioactive standards must be NIST traceable.

10.18 Sodium hydroxide, 10N: Dissolve 400 g NaOH in 500 mL deionized water, cool, and dilute to 1.0L with ASTM Type II DI Water.

10.19 Sulfuric acid, 18N: 50% V/V, ACS grade. Yttrium carrier, 18 mg/mL: Add 22.85g Y_2O_3 to an Erlenmeyer flask Add 50mL of ASTM Type II DI water. Carefully add 40mL of concentrated nitric acid. Heat the mixture to boiling while stirring on a magnetic stirring hotplate. The solution must heat to boiling. Additional water and nitric acid may be added as necessary to aid in dissolution. Measure the additions of Type II DI water and concentrated nitric acid and record the reagent volumes added into the reagent preparation logbook. Scrape the bottom of the Erlenmeyer flask with a Teflon® scraper if the yttrium oxide is caked onto the glass to enhance dissolution. Once the yttrium oxide has completely dissolved, add a quantity of concentrated nitric acid so that the total volume of

concentrated nitric acid added is 100 mL. Do not add more than 100mL of nitric acid total. Upon complete dissolution, remove the beaker from the hotplate and cool completely. Dilute to 1L with ASTM Type II DI water.

11. Calibration

11.1 Cell/Detector calibrations are performed uniformly, independent of sample analysis matrix type. The calibration values determined by application of this process are applied universally for all matrices for which the analytical process is defined in this SOP.

11.2 Cell/Detector calibrations must be performed on an annual basis.

11.3 Transfer between 500 to 1500 dpm of radium-226 (25mL volume) standard into a bubbler that has been designated for calibration purposes only.

11.4 Note: The specified calibration source activities have been optimized to allow manageable count times for individual sources.

11.5 Attach the bubbler to the radon assembly.

11.6 With the scintillation cell disconnected, bubble helium (or nitrogen) gas through the solution for 20 minutes to remove all Rn-222.

11.7 Close both stopcocks on the bubbler to establish zero time for ingrowth of Rn-222. Record the date and the time of the calibration source flushing on the appropriate bench sheet. Store the bubbler to allow ingrowth of Rn-222.

11.8 Following adequate ingrowth time for the calibration source, proceed with steps 12.12 through 12.18, Radon Emanation Technique.

11.8.1 EPA Method 903.1, “Radium-226 in Drinking Water Radon Emanation Technique” requires a minimum of 18 hours of ingrowth for the calibration. Pace has adopted a minimum ingrowth period of four days for calibrations and samples.

11.9 The calibration constant includes the de-emanation efficiency of the system, the counting efficiency of the cell, and the alpha activity contributed by Po-218 and Po-214, which will be in equilibrium with Rn-222 when the sample is counted approximately 4 hours (minimum of 3 hours) after the de-emanation. Calibration sources must be counted until a minimum of 10,000 counts has been obtained.

11.10 Proceed to Section 13 to determine the cell calibration constant.

11.11 Following the cell counting, flush the cell by alternating vacuum and room air to remove Rn-222 from the cell. Store the cell for a minimum of 24 hours prior to use for samples. This delay allows adequate decay of Rn-222 daughters. Lucas cells must be counted for a minimum of ten minutes to determine the cell background prior to use for sample analysis.

11.12 The bubbler used for Ra-226 calibration should not be used for sample analysis. It should be set aside and retained for future calibrations. The

cell/detector combination must be calibrated annually. Additionally, calibrations must be performed if either the cell or the detector undergo any major maintenance. New cells and cells returned from a repair facility must be given a new ID.

11.13 For cells previously successfully calibrated, the new cell constant should be within 10% of the previously acceptable cell constant (CAL A) counted in the same detector. Typical cell constants will be around 2.2 to 2.5, and may vary for the same cell among each individual detector. For a new calibration attempt (CAL B), any deviation outside of the 10% rule may indicate a problem during calibration (CAL B) and the calibration should be re-performed using a different calibration source (bubbler). If the new calibration value (CAL C) is within 5% of the most recent calibration value (CAL B), the newest calibration value (CAL C) should be used as the cell/detector calibration value. If the new calibration value (CAL C) is greater than 5% of the most recent calibration value (CAL B) but within 10% of the previously-implemented calibration value (CAL A), this indicates that the process in performing CAL B was compromised. In this case, the newest calibration value (CAL C) should be used as the cell/detector calibration value. Cells for which calibration values do not comply with the listed requirements must be removed from future service until appropriate corrective action has been applied and documented. At a minimum, prior to repeating the calibration process, check the cell for damage, especially around the stopcock, base of the stem, and the window, which could cause gas loss during calibration.

11.14 Cell constants will vary slightly based on the imperfect mechanism of applying the zinc sulfide interior coating, as well as minor differences in cell volume. Cells which cannot be calibrated should be sent out for repair which will include resealing the stopcock stem, re-applying the zinc sulfide coatings, and cleaning and replacing the glass surface.

12. Procedure

Unless specified otherwise, the documented analysis process must be followed, as written, including the order of analytical process and the addition of chemicals

12.1 Weigh approximately 500g of aqueous sample into an appropriately sized glass beaker. Record the observed mass of the sample added to the beaker (do not remove any sample from the beaker). The actual amount of sample used may vary based on sample availability and expected or possible matrix interferences, but the routine amount is approximately 500 grams. When using < 500g, samples should be diluted with ASTM Type II DI water to the 500 ml mark on the beaker, and acidified with nitric acid.

12.2 Prepare a Method Blank (MB), Laboratory Control Sample (LCS), and Laboratory Control Sample Duplicate (LCSD), by weighing about 500g of ASTM Type II DI water into a labeled beaker, and record the actual mass observed in the logbook. Add 2.0 ml of concentrated nitric acid to the MB, LCS, and LCSD. Add the appropriate amount of Ra-226 spike

solution to the LCS and LCSD based on the requirements listed in Section 14.6.3 of this SOP.

12.3 If a Matrix Spike (MS) is prepared (MS is required when analyzing drinking water samples or when specified by the client), add the appropriate amount of spike solution to the MS sample based on the requirements of Section 14.9.2 of this SOP.

12.4 To all samples including the QC samples, add 5mL 1N citric acid solution and a few drops methyl orange indicator. The solution should be red. If the solution is not red, check the solution pH with pH indicator strips capable of determining pH 0-14. The sample pH must be <2.0. Otherwise, refer to Section 8.2 regarding sample preservation requirements.

12.5 Add 1mL lead carrier (150 mg/mL), 1mL yttrium carrier (18 mg/mL) and 2.0mL barium carrier (16 mg/mL). Add a suitable amount of barium-133 tracer based on the guidance in section 14.11 if yield determination is to be made by radio-tracer counting. Stir well. Heat to incipient boiling and maintain at this temperature for 30 minutes.

12.6 Add a few drops methyl orange indicator again as it is destroyed upon prolonged heating. Add 15N NH_4OH until a definite yellow color is obtained, then add a few drops excess. Precipitate lead and barium sulfates by adding 18N H_2SO_4 until the red color reappears, then add 0.25mL excess. Add 5mL $(NH_4)_2SO_4$ (200 mg/mL) and stir vigorously until a precipitate forms. Allow the solution to heat for a minimum of 30 minutes, and then remove the samples from the hot plate to cool.

12.7 Allow the sample precipitate to settle overnight or for a minimum of 2 hours until completely clear; then siphon most of the supernatant liquid and discard, saving the precipitate.

12.8 Transfer the precipitate with the aid of 0.1N H_2SO_4 to a 50mL disposable centrifuge tube; centrifuge, and discard the supernatant liquid.

12.8.1 For wastewater samples or samples containing suspended solids, add 20mL of 16N HNO_3 to each centrifuge tube and heat in a hot water bath for 30 minutes. This step will aid in the removal of interfering element components.

12.8.2 Centrifuge and discard acid solution into an appropriate waste stream. Rinse the precipitate in 20mL 16N HNO_3. Shake to mix, centrifuge, and discard acid rinses into an appropriate waste stream.

12.8.3 Rinse the precipitate two times with 10mL aliquots of 0.1N H_2SO_4. Add the acid to the precipitate in the centrifuge tube, cap and vortex to rinse. Centrifuge and discard the supernate into the appropriate waste stream.

12.9 Add 25mL 0.25M EDTA solution to the sulfate precipitate. Vortex the samples and heat in a water bath to enhance dissolving of the precipitate.

If the precipitate does not readily dissolve, add 10N NaOH solution dropwise. Do not add more than 7 drops total of the 10N NaOH.

12.9.1 If there is still undissolved material, centrifuge the sample and transfer the solution to a clean disposable centrifuge tube. Submit the supernate solution for gamma spectroscopy counting for Ba-133 determination. If the resulting yield is within acceptable limits, continue with the analysis. If it is not within the expected limits, combine the supernate and the solid material and reheat.

12.10 Ensure that the sample solution volume is 25mL (+/- 1 mL) using the graduated markings on the centrifuge tube as a guide. If the sample volume is less than 25 mL, dilute the sample to 25 mL(+/- 1 mL) with 0.25M EDTA solution using the graduated markings on the centrifuge tube as a guide.

12.10.1 Optionally: Proceed to Section 12.21 to determine the radiotracer yield prior to transferring the solution to the bubbler. If this option is used, extreme care must be exercised when transferring the solution from the centrifuge tube to the bubbler in order to ensure that it is performed quantitatively.

12.11 Centrifuge and transfer the supernate to a labeled radon bubbler. Discard the centrifuge tube into an appropriate waste stream.

12.12 Connect the bubbler to the helium (nitrogen) source then open both the upper and the lower stopcocks of the radon bubbler and de-emanate the solution by slowly passing helium (nitrogen) gas through the bubbler for about 20 minutes.

12.12.1 Add 2 drops of antifoaming agent to the bubbler if excessive foaming occurs while passing the helium (nitrogen) through the solution.

12.13 Close the two stopcocks and record the time. Store the solution to allow for ingrowth of Rn-222.

12.13.1 A minimum of 4 days of ingrowth is required.

12.14 At the end of the storage period, fill the upper approximate half of an absorption tube with magnesium perchlorate and the lower approximate half with ascarite. Magnesium perchlorate may clump over time preventing easy transfer to the absorption tube; if necessary, grind a portion of the reagent using a mortar and pestle prior to adding to the tube. Attach the drying tube to the vacuum monitoring manifold with a rubber o-ring and clamp. Ensure the end with the white magnesium perchlorate is at the top, and the ascarite end is at the bottom.

12.14.1 Note:For minimizing corrections that would be required in subsequent calculations, the voids above the bubbler must be kept very small. Capillary tubing should be used whenever possible, and the drying tube volume with the ascarite and magnesium chlorite must be kept to a minimum. A typical system consists of a drying tube 10 cm x 1.0 cm (I.D.), with each

of the drying agents occupying 4 cm and being separated by small glass wool plugs. The column can be reused several times before the chemicals need to be replaced as long as the ascarite appears dry and not hardened.

12.15 Attach the sample bubbler to the drying tube so the liquid containing portion of the bubbler is directly below the drying tube, and attach the helium (nitrogen) supply hose to the bubbler inlet (the thin capillary tube side).

12.16 Evacuate the desired Lucas cell using a vacuum pump and close the stopcock. Ensure a background count has been performed for the cell and detector combination prior to each use. Using the appropriate clip, firmly attach the Lucas cell to the top of the vacuum monitoring manifold connected to the correct bubbler.

12.17 Open the stopcock on the scintillation cell and check the vacuum gauge to ensure that vacuum is maintained. The gauge should indicate a vacuum of approximately -20 to -25 psi relative to atmospheric. Gradually open the outlet stopcock on the bubbler. When the stopcock is fully open and no further significant bubbling takes place, the pressure gauge should still indicate a vacuum, but less vacuum relative to atmospheric, approximately -15 to -20 psi

12.18 Adjust the helium (nitrogen) gas pressure so that the gas flows at slightly above atmospheric pressure.

12.19 Gradually open the inlet stopcock on the bubbler using the bubbling as a guide. Continue bubbling with the helium (nitrogen) gas until the vacuum gauge indicates neutral (atmospheric) pressure. The de-emanation process should take approximately 15-20 minutes to complete. Toward the end of the de-emanation, when the vacuum is no longer effective, it will be necessary to gradually increase the helium (nitrogen) gas pressure. When the system is at atmospheric pressure, close the inlet and outlet stopcocks of the cell and bubbler, shut off the gas, and disconnect the tubing from the bubbler inlet. Record this time as the beginning of the Rn-222 decay and ingrowth of Rn-222 daughters.

12.20 Store the scintillation cell for approximately 4 hours (minimum of 3 hours) to ensure equilibrium between radon and radon daughters. Proceed to Section 12.22 to “count” the Lucas cells.

12.21 If not already determined in Step 12.10, transfer the sample bubbler contents to a labeled 50mL centrifuge tube.

12.21.1 If gamma counting is to be performed to determine yield by Ba-133 counting, dilute the tube contents to a final volume of 30mL with ASTM Type II DI water, cap the tubes, shake vigorously, and submit them to the analyst responsible for gamma counting.

12.21.2 Barium yield by gravimetric assessment (used if Ba-133 is unavailable or not suitable to sample matrix).

12.21.2.1 If yield assessment is to be made by gravimetric measurement of barium sulfate, proceed as follows:

12.21.2.2 Add 1mL of ammonium sulfate solution (200 mg/mL) and stir thoroughly. Add 2mL conc. acetic acid until barium sulfate precipitates. Digest in a hot water bath for 10 minutes. Cool, centrifuge, and discard the supernate.

12.21.2.3 Slurry the precipitate with 10ml of ASTM Type II DI water, centrifuge and discard the supernate.

12.21.2.4 Slurry the precipitate with 7mL of ASTM Type II DI water and transfer to a tared, 2-inch stainless steel planchet. Dry the precipitate under a heat lamp, cool and re-weigh for yield determination. Refer to Section 13 of this SOP for barium recovery calculation.

12.22 Lucas Cell Counting

12.22.1 Perform the required daily source check prior to counting cells for daily background. Daily source check counting and routine maintenance for the Ludlum Counters is specified in the associated instrument operations SOP, PGH-R-065, current revision.

12.22.2 Remove the top cover from the photomultiplier tube (PMT) unit or counter, and remove the rubber protector from the base of the Lucas cell. Center the Lucas cell on the glass platform on the top of the base of the counter and carefully replace the top cover.

12.22.3 Press the top cover down firmly to ensure the counting rod makes contact with the counter base. There should be an audible click when the counting rod is properly engaged. Tighten the screws on both sides of the top cover to ensure the counting rod remains engaged through sample counting.

12.22.4 Set the count time on the front of the Model 2000 Scaler unit to the count time necessary to meet the desired reporting limit. Count times should not be less than 10 minutes, and should not routinely exceed 20 minutes.

12.9.4.1 Count times longer than 20 minutes should only be used for drinking water samples where it is necessary to achieve the Ra-226 reporting limit of 1.0 pCi/L.

12.22.5 Press the COUNT button on the front of the scaler unit. Record this time as the count start time in the appropriate section of the analysis logbook.

12.9.4.2 A red light above the COUNT button will light to indicate the unit is active. Observe counts on the LED display to ensure the counting rod is correctly engaged on the counter.

12.9.4.3 Do not remove the top cover while the unit is counting as this may damage the photomultiplier tube.

12.22.6 Once the red light has turned off, record the counts from the LED display into the appropriate section of the analysis logbook.

12.22.7 Unscrew the screws on the sides of the top cover of the counter and carefully lift the top cover off of the counter ensuring the cell is not knocked over. Return the rubber protector to the base of the Lucas cell, and set the Lucas cell aside for evacuation.

12.22.8 Repeat steps 12.22.2 through 12.22.7 until all Lucas cells have been counted.

12.22.9 Following counting and after data have been processed, flush the Lucas cell by alternating vacuum and helium (nitrogen) gas to remove Rn-222 from the cell. Store the cells with vacuum applied for a minimum of 3 hours prior to using for another sample. A new background must be counted on the cell prior to use.

13 Calculations

13.1 Refer to Attachment I of this SOP for Ra-226 associated calculations.

13.1 Any verified result for drinking water that exceeds the maximum contaminant level (MCL) established for Radium-226 must be reported to the appropriate personnel and agencies according to the specific requirements of the state where the water was sampled. The directions for reporting any results that exceed the MCL limits are documented in the State Drinking Water Emergency Reporting Requirements Binder and in Pace SOP PGH-C-025, current revision.

13.2.1 The Ra-226 MCL for drinking water is defined as >5.0 pCi/L Ra-226 individually or when summed with Ra-228.

14 Quality Control

14.1 General guidelines for drinking water samples with results that exceed the Maximum Contaminant Level are specified in Pace SOP PGH-C-025, current revision, and include the following: (All steps are to be conducted as soon as the exceedence has been identified.)

14.1.1 Verify the result(s) to ensure that there were no transcription or calculation errors and that all QC results are within the acceptable limits. Correct any problems and determine the new result. If there were no errors or the result still exceeds the MCL continue with the reporting process.

14.1.2 Immediately notify the Department Manager/Supervisor, and QA department that a reportable result has been identified. Use telephone notifications to inform the contact people if the variance is identified after hours along with an e-mail follow up to document the event.

14.1.3 Refer to the State Drinking Water Emergency Reporting Requirements Binder for the state specific information regarding the proper course of action to take. Time is of the essence during this process with some of the state reporting requirements as short as 1 hour from the verification of an exceedence.

14.2 Each analyst who performs this test must satisfactorily complete a Demonstration of Capability Study as documented in Section 3.4 of the most recent revision of the Quality Assurance Manual.

14.2.1 The DOC study results are evaluated against the LCS acceptance limits.

14.2.2 IDOC and DOCs for drinking water methods must be performed at a spiking level between the sensitivity level and MCL.

14.2.3 Continuing DOCs must be performed annually by each analyst who may be expected to perform the analysis during the course of the year. A Continuing DOC may be compiled from 4 successive LCS analyses from multiple batches, and are evaluated against the LCS acceptance limits.

14.3 Daily instrument Quality Control checks must be completed following the instructions detailed in the SOP for alpha scintillation counters, current revision.

14.4 See Appendix II for performance indicator evaluation calculations and criteria. Numerical performance indicators may be used to assess QC for non-drinking water samples when the default assessment indicates a QC failure. The numerical performance indicator must be within +/- 3 for all other matrices. The z-score for precision assessment may be used for drinking waters with the approval of the Department Manager/Supervisor using the +/- 2 specification.

14.5 Method Blank (MB)

14.5.1 One MB must be prepared for each analytical batch. The purpose of the MB is to monitor for cross contamination during the analytical process. When available, the MB should be prepared from a similar matrix as samples contained in the analytical batch. If appropriate blank matrix material is not available, ASTM Type II DI water (Reagent Blank) must be carried through the procedure. A reagent blank may be used for sample correction purposes following approval of a Department Manager/Supervisor or a specified designee and affected clients.

14.5.2 The results of the method blank must be <RL. The reporting limit for Ra-226 is 1.0 pCi/L.

14.5.2.1 If the method blank is out of control, individual sample results may still be reportable if results are less than the CRDL (contract required detection limit) or > 10 times the blank result. Relative sizes of the sample and blank

aliquots must be factored when making this determination (raw counts).

14.6 Laboratory Control Sample (LCS)

14.6.1 One LCS must be prepared for each analytical batch.

14.6.2 A typical detection limit is 1.0 pCi/L for Ra-226.

14.6.3 The Ra-226 spike activity must be > 2 times the detection limit.

14.6.4 A standard reference material (SRM) containing a known concentration of radium-226 radioactivity in the same matrix as the batch is analyzed with the batch.

14.6.4.1 If this material is not available, a well-characterized material (WCM) may be used.

14.6.4.2 If neither of these are available, ASTM Type II DI water may be spiked with the appropriate radium-226 standard.

14.6.5 Percent Recovery Calculation

$$\%REC = \frac{(LCSConc)}{TrueValue} * 100$$

Where:

LCSConc = Analytical result of the LCS
TrueValue= Known concentration of the LCS

14.6.6 LCS %REC acceptance limits are 73 - 135%.

14.7 Laboratory Control Sample Duplicate (LCSD)

14.7.1 A LCSD is not required for radium-226 analysis; however analysis of an LCSD must be utilized to measure batch precision whenever adequate sample volume is not available for sample DUP analysis. The LCSD must be prepared in an identical fashion as the LCS and processed identically as for other samples.

14.7.2 The LCSD must pass the acceptance criteria for the LCS and the criteria established for duplicate precision (RPD 32% or less).

14.7.3 If the LCS and LCSD both pass %REC criteria, but fail RPD criteria, the batch results may be qualified and reported at the discretion of the analyst with guidance from the Department Manager/Supervisor.

14.8 One Duplicate Sample (DUP) must be randomly assigned within each batch. The purpose of the sample DUP is to measure precision of the analytical process. Laboratory duplicates are not intended to assess precision related to the sample collection process. Sample collection precision can only be assessed through collection of duplicate samples at

the time of sample collection. The sample DUP is a duplicate volume of sample processed identically as other samples in the analytical batch.

14.8.1 Relative Percent Difference Calculation

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

Where:

R1 = Result Sample 1
R2 = Result Sample 2

14.8.2 Duplicate sample RPD acceptance limits are <32% for radium-226.

14.8.3 Sample duplicate criteria cannot be applied if results are below their associated MDC.

14.8.4 Drinking water samples from the state of Arizona must be batched at a frequency of 1 duplicate for every 10 samples or fewer.

14.9 Sample Matrix Spikes (MS)

14.9.1 Because this analytical method requires the use of carriers or radiotracers for yield determination, PASI's default QC policy is that a sample matrix spike (MS) is not required for radium-226 analysis with the exception of drinking water analysis.

14.9.2 Typical detection limits for Ra-226 are 1 pCi/L. The spike amount must be > 10 times the detection limit.

14.9.3 The MS is prepared by spiking a portion of radium-226 radioactivity solution into a portion of one sample in the batch and processing identically as for other samples.

14.9.4 The purpose of the MS is to assess the affect of sample components on the analytical process. The volume of sample used for the MS must be equivalent to the volume used for sample analysis.

14.9.5 Percent Recovery Calculation

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for the LCS and Surrogate Calculations

14.9.6 MS acceptance limits are 71 - 136% for radium-226.

14.10 Sample Matrix Spike Duplicates (MSD)

14.10.1 A sample Matrix Spike Duplicate (MSD) is not required for this analysis. When required by the customer/contract, a MSD must

be prepared for each analytical batch. The MSD must be prepared as a duplicate of the MS.

14.10.2 The MSD must pass the acceptance criteria established for the MS recovery and the criteria established for duplicate precision.

14.10.3 An MS/MSD sample analysis may be performed instead of a sample duplicate analysis. If MS/MSD are prepared instead of a sample duplicate, and the batch includes drinking water samples from the state of Arizona, the duplicate analysis criteria for frequency in Section 14.8.4 of this SOP must be met.

14.11 PASI's default criteria for carrier and/or tracer yield are 30-110% of the expected value.

14.11.1 Barium-133 is routinely used as the tracer for Ra-226 analysis. Since Ba-133 has no potential for interference during sample counting, the amount utilized as a tracer has been optimized to ensure a minimum of 400 tracer counts is achieved in five minutes during yield analysis utilizing a sodium iodide gamma detector. This amount is approximately 6500 dpm Ba-133.

14.11.2 The amount added to each sample is consistent and added with a verified pipette, but may be changed with each new Ba-133 standard. Typical Ba-133 standards are prepared at a level between 12000 and 16000 dpm/ml depending on the concentration of the Ba-133 source purchased from a NIST traceable vendor.

14.12 Summary of QC related Activities:

Method Blank	One per Batch
Reagent Blank	One per Batch (as required by client)
Duplicate Sample	One per Batch or a frequency of 10% for batches containing samples from Arizona
Matrix Spike	One per Batch (for drinking waters or as required by client)
Matrix Spike Duplicate	One per Batch or a frequency of 10% for batches containing samples from Arizona (as required by client)
Laboratory Control Sample	One per Batch
Laboratory Control Sample Dup	One per Batch in the absence of Duplicate sample

14.13 Corrective Actions for Out-Of-Control Data

14.13.1 Method Blank (Reagent Blank) (MB/RB) – Individual samples that do not meet the acceptance criteria must be reanalyzed. If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

14.13.2 Duplicate (DUP) – DUP analysis that fails the replicate test must be reanalyzed to determine if analytical failure or sample heterogeneity was the cause of the problem.

14.13.3 Matrix Spike Recovery (MS) – MS recoveries that fail high and outside of control criteria with a sample result that is less than the reporting limit may be reported with narration. Additionally, MS recoveries that fail low and outside of control criteria for Drinking Water samples with a sample result that is greater than the MCL must be reported with comment as potentially biased high due to matrix interference. Otherwise, MS recoveries that do not meet the acceptance criteria must have that sample reanalyzed. If a Matrix Spike Duplicate is also analyzed and the recovery is comparable to the MS, the results are reported and noted in the final report. Matrix effect must be determined by re-analysis of the MS/Sample pair or demonstration of acceptable precision between a MS/MSD pair.

14.13.3.1 The analyst must evaluate the MS results to attempt to determine the cause of the failure. All decisions made must be documented.

14.13.4 Matrix Spike Duplicate (MSD) - If an MSD is analyzed and the recovery is comparable to the MS, the results are reported with qualification in the final report.

14.13.5 Laboratory Control Sample (LCS) – If an LCS analysis does not meet the acceptance criteria, the entire analytical batch must be re-prepped and reanalyzed.

14.13.5.1 The results of the batch may be reported, with qualification in the final report, if the LCS recoveries are high and the sample results within the batch are less than the reporting limit.

14.13.6 Laboratory Control Sample Duplicate (LCSD) – If an LCSD does not meet the recovery acceptance criteria, the entire analytical batch must be reanalyzed.

14.13.6.1 The results of the batch may be reported, with qualification, if the LCS recoveries are high and the sample results within the batch are less than their the reporting limit, and duplicate precision meets the acceptance criteria.

14.13.7 If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report. Chemical/Tracer recoveries: If the chemical and/or tracer recovery is outside of the acceptance criteria, the sample must be reanalyzed. If a matrix interference is suspected to be the cause, sample reanalysis should be performed with a lower volume to minimize interferences.

14.13.7.1 Acceptance criteria for gravimetric carriers are 30-110% and for radioactive tracers the criteria are 10-110%. Yields as high as 130% may be reported with the approval of the Department Manager/Supervisor.

15 Method Performance

15.1 Laboratory control samples are analyzed with each batch, the results are charted to monitor control limits and trending. Each analyst must read and understand this procedure with written documentation maintained in their training file on the Learning Management System (LMS).

15.2 An initial demonstration of capability (IDOC) study must be performed. A record of the IDOC will be maintained on file in each analysts training file in the LMS.

15.3 On an annual basis, each analyst will complete a continuing demonstration of capability (CDOC).

16 Pollution Prevention and Waste Management

16.1 Place radioactive waste into appropriate receptacles.

16.2 Discard acidified samples and unusable standards into proper waste drains.

16.3 Dispose of waste materials in accordance to type: Non-hazardous, hazardous, non-radioactive, radioactive or mixed.

17 References

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17.13 Table of Radioactive Isotopes, Brown and Firestone, Shirley editor, John Wiley & Sons, 1986.

17.14 Youden, W. J. "Statistical Techniques for Collaborative Tests." Statistical Manual of the AOAC Association of Official Analytical Chemists, Washington, D. C. 1975.

17.15 "American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories", ANSI N42.23-1996.

17.16 Department of Defense Quality System Manual for Environmental Laboratories (DoD QSM), current version.

17.17 "Manual for the Certification of Laboratories Analyzing Drinking Water" Fifth Edition, January 2005, EPA 815-R-05-004.

17.18 National Primary Interim Drinking Water Regulations (NIPDWR), Part 141.15.

17.19 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (most recently approved revision).

17.20 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.

17.21 Pace Analytical Services, LLC. - Pittsburgh Laboratory Quality Assurance Manual, current version.

17.22 Pace SOP PGH-R-003, current revision (Analysis of Water Samples for Ra-228).

17.23 Pace SOP PGH-R-065, current revision (Alpha Scintillation Counter Operations).

17.24 Pace SOP PGH-C-025, current revision (MCL Violation Reporting).

17.25 Pace SOP PGH-C-027, current revision (Deionized Water Quality and Suitability).

17.26 SOP PGH-C-032, Support Equipment, current version.

17.27 SOP PGH-Q-038, Laboratory Equipment, current version.

17.28 SOP PGH-Q-040, Internal and External Audits, current version

17.29 SOP PGH-Q-039, Corrective and Preventative Action, current version.

17.30 SOP S-ALL-Q-020, Training, current version.

17.31 SOP S-ALL-Q-028, Lab Track, current version.

18 Tables, Diagrams, Flowcharts, Appendices, etc.

18.1 Attachment I: Calculations

18.2 Attachment II: Numerical Performance Indicators

19 Method Modifications

19.1 The strict co-precipitation technique specified in EPA 903.1, “Radium-226 in Drinking Water” generates a sulfate precipitate that is not easily dissolved when following the detailed methodology. The precipitation technique detailed in this SOP generates a soluble precipitate that is easily dissolved in EDTA solution. This minimizes the potential for reporting erroneous carrier yields and limits the spread of insoluble contamination in the bubbler system.

19.2 The barium/radium sulfate co-precipitation technique utilized in this SOP has been enhanced to allow for quality improvements in analysis and production efficiency. Excluding the addition of strontium carrier, the co-precipitation technique utilized in steps 12.1 through 12.7 of this SOP mirrors the precipitation technique employed in Pace SOP PGH-R-003, current revision, Analysis of Water Samples for Ra-228. Excluding the modifications specifically cited in this SOP, modifications between Pace SOP PGH-R-003, current revision, and EPA 904.0, “Radium-228 in Drinking Water” are documented in Section 19 of Pace SOP PGH-R-003, current revision.

19.3 The addition of barium carrier (16 mg/mL) and yttrium carrier (18 mg/mL) at step 12.5 of this SOP are added in reverse order that is cited in Pace SOP PGH-R-003, current revision, and EPA 904.0, “Radium-228 in Drinking Water.”

19.4 EPA Method 903.1 specifies the dissolution of the barium/radium co-precipitate generated in this procedure using 20 mL of 0.25 M EDTA solution. For some samples, it is necessary to use additional EDTA

solution to dissolve the barium sulfate precipitate. EPA method 904.0 for Ra-228 analysis requires an initial addition of 25 mL of EDTA solution to dissolve the barium/radium co-precipitate. In order to maintain consistency on a sample-level basis, Pace uses 25 mL of EDTA for precipitate dissolution. Adhering to the strict solution volume prior to sample analysis ensures consistency between sample analysis volumes and calibration solution volumes, thereby ensuring consistent exchange of radon and nitrogen gas when emanating radon gas from the sample bubblers.

19.5 When using bubblers of the exact dimension specifications described in EPA 903.1, there is a high probability of loss of sample solution contained in the bubblers when emanating radon from the bubbler. This can lead to contamination of the manifold system, including the Lucas cell used for analysis. Pace utilizes a bubbler design of larger dimensions so as to limit the negative impact of sample overflow. Pace ensures the appropriate ratio of radon to nitrogen gas exchange by using equivalent bubbler solution volumes between samples and calibration sources.

19.6 The preparation of the yttrium carrier solution documented in step 10.20 of this SOP does not explicitly follow the specifications of EPA Method 904.0. Additional concentrated nitric acid and ASTM Type II DI water are added at the onset of yttrium oxide dissolution so as to enhance the dissolution. The final ratio of yttrium to nitric acid solution for the yttrium carrier prepared within this SOP matches the ratios specified in EPA 904.0.

19.7 Counting the samples (and calibration cells) after only 3 hours from de-emanation instead of 4 hours as specified in EPA 903.1 allows for increased productivity and results in an error of < 0.47% for high activity sample and is negligible for low activity samples.

19.8 Concentration of radium by co-precipitation with barium is quantitative, however, collection of the precipitate is highly variable and is relative to individual analyst technique. Since there is inherent room for losses, any assumption that 100% of the sample makes it from initial precipitation, dissolution, and into the bubbler for analysis, biases the sample results low. Although the use of a tracer (Ba-133) is not specified in EPA 903.1, Pace's barium recovery measurement technique was determined by accrediting authorities to improve method precision and accuracy and was thereby allowed.

19.9 The use of anti-foaming agent in the bubbler to prevent samples from foaming and resulting in sample loss is not specified in EPA 903.1. The use and addition does not adversely impact the Ra-226 results.

19.10 Due to a world-wide shortage of helium gas, nitrogen gas is used as an alternate gas for the specified emanation process.

19.11 EPA Method 903.1 specifies to use 1,000 mL of sample for analysis. Pace analyzes approximately 500 mL of sample for analysis. All samples are measured by mass as specified within this SOP. Pace further

restricts the analysis volume for samples known to contain matrix components that would limit analyte recovery. Pace restricts the analysis volume so as to limit the quantity of waste generated and to enhance productivity. The required detection limit of 1 pCi/L is achieved using a 500 gram analysis quantity.

19.12 For the preparation of 0.25M EDTA, EPA Method 903.1 specifies to add sodium hydroxide to DI water then heat prior to adding the solid EDTA for dissolution. The heat generated from the dissolution of sodium hydroxide in water is sufficient to dissolve the EDTA and so, hot-plate heating is not performed.

20 Revisions

Document Number	Reason for Change	Date
PGH-R-007-11	1. Table of contents added. 2. Modified Section 11.6.1 to require minimum ingrowth time for calibration cells. 3. Modified Section 12.10 to require minimum ingrowth time for samples. 4. Clarified step 12.17 to document when gravimetric recovery for barium yield is allowed. 5. Section 15 modified to “Method Performance.” “Pollution Prevention and Waste Management” Section moved to Section 16. 6. “References” Section moved to Section 17. 7. “Tables, Diagrams, Flowcharts, Appendices, etc.” Section moved to Section 18. 8. “Deviations from Promulgated Methods” changed to Method Modifications Section moved to Section 19. 9. Revised Equation 7 to document the recovery calculation for Ba yield assessment. 10. Section 14: Added 14.7.2.1 to discuss yield criteria. Also modified Section to discuss non-conformance tracking with LabTracks and evaluating usefulness of data and qualifying or narrating to allow for when clients do not want a final report. 11. Section 17: Added references, ANSI N42, 23, TNI Standard, and DoD QSM. 12. Defined variables into Calculations Section for Ba recovery calculation. 13. Definition of UE4 in the Calculations Uncertainty Section was changed to require approval of the Department Supervisor for UE4. 14. Inserted Calculation number 15, the critical level calculation.	4/19/2012
PGH-R-007-12	1. Updated Table of Contents to include Attachments I and II. 2. Section 3.1, 10.12 – added nitrogen gas as optional to helium, and also included in parenthesis wherever helium is	26June2013

Document Number	Reason for Change	Date
	mentioned. 3. Section 6.5 and 12.1 – Included comment about recording observed masses and not targeting sample aliquots. 4. Section 10.2 – Added Type II DI SOP reference. 5. Section 11– defined calibration constant acceptance criteria and optimal cell constant range, process for recalibrating, assigning Ids, and repair. 6. Section 12.2 – added comment about how to proceed if methyl orange pH indicator is not red in sample. 7. Section 12 – clarified where each piece is attached when performing the radon emanation into the Lucas cell. 8. Section 12.20 – added instructions on counting cells and reference instrument operation SOP. 9. Section 13.2 and 14.1 – added to provide instruction for recognizing and reporting DW MCL exceedances. Pace SOP PGH-C-025 referenced. 10. Section 14.4 – added comment regarding using numerical indicators to assess QC and applicability. 11. Section 14.8.4 and 14.10.3 – added to include duplicate sample analysis requirement of 10% for Arizona, and 5% for all else. 12. Entire document – changed all references of DI water to ASTM Type II DI water to conform to method verbiage.	
PGH-R-007-13	1. Annual SOP review. 2. Section 4.2 and 4.3 – Added comments regarding preventing sources of cross contamination during emanation and in bubblers due to solids, 3. Section 6.6 – Added comment regarding documenting the use of pipette, graduated cylinders, or balances for sample measuring. 4. Section 8.2 – Inserted requirements for pH verification and documentation. 5. Section 11 – Clarified calibration acceptance criteria. 6. Section 12.1 to 12.3 – Inserted instructions for diluting samples, preparing QC samples, and ensuring QC are spiked prior to the addition of all other chemicals. 7. Section 14 – Added DOC and CDOC requirements. 8. Section 14 – Added MSD frequency for AZ DW samples. 9. Section 14.13.2 – Updated to discuss failed LCS/LCSD RPD actions and qualification. 10. Section 17 – Added DW Manual reference and NIPDWR reference. 11. Section 19.2 – Updated to point to the concurrent steps in the Ra-228 SOP PGH-R-003, instead of the EPA 904.0 method. 12. Section 19 – Added use of anti-foam agent and nitrogen gas into deviation from methods. 13. Edited for spelling and grammatical errors. 14. Reformatted document.	13Jul2014

Document Number	Reason for Change	Date
PGH-R-007-14	1. Removed from section 5.1: Analysts must be trained as radiation workers and personal dosimeter worn. 2. Section 12.17 - Clarified the observed changes in vacuum indicated on the gauge during sample emanation. 3. Section 14.11 – Added typical Ba-133 tracer concentrations and the determination of the tracer levels.	20Feb2015
PGH-R-007-15	1. Section 10.20-Updated the preparation of yttrium carrier (18 mg/mL) in order to match the analytical process and to more closely match the method process. 2. Sections 12.9 and 12.10-Modified the volume of 0.25M EDTA solution used in the process to match the volume used for process calibration. This standardization of the volume of EDTA used, ensures consistency between samples and the calibration process. 3. Updated section 19, Method Modifications to address changes to this SOP revision. Added a modification specifying differences between the bubbler used and the bubbler specifications in the method, EPA 903.1.	15July2015
PGH-R-007-16	1. Section 12.20 corrected to specify the correct reference section for Lucas Cell Counting. 2. Section 19 updated to include the analysis volume difference between this SOP and the method, EPA 903.1.	14Dec2015
PGH-R-007-17	1. Updated Inc. to LLC. 2. Updated PGH-R-064 to PGH-R-065. 3. Updated section 19.2 to add: excluding the addition of strontium carrier. 4. Added section 19.3 to add: The addition of barium carrier (16 mg/mL) and yttrium carrier (18 mg/mL) at step 12.5 of this SOP are added in reverse order that is cited in Pace SOP PGH-R-003, current revision, and EPA 904.0, “Radium-228 in Drinking Water.”	21Dec2016
S-PGH-R-007-rev.18	1. Section 8.2 samples must be held minimum of 24 hours. 2. Modified section 10.10 to remove the requirement to heat EDTA solution during preparation. Heating is not necessary to prepare the solution. 3. Modified section 10.19 to document purchase and use of pre-diluted 18 N sulfuric acid from the manufacturer. 4. Modified section 12.14 to specify filling of absorption tube with half ascarite and half magnesium perchlorate to enhance removal of moisture known to be present in the samples. 5. Modified section 12.21 to remove the requirement to rinse bubblers into the tracer-recovery phase. 6. Modified sections 12.17 and 12.19 to remove the requirement to close the bubbler stopcock when bubbling ceases. The emanation system, including the bubbler is designed to prevent leakage. Closing the bubbler stopcock creates system structure pressure that could create loss of analyte. 7. Section 12.22.9 modified to specify use of inert helium or	08Feb2018

Document Number	Reason for Change	Date
	nitrogen gas for cell flushing, rather than room air. 8. Sections 14.13.3 and 14.13.4 modified to clarify the corrective actions for failed sample matrix spikes and/or sample matrix spike duplicates.	

Attachment I
Calculations

The **radium-226 concentration** of a sample is calculated according to the following equations:

Eq. 1 $$Act = \frac{(S_A - B_A)}{(Denom)}$$

Eq. 2 $$Denom = E * V * 2.22 * I_S * D_1 * D_2 * R$$

Eq. 3 $$E = \frac{(S_C - B_C)}{(A_C * I_C * D_{1C} * D_{2C})}$$

Eq. 4 $$I_S = 1 - e^{-\lambda t1}$$

Eq. 5 $$D_1 = e^{-\lambda t2}$$

Eq. 6 $$D_2 = (1-e^{-\lambda t3})/(\lambda t_3)$$

Eq. 7 $$R = \frac{M_B}{T_B} \text{ OR } R = \frac{M_{Ba133}}{T_{Ba133}}$$

Eq. 8 $$I_C = 1 - e^{-\lambda t4}$$

Eq. 9 $$D_{1C} = e^{-\lambda t5}$$

Eq. 10 $$D_{2C} = e^{-\lambda t6}$$

Where:

Act	=	Radium-226 sample concentration in pCi/unit (L, g, F, etc.)
S_A	=	gross count rate for the sample (in cpm)
B_A	=	cell background (in cpm)
S_C	=	gross count rate for the calibration source (in cpm)
B_C	=	calibration source cell background (in cpm)
A_C	=	activity of the calibration source in dpm
2.22	=	conversion factor from dpm to pCi
E	=	cell efficiency in cpm/dpm of radon-222
V	=	sample volume, mass, or fraction (in L, g, or %filter, etc)
R	=	fractional recovery of barium sulfate or Ba-133 tracer
M_B	=	mass of barium sulfate recovered (in mg)
M_{Ba133}	=	Measured sample Ba-133 Net Cts from the sodium iodide counter yield measurement
T_B	=	standardized barium carrier conc. (in mg barium sulfate per mL of barium carrier used)
T_{Ba133}	=	Measured Net Cts of the reference source for the specific sodium iodide detector used for the sample recovery determination
I_S	=	Radon-222 ingrowth factor for the sample

D_1	=	Radon-222 decay between second de-emanantion and counting for the sample
D_2	=	Radon-222 decay during counting for the sample
I_C	=	Radon-222 ingrowth factor for the calibration source
D_{1C}	=	Radon-222 decay between second de-emanantion and counting for the calibration sample
D_{2C}	=	Radon-222 decay during counting for the calibration source
λ	=	Decay constant of radon-222, (0.000125883 min^{-1})
t_1	=	the elapsed time in minutes between the first and second de-emanations for the sample
t_2	=	the elapsed time in minutes between the second de-emanation and sample counting for the sample
t_3	=	the sample counting time in minutes
t_4	=	the elapsed time in minutes between the first and second de-emanations for the calibration source in hours
t_5	=	the elapsed time in minutes between the second de-emanation and calibration source counting in hours
t_6	=	the calibration source counting time in minutes
Measured	=	either barium sulfate mass recovered in mg or Ba-133 measured by gamma spec
Expected	=	standardized barium sulfate target (in mg) or Ba-133 reference value (in pCi/L)

The sample specific **counting uncertainty** is calculated as follows.

Eq. 11 $$Counting\ Uncertainty = \frac{1.96 * \sqrt{((S_A/t_3)) + ((B_A/t_7)}}{Denom}$$

Where:

t_7 = background count time in minutes

As summed background and analyte count rates approach zero, assumptions underlying the uncertainty calculation are violated and it will return an unrealistic value of zero (0) uncertainty when zero summed counts are observed. The following equation provides a more accurate estimate of count uncertainty at zero and near-zero count rates.

Eq. 12 $$ZeroUnc = ZeroActFact / SmplTime$$

Note 1: Depending on sample type and contract requirements the zero activity factor may be either 3.0 or 2.71. PASI's default is 2.71 consistent with the current version of ANSI N42.23. Bioassay samples must be calculated using 3.0 to be consistent with ANSI N13.30

Note 2: The Zero Count Uncertainty is compared to the count uncertainty above. The larger of the two is used as the counting uncertainty in subsequent total error calculations.

The error term is further evaluated to provide an estimate of total error hereafter referred to as the *Combined Standard Uncertainty* (CSU a.k.a. TPU).

Eq.13

$$\text{CSU (pCi/unit)} = \sqrt{(\text{CountingUncertainty})^2 + (UE1 * Act)^2 + (UE2 * Act)^2 + (UE3 * Act)^2 + (UE4 * Act)^2}$$

UE1, UE2, UE3, and UE4 represent partial derivatives estimating the relative uncertainty at the ***95% confidence interval*** for various factors in the activity calculation as follows:

UE1 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with preparation (e.g., sample aliquot or transfers and splits prior to addition and equilibration of tracer).

UE2 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with analysis (e.g., peak integration, peak overlap, tracer contaminants).

UE3 represents combined factors estimating relative uncertainty (fractional) associated with yield correction (e.g., count uncertainty for tracer peak, SRM known value, tracer volume or mass aliquot, tracer equilibration efficiency).

UE4 represents the factor estimating additional uncertainty (activity) associated with an individual sample -- to be used in exceptional circumstances with approval of The Department Supervisor and appropriate documentation and narration only.

The Minimum Detectable Concentration (MDC) is calculated per guidance of ANSI N42.23 and N13.30 as:

Eq. 14

$$\text{MDC} = \frac{4.65 * \sqrt{(B_A) * t_3} + ZeroActFact}{t_3 * \text{Denom}}$$

Where B_A, t_3, *ZeroActFact*, and Denom have previously been identified.

The critical level (Lc) is calculated per guidance of ANSI N42.23 as:

Eq. 15

$$Lc = \frac{1.65 * \sqrt{(B) * (1/Ts + 1/Tb)}}{Denom}$$

Where:

B, T_s, T_b, *ZeroActFact*, and Denom are as previously defined.

Attachment II
(Numerical Performance Indicators)

1. **Method Blank (MB)**

1.1 The numerical performance indicator for the method blank is calculated by:

$$Z_{Blank} = \frac{x}{u(x)}$$

Where:
x = measured blank activity
u(x) = standard uncertainty (1 sigma) in the blank measurement

1.2 MB performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. MB performance indicator values should be recorded on a control chart.

2. **Laboratory Control Sample (LCS)**

2.1 The numerical performance indicator for a laboratory control sample is calculated by:

$$Z_{LCS} = \frac{x - c}{\sqrt{u^2(x) + u^2(c)}}$$

Where:

x = Analytical result of the LCS
c = Known concentration of the LCS
$u^2(x)$ = combined standard uncertainty (1 sigma) of the result squared.
$u^2(c)$ = combined standard uncertainty (1 sigma) of the LCS value squared.

2.2 LCS performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. Performance indicator values should be recorded on a control chart.

3. Duplicates (DUP)

3.1 These criteria are applicable for the evaluation of the Duplicate, Matrix Spike Duplicate and Laboratory Control Sample Duplicates.

3.2 The numerical performance indicator for laboratory duplicates is calculated by:

$$Z_{Dup} = \frac{x_1 - x_2}{\sqrt{u^2(x_1) + u^2(x_2)}}$$

Where:
x_1, x_2 = two measured activity concentrations

$u^2(x_1)$, $u^2(x_2)$ = the combined standard uncertainty (1 sigma) of each measurement squared.

3.3 Duplicate sample performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. DUP performance indicator values should be recorded on a control chart for each QC sample type (Dup, MSD, LCSD)

4. **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

4.1 The numerical performance indicator for a matrix spike sample is calculated by:

$$Z_{MS} = \frac{x - x_0 - c}{\sqrt{u^2(x) + u^2(x_0) + u^2(c)}}$$

Where:
x = measured concentration of the spiked sample
x_0 = measured concentration of the unspiked sample
c = spike concentration added
$u^2(x)$, $u^2(x_0)$, $u^2(c)$ = the squares of the respective combined standard uncertainties (1 sigma) of these values.

4.2 MS performance for all matrices is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. MS performance indicator values should be recorded on a control chart.

ATTACHMENT C-25

ANALYSIS OF WATER SAMPLES FOR RA-228 CONTENT-904.0
PACE, PITTSBURGH

Document Information

Document Number: ENV-SOP-GBUR-0064 **Revision:** 00

Document Title: Analysis of Water Samples for Ra-228 Content - 904.0

Department(s): Rad Chem

Previous Document Number: S-PGH-R-003-rev.19

Date Information

Effective Date: 08 Feb 2018

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Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

STANDARD OPERATING PROCEDURE

Analysis of Water Samples for Ra-228

Methods: EPA 904.0 and 9320/SM7500-RaD (Ra-228)

SOP NUMBER:	S-PGH-R-003-rev.19
REVIEW:	R. Kinney
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	PGH-R-003-18
REVIEW DATE:	Upon Procedural Change

APPROVALS

Maureen K. DeKubeis
Senior Quality Manager — 02/08/18 Date

Department Manager/Supervisor — 02/08/18 Date

PERIODIC REVIEW
SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date

TABLE OF CONTENTS

1. Purpose

1.1 This SOP documents the analytical procedure to be used for the analysis of drinking water and other aqueous samples for Ra-228 content. This SOP is based on EPA 904.0, 9320, and SM7500-RaD.

2. Scope and Application

2.1 This procedure is applicable for the analysis of radium-228 in drinking water, wastewater, and other aqueous matrices. Without qualification, this procedure, as written, is compliant with Method 904.0 of "*Prescribed Procedures for Measurement of Radioactivity in Drinking Water,* EPA-600/4-80-032", Method 9320 of "*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (SW846), Volume 1C, Third Edition", and Standard Method 7500-Ra D of "*Standard Methods for the Examination of Water and Wastewater*".

2.2 Radium-228 in an aqueous sample is determined by the isolation and direct measurement of Ac-228, the beta-emitting daughter of Ra-228, utilizing a gas flow proportional counter.

2.3 The efficiency of the Gas flow proportional counting (GFPC) system used for radioactivity measurement fluctuates relative to the energy of radioactivity emissions. Actinium-228 has a half life of 6.3 hours, so it is not practical to perform system calibrations directly with Ac-228, therefore, a beta isotope of similar energy is utilized for calibration purposes. This SOP requires that Sr-89 be utilized for calibration purposes.

2.4 Pace Analytical services, LLC. (PASI) applies isotope decay correction only in instances where the total impact in the analysis result is 2% or greater. Assuming a maximum hold time of 180 days, a 2% isotope decay would occur only for radioisotopes with a half-life of 17.14 years or less. The parameters reported using this SOP are affected by this policy and results for Ra-228 are decay corrected for the time from the collection date and time supplied by the client to the start of instrument analysis.

3. Summary of Method

3.1 Radium in aqueous samples is pre-concentrated by co-precipitation with barium and lead as a sulfate. Barium and radium are isolated from lead by repeated precipitation as a sulfate from EDTA solution. The final purified barium/radium sulfate precipitate is dissolved in EDTA solution and stored to allow ingrowth of Ac-228, the beta-emitting daughter of Ra-228.

3.2 Following the ingrowth of Ac-228, potentially interfering radioisotopes of lead that may be present are removed by precipitation as lead sulfide. Actinium-228 in the sample is separated by co-precipitation with yttrium as hydroxide, then converted to yttrium oxalate and mounted for beta counting by GFPC. Radium recovery is determined by gravimetric measurement of barium sulfate precipitate recovered during analysis, or by use of Ba-133 as a radiotracer. Actinium recovery is determined by gravimetric measurement of yttrium oxalate precipitate recovered during analysis.

4. Interferences

4.1 The presence of high levels of Sr-90 in a water sample will produce a positive bias to the Ra-228 activity measured.

4.2 Elemental barium contained in the water sample will result in a falsely high gravimetrically measured barium yield and will result in an underestimation of Ra-

228 content. This interference will not affect the yttrium or Ba-133 results. Ba-133 radiotracer is utilized to eliminate possible chemical recovery biases which would occur if stable barium was present in the sample and yield determination was performed gravimetrically.

4.3 In some samples, unidentified sample interferences can cause Ba-133 to carryover to the final source planchet, elevating the sample beta count rate, and biasing the sample result high. Additional instructions for determining if Ba-133 carryover has occurred are outlined in Section 12.5 of this SOP.

4.4 Samples containing excessive sodium and calcium will cause excess sulfate precipitate to form during the initial sulfate precipitation steps. Excessive sulfate precipitates can cause low chemical yields, because the additional precipitate may not completely dissolve in EDTA during the initial ingrowthing steps. Additional steps in Section 12.7 are designed to minimize the interference of sodium and calcium sulfate precipitates.

5. Safety

5.1 Procedures must be carried out in a manner that protects the health and safety of all personnel.

5.2 At a minimum, eye protection, a laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves will be cleaned immediately.

5.3 When mixing or diluting acids, always add the acid slowly to water and swirl. Dilution of acids must always be done in a hood. Appropriate eye-protection, gloves, and lab coat must be worn.

5.4 Exposure to radioactivity and chemicals must be maintained as low as reasonably achievable. Samples known to be hazardous and/or radioactive, must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 In order to minimize the potential for cross contamination of high and low levels of radioactive samples, good housekeeping and good laboratory practices are essential and must be strictly adhered to.

5.6 Organic samples of unknown content must be handled with extreme caution and under the direct instruction of a department manager or manager-specified designee. Direct treatment of organic matrices with strong oxidizing chemicals such as nitric acid and/or hydrogen peroxide is strictly prohibited.

5.7 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the SDS files maintained in the laboratory and in the PASI-PGH Chemical Hygiene Plan.

6. Definitions

6.1 See the Glossary Section of the most recent version of the Pace Analytical Services, LLC. Quality Assurance Manual for commonly used laboratory terms.

6.2 Batch: For all matrices, an analytical batch contains 20 or fewer samples of a similar matrix, prepared at the same time, by the same analyst, using the same reagents.

6.3 Throughout this procedure, approximate weights and measures will be designated by the use of whole numbers when referring to masses exceeding 1gor volumes in excess of1mL. Measurements of masses and volumes so designated can be made with top loading balances, graduated cylinders, etc. For approximate measures below 1gor1mL, the word "approximately" must be used prior to the described mass or volume.

6.4 Throughout this procedure, exact or critical mass and volumes will be designated by the use of one or more decimal places. Measurements of mass and volumes so designated should be made with accurate analytical instruments such as analytical balances, calibrated pipettes, etc.

6.5 Any reference to a “hot water bath” indicates a container filled with water, which has been heated to a temperature just below the boiling point of the water.

6.6 When aliquotting samples on a balance, the observed mass on the balance must be recorded in preparation logbooks to the lowest mass indicated on the balance. Sample aliquot masses must not be targeted. Once sample is removed from the sample container and transferred to a beaker, it must not be removed from the beaker.

6.7 The method utilized for obtaining the sample aliquot, whether on a balance, in a graduated cylinder, or by pipette, must be clearly annotated in the preparation logbook.

7. Responsibilities and Distribution

7.1 General Manager/Assistant General Manager (GM/AGM)

7.1.1 The GM/AGM has the overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the laboratory involving the collection and reporting of analytical data.

7.1.2 The GM/AGM and Senior Quality Manager/Quality Manager have final review and approval authority for all SOPs prepared within the laboratory.

7.2 Senior Quality Manager/Quality Manager (SQM/QM)

7.2.1 The SQM/QM will maintain a master file of all SOPs applicable to the operations departments.

7.2.2 The SQM/QM will assign a unique number to each SOP prepared prior to approval and distribution.

7.2.3 The SQM/QM will distribute SOPs to applicable personnel and maintain an accurate accounting of such distribution to ensure that the SOPs, in the hands of the users, are current and complete.

7.3 Department Manager/Supervisor

7.3.1 The Department Manager/Supervisor is responsible for ensuring all staff members read, follow, and are adequately trained in the use of the SOPs

7.3.2 The Department Manager/Supervisor coordinates the preparation and revision of all SOPs within the department whenever a procedure changes.

7.3.3 The Department Manager/Supervisor provides initial approval of all SOPs within the department.

7.3.4 The Department Manager/Supervisor makes recommendations for SOP revision to the SQM/QM via written memo.

7.4 Individual Staff

7.4.1 Individual staff members are responsible for adherence to the specific policies and procedures contained in the applicable SOPs.

7.4.2 Individual staff members will only use a signed, controlled copy of the SOP. Each person may make recommendations to the Department Manager/Supervisor for revising SOPs as the need arises.

7.4.3 Personnel are responsible for ensuring that any deviations from this SOP are reported to the Department Manager/Supervisor.

8. Sample Collection, Preservation, and Handling

8.1 Aqueous samples

8.1.1 Containers used for sample collection must never be re-used. Either plastic or glass containers may be used for sample collection.

8.1.2 Aqueous samples must be preserved at the time of collection by adding enough concentrated (16N) HNO_3 to the sample to make the sample pH <2. Typically, two mL of 16N HNO_3 per liter of sample is sufficient to obtain the desired pH. Samples must be preserved within five days of collection. If samples are collected without preservation, they must be received by the laboratory and preserved within five days of collection. Following preservation with acid, samples must be held in the original container for a minimum of 24 hours, and the pH must be rechecked by laboratory personnel prior to removing sample for analysis. The pH recheck date and time, the initials of the analyst verifying the pH, as well as any adjustments or notes regarding the preservation must be recorded in the pH Verification Logbook.

8.1.2.1 For dissolved analysis, samples must be filtered through a 0.45μm membrane filter and preserved to a pH <2.

8.1.2.2 For total analysis, the sample is not filtered, but is preserved.

8.1.3 Refrigeration is not required for aqueous samples.

8.1.4 The maximum hold time for samples analyzed by this procedure is 180 days between sample collection and sample analysis.

9. Equipment and Supplies

9.1 Gas Flow Proportional Counting System. (Low background beta <3 cpm). Refer to SOP PGH-R-002, current revision “Gas Flow Proportional Counter Operation” for instructions on GFPC system operation.

9.2 Software supplied with the instrument to control instrument operation. Refer to SOP PGH-R-002, current revision “Gas Flow Proportional Counter Operation” for applicable software details.

9.3 Computer capable of running the Gas Flow Proportional Counter System software, monitor, mouse, keyboard, and printer. Refer to SOP PGH-R-002, current revision “Gas Flow Proportional Counter Operation” for computer hardware specifications.

9.4 Electric hot plate.

9.5 Centrifuge, capable of greater than 2500 rpm.

9.6 Centrifuge tubes, 50 mL high density polyethylene, or equivalent.

9.7 Filter paper, Fisherbrand Q2, 11.0cm diameter, or equivalent.

9.8 Membrane filter, 0.45μm, 47mm, Metricel®, or equivalent.

9.9 Drying lamp.

9.10 Glassware, various sizes.

9.11 Stirring Rods, glass.

9.12 Stainless steel counting planchets. 2 inch diameter, 1/8 inch deep.

9.13 Analytical balance (capable of measuring to 0.0001g).

9.14 Top Loading balance (capable of measuring to 0.01g).

9.15 Vortex mixer.

10. Reagents and Standards

10.1 Reagents should be prepared from reagent grade chemicals, unless otherwise specified below. Reagent water must be at least ASTM Type II quality or better. NOTE: Consult the Safety Data Sheets for the properties of these reagents, and how to work with them.

10.2 Distilled or deionized (DI)water. ASTM Type II generated as specified in Pace SOP PGH-C-027, current revision.

10.3 Acetic acid, 17.4N: glacial CH_3COOH (conc.), sp. Gr. 1.05, 99.8%.

10.4 Ammonium hydroxide, 15N: NH_4OH (conc.), sp. Gr. 0.90, 56.6%.

10.5 Ammonium oxalate, 5%: Dissolve 50.0g $(NH_4)_2C_2O_4 \cdot H_2O$ in boiling ASTM Type II DI water, cool, and dilute to 1.0 L with ASTM Type II DI water.

10.6 Ammonium sulfate, 200mg/mL: Dissolve 200g $(NH_4)_2SO_4$ in water and dilute to 1.0L with ASTM Type II DI water.

10.7 Ammonium sulfide, 2%: Dilute 2.0mL $(NH_4)_2S$, (20-24%), to 20mL with ASTM Type II DI water.

10.8 Barium carrier, 16mg/mL, standardized. Dissolve 28.46 g $BaCl_2 \cdot 2H_2O$ in water, add 5.0mL 16N HNO_3, and dilute to 1.0L with ASTM Type II DI water.

10.9 Barium-133 standard solution, NIST traceable for use as a radiotracer for yield monitoring.

10.10 Citric acid, 1M: Dissolve 192.0g $C_6H_8O_7 \cdot H_2O$ in water and dilute to 1.0L with ASTM Type II DI water.

10.11 EDTA reagent, basic, (0.25M): Dissolve 20 g NaOH in 750mL water and slowly add 93g disodium ethylenedinitriloacetate dihydrate, $(Na_2C_{10}H_{14}O_8N_2 \bullet 2H_2O)$ while stirring. After the salt is in solution, dilute to 1L with ASTM Type II DI Water. The heat generated from the solid sodium hydroxide added to DI water should be sufficient so as to support complete dissolution of EDTA. If the EDTA does not readily dissolve, gradually heat the reagent until dissolved.

10.12 Lead carrier, 150mg/mL: Dissolve 239.7 g $Pb(NO_3)_2$ in ASTM Type II DI water, add 5.0mL 16N HNO_3 and dilute to 1.0 L with ASTM Type II DI water.

10.13 Lead carrier, 1.5 mg/mL: Dilute 10mL lead carrier (150 mg/mL) to 1.0L with ASTM Type II DI water.

10.14 Methyl Orange Indicator, 0.1%: Dissolve 1.0g methyl orange indicator in 1.0L ASTM Type II DI water.

10.15 Nitric acid, 16N: HNO_3 (conc.), sp. gr. 1.42, 70.4%.

10.16 Nitric acid, 6N: Add 375mL of 16N HNO_3 to 500mL of deionized water, cool and dilute to 1Liter with ASTM Type II DI water.

10.17 Nitric acid, 1N: Dilute 62.5mL 16N HNO_3 to 1Liter with deionized water.

10.18 Radium-228 standard solution, NIST traceable for use as a Laboratory Control Sample spiking material.

10.19 Sodium carbonate, 2N: dissolve 124 g $Na_2CO_3 \cdot H_2O$ (or 106 g Na_2CO_3) in ASTM Type II DI water and dilute to 1Liter with ASTM Type II DI water.

10.20 Sodium hydroxide, 18M: Dissolve 720g NaOH in ASTM Type II DI water and dilute to 1.0L with ASTM Type II DI water.

10.21 Sodium hydroxide, 10M: Dissolve 400g NaOH in ASTM Type II DI water and dilute to 1.0L with ASTM Type II DI water.

10.22 Strontium carrier, 10mg/mL: Dissolve 24.16 g $Sr(NO_3)_2$ in ASTM Type II DI water and dilute to 1.0L with ASTM Type II DI water.

10.23 Strontium-89 standard solution, NIST traceable for use in instrument calibration.

10.24 Sulfuric acid, 18N: Cautiously mix 1 volume 36N H_2SO_4 (conc.) with 1 volume of ASTM Type II DI water in a tub of ice or cool water. As water evaporates replace with ASTM Type II DI water to desired final volume.

10.25 Sulfuric acid, 0.1N: Dilute 5.56 mL of 18N sulfuric acid to 1.0L using ASTM Type II DI water.

10.26 Yttrium carrier, 18 mg/mL: Add 22.85 g Y_2O_3 to an Erlenmeyer flask Add 50mL of ASTM Type II DI water. Carefully add 40mL of concentrated nitric acid. Heat the mixture to boiling while stirring on a magnetic stirring hotplate. The solution must heat to boiling. Additional water and nitric acid may be added as necessary to aid in dissolution. Scrape the bottom of the Erlenmeyer flask with a Teflon scraper if the yttrium oxide is caked onto the glass. Do not add more than 100mL of nitric acid total. Upon complete dissolution, remove the beaker from the hotplate and cool completely. Dilute to 1Liter with ASTM Type II DI water.

10.27 Yttrium carrier, 9 mg/mL: Dilute 500mL yttrium carrier (18 mg/mL) to 1.0L with ASTM Type II DI water. Standardize this carrier in accordance with the steps outlined in section 11.

10.28 Strontium-yttrium mixed carrier, 0.9 mg/mL Sr^{+2} – 0.9 mg/mL Y^{+3}:

10.28.1 Solution A: Dilute 100mL yttrium carrier (18 mg/mL) to 1.0L with ASTM Type II DI water.

10.28.2 Solution B: Dissolve 4.348 g $Sr(NO_3)_2$ in ASTM Type II DI water and dilute to 1.0L with ASTM Type II DI water.

10.28.3 Combine Solutions A and B and label.

11. Calibration

Beta radioactivity emissions are inhibited by the medium through which they must travel. For this reason, when counting radioactivity emissions by gas flow proportional counting, system efficiency decreases as sample residue thickness increases. Over the applicable mass range for this method, this “self-attenuation” effect may be insignificant with certain

gas flow counting systems. If it can be demonstrated that counting efficiency fluctuates by less than 2% over the applicable mass range, for a particular instrument, then calibrations need not take mass-attenuation into account. Otherwise, an appropriate system calibration that corrects for this "self-absorption" characteristic must be performed. Detector calibrations are performed universally independent of sample analysis matrix type. The calibration values determined by application of this process are applied universally for all matrices for which the analytical process is defined in this SOP.

11.1 Beta self-absorption calibration using Sr-89:

11.1.1 The applicable counting mass range for this method is between approximately 10 mg and approximately 30 mg. If it has been demonstrated that the instrument efficiency varies less than 2% over this mass range for the analyte of interest (Sr-89), then correction for self-attenuation is not required for this method. If self-attenuation is not applicable, then instrument calibration should be performed using a minimum of four calibration sources prepared at the optimum sample analysis mass (approximately 20 mg).

11.1.2 For a mass attenuation calibration using Sr-89, to labeled, disposable centrifuge tubes, add varying amounts of strontium carrier solution (10 mg/mL) which will generates a final theoretical strontium carbonate mass range that covers the practical mass range. This expected residue mass range is based on the routine gravimetric target mass for analysis andPASI's minimum and maximum allowable gravimetric recovery limits for this test. An example calibration source setup may be as follows:

Cal. Source Number	Volume of Sr carrier (10 mg/mL)
1	0.5 mL
2	1.0 mL
3	1.5 mL
4	2.0 mL
5	2.5 mL

11.1.3 For a calibration where the average of four efficiencies will be used, add 2.0mL of standardized strontium carrier to each of four labeled centrifuge tubes.

11.1.4 Add between 500 and 1500 dpm (by mass) of a NIST traceable Sr-89 standard to each calibration tube and record the standard mass on the bench sheet. Equivalent amounts of Sr-89 solution should be used for each source. Dilute each calibration solution to approximately 20 mL with ASTM Type II DI water.

Note: The specified calibration source activities have been optimized to allow manageable count times for individual sources. Maximum calibration source activities have been set to minimize the potential impact of any cross contamination within the detector system.

11.1.5 Add 5 mL of conc. NH_4OH solution to each calibration tube.

11.1.6 Add 5 mL of 2N Na_2CO_3 solution to each tube, then heat them in a hot water bath for 15 minutes. Remove each calibration tube from the hot water bath and allow it to cool.

11.1.7 Centrifuge each calibration source for 10 minutes and discard the supernate.

11.1.8 Wash the carbonate precipitate with 20mL of ASTM Type II DI water. Centrifuge and discard the supernate.

11.1.9 Slurry the carbonate precipitate with a few mL of ASTM Type II DI water and quantitatively transfer it to a tared, 2-inch stainless steel planchet.

11.1.10 Dry the calibration sources under a heat lamp.

11.1.11 Re-weigh each source to determine the mass of strontium carbonate recovered.

11.1.12 Count calibration sources in a low-background gas flow proportional counting system as detailed in the instrument SOP, PGH-R-002, "Gas Flow Proportional Counter Operation" current revision. Count each calibration source in each detector requiring calibration long enough to acquire 10,000 net beta counts. Perform efficiency calculations as detailed in Attachment 1 of this SOP.

11.2 Calibration curve acceptance criteria

Calibration curves generated by the process detailed in this SOP must meet the following minimum criteria to be used for sample analysis.

11.2.1 Instrument mass-attenuation calibration must include a minimum of five calibration points for each detector being calibrated. Non-mass-attenuation calibrations must include a minimum of four calibration points with final source mass near the optimum method mass of 20 mg.

11.2.2 If the RSD between all calibration efficiency points is less than 5%, the average efficiency of the calibration points should be calculated and used in all sample calculations.

11.2.3 If the RSD of the efficiencies of the calibration points is greater than 5%, plot the system efficiency (as cpm/dpm) versus source mass (in mg) for each calibration source. Utilize a least squares curve-fitting, exponential or polynomial whichever yields the best fit against the measured data.

11.2.4 Following regression analysis, measured pCi values for each calibration source must be calculated using the calibration curve. Each measured source should be within 10% of known. If the value is not within 10%, assess the point using a z-score. If the z-score for the point is greater than 2.56, the point must be removed from use for calculation purposes. Calibration points may not be removed from the calibration curve without approval of the Department Manager/Supervisor.

11.2.5 Following the removal of individual points, the efficiency must be recalculated using the data for the remaining calibration sources and the calculation process must be repeated until the criteria established in this SOP have been met. A narrative discussing technical justification for modifications or exclusions of any calibration point must be created and kept with the calibration data.

11.3 Calibration Frequency

11.3.1 Calibrations or calibration re-verification for tests associated with drinking water analyses must be performed on an annual basis.

11.3.2 As allowed by specifications within the Manual for the Certification of Laboratories Analyzing Drinking Water, calibration sources may be retained for calibration re-verification purposes. Since Sr-89 is the isotope used for determining efficiency for Ra-228 calculations, and the

half-life is less than 2 months, it is not practical to save Sr-89 calibration sources and reuse them to verify a calibration. New sources must be prepared for verification or calibrations and analyzed annually.

11.3.3 New sources used for verification purposes must be prepared at a similar mass and activity as those used in the initial calibration. Depending on the type of initial calibration used, a minimum of three sources of varying mass must be used to verify a mass attenuation curve, or a minimum of two sources of similar mass must be used to verify an average efficiency.

11.3.4 For the mass attenuation verification, the calculated activity of each of the three verification sources must be within 10% of their target source activity. Any detector not meeting this criteria requires a full calibration as outlined beginning in Section 11.1.2 of this SOP.

11.3.5 For an average efficiency verification, the calculated average efficiency of the two verification sources must be within 10% of the average efficiency determined in the initial calibration. Any detector not meeting these criteria requires a full calibration as outlined beginning in Section 11.1.3 of this SOP.

11.4 Yttrium carrier standardization

11.4.1 Yttrium carrier standardization must be performed prior to analyzing samples in which the yttrium carrier was used.

11.4.2 Add 1.0mL of yttrium carrier (9mg/mL) to each of 5 labeled centrifuge tubes.

11.4.3 Add 20mL 0.25M EDTA solution to each centrifuge tube.

11.4.4 Add 5mL 18M NaOH, stir well, and digest in a hot water bath until yttrium hydroxide coagulates. Centrifuge samples and discard the supernatant.

11.4.5 Dissolve the samples in 2.0 mL of 6N HNO_3, add 5.0 mL of ASTM Type II DI water, and re-precipitate yttrium as a hydroxide by adding 3.0 mL of 10N NaOH.

11.4.6 Heat the samples in a hot water bath for 12 minutes. Centrifuge the samples and discard the supernate.

11.4.7 Dissolve the precipitate in 1.0 mL of 1.0 N HNO_3. Cap the samples and vortex to mix. If cloudy, add 1.0 N HNO_3 dropwise until the solution is clear, up to 6 drops.

11.4.8 Heat the samples in a hot water bath for 3 minutes.

11.4.9 Remove the samples and add 3 mL ASTM Type II DI water and 2.0 mL of 5% ammonium oxalate solution to precipitate yttrium oxalate.

11.4.10 Cap and vortex the samples and return them to the hot water bath for an additional 3 minutes.

11.4.11 Remove the samples form the hot water bath, centrifuge, and discard the supernate.

11.4.12 Add 10.0mL of ASTM Type II DI water to each sample, and 6 drops each of the 1.0 N HNO_3 and 5% ammonium oxalate solution.

11.4.13 Cap and vortex the samples to break up the precipitate.

11.4.14 Return the samples to the hot water bath for 3 minutes. Remove and centrifuge the samples, discard the supernate.

11.4.15 Slurry the precipitate in 6.6 mL of ASTM Type II DI water and quantitatively transfer the contents to a tared planchet placed under a heatlamp. Evaporate the samples to dryness and allow them to cool completely before reweighing.

11.4.16 Enter the tare and gross masses into the yttrium standardization calculation spreadsheet. Calculate the average mass of the five standards and the corresponding percent RSD and standard deviation among them.

11.4.17 The five standard masses must agree within 5% RSD and within ± 3 standard deviations to use the average mass.

11.4.18 Enter the average concentration into the Ra-228 activity calculations spreadsheet as the yttrium target mass for chemical recovery purposes.

11.5 Barium Carrier standardization

11.5.1 Pace employs Ba-133 tracer as the default yield monitor in this procedure. The following steps are included in this SOP in the event the Ba-133 yield assessment cannot be made. Barium carrier standardization is only necessary to perform when required.

11.5.2 Pipette 2.0mL barium carrier to each of 5 labeled centrifuge tubes.

11.5.3 Add 20mL 0.25M EDTA solution and 5mL 18N NaOH to each centrifuge tube.

11.5.4 Add 4mL 16N HNO_3 and swirl to mix.

11.5.5 Add 2.0mL ammonium sulfate (200 mg/mL) .

11.5.6 Add 3mL acetic acid. Cap the samples and vortex to mix.

11.5.7 Heat in a hot water bath for 15 minutes until the precipitate settles.

11.5.8 Centrifuge and discard the supernatant. Rinse the precipitate with 15mL of ASTM Type II DI water. Vortex vigorously.

11.5.9 Centrifuge and discard the supernatant.

11.5.10 Slurry the precipitate in 7mL of ASTM Type II DI water and transfer quantitatively to a tared 2 inch stainless steel planchet. Heat to dryness under an infrared lamp.

11.5.11 Calculate the average of the five standards masses and the corresponding percent RSD and standard deviation among them. The five standard masses must agree within 5% RSD and within ± 3 standard deviations to use the average mass.

11.5.12 Enter the average concentration into the Ra-228 activity calculations spreadsheet as the barium target mass for chemical recovery purposes.

12. Procedure

Unless specified otherwise, the documented analysis process must be followed, as written, including the order of analytical process and the addition of chemicals.

12.1.1 Weigh 800g of aqueous sample into an appropriately sized beaker. Record the observed measured mass of sample to the lowest decimal on the balance. Do not remove sample from the beaker once it has been

added. The actual quantity of sample used may be less than 800 grams if matrix interferences are expected or there is limited sample quantity available to the laboratory. If less than 800g of sample is used, dilute the sample with ASTM Type II DI water to the 800mL mark on the beaker. Fortify the pH of diluted samples by adding 2mL of HNO_3. The cause for utilizing reduced sample volume must be recorded in the analytical logbook. Some sample dilution comments could include; 1. Limited sample was available for analysis, 2. Sample matrix interferences expected, 3. Elevated historical or anticipated sample activity.

12.2 Prepare a Method Blank (MB), Laboratory Control Sample (LCS), and Laboratory Control Sample Duplicate (LCSD) by weighing 800g of ASTM Type II DI water into an appropriately sized beaker. Add 2.0mL of concentrated nitric acid to the MB, LCS, and LCSD. Add the appropriate amount of Ra-228 spike solution to the LCS and LCSD based on the requirements listed in section 14.6.3.

12.3 If a Matrix Spike (MS) is prepared, add the appropriate amount of Ra-228 spike solution to the MS sample based on the requirements listed in section 14.9.2.

12.4 To all samples and QC, add 5mL of 1M citric acid ($C_6H_8O_7 \cdot H_2O$) and a few drops methyl orange indicator and stir. The solution should be red.

12.4.1 If the solution does not turn red, verify the pH of the original sample. If the pH is correct (acidic <2.0), proceed with the analysis. If the pH is not correct, go to Section 8.1.2.

12.5 Add 1mL lead carrier (150 mg/mL), 2mL strontium carrier (10 mg/mL), 2.0mL barium carrier (16 mg/mL), and 1mL yttrium carrier (18 mg/mL). PASI's default procedure for yield determination is made using Ba-133 radiotracer. Add an appropriate quantity of Ba-133 to each sample according to the guidelines in section 12.33.1 of this SOP. Stir well. Heat to incipient boiling and maintain at this temperature for 30 minutes.

12.5.1 Note: The default yield determination method is the use of Ba-133 tracer with tracer recovery determined by sodium iodide counting. For some samples, the Ba-133 tracer carries over to the sample planchet used for measurement of Ac-228 by GFPC counting. The sample components or interferences that are the cause of this phenomenon have not been identified. Discoloration of the final source prepared for Ac-228 counting may be an indication that interference exists, especially for samples yielding a dark oxalate precipitate for the count source.

12.5.2 The presence of Ba-133 on the Ac-228 (Y) oxalate planchet can be confirmed by direct qualitative counting on a gamma spectrometer or recounting the sample planchet on a GFPC detector the following day. Contributions to the beta count rate which are caused by Ba-133 carryover will be consistent from one day to the next due to the relatively long half life of Ba-133 versus the short half life of the Ac-228.

12.5.3 Unless samples have historically indicated the pattern of interference indicated in 12.5.1, all samples should be analyzed using the Ba-133 tracing approach. For all cases, the quantity of barium carrier added is the same.

12.5.4 Samples exhibiting Ba-133 carryover may be re-ingrowthed and reanalyzed providing the Ba-133 tracer yield is significant enough to be able to meet the required MDL upon reanalysis (greater than 50%). Otherwise, the sample should be reprepped using a lower aliquot, and

possibly using stable barium carrier in lieu of Ba-133 tracer to determine chemical yield.

12.6 If necessary, add a few drops methyl orange indicator again as it is destroyed upon prolonged heating. Add 15N NH_4OH until a definite yellow color is obtained, then add a few additional drops. Precipitate lead and barium sulfates by adding 18N H_2SO_4 until the red color reappears, then add 0.25mL in excess. Add 5mL of $(NH_4)_2SO_4$ (200 mg/mL) and stir the sample vigorously until a precipitate forms. Allow the solution to heat for a minimum of 30 minutes and remove the samples to cool.

12.7 Allow the sample precipitate to settle overnight or for a minimum of 2 hours, and siphon off most of the supernatant liquid and discard, saving the precipitate.

12.7.1 The layer of precipitate should be relatively thin on the bottom of the beaker. If a significant amount of precipitate forms, it may be an indication of the presence of excessive sodium or calcium in the sample.

12.7.2 Sodium sulfate and calcium sulfate are soluble in cold water. Calcium sulfate, in particular, has an inverse solubility relationship with temperature, so the colder the water, the more calcium sulfate dissolves.

12.7.3 To remove most of the extra sodium and calcium sulfate precipitate, add ASTM Type II DI water to the beaker up to 800 mL mark on the beaker, stir the sample vigorously, remove the stir rod, and place the sample beaker in an ice water bath or refrigerator for a minimum of thirty minutes or until the precipitate settles completely.

12.7.4 Siphon off the supernatant liquid and discard, saving the precipitate.

12.7.5 Repeat steps 12.7.3 and 12.7.4 two additional times, and proceed to step 12.8 to transfer the reduced precipitate to a centrifuge tube.

12.8 Transfer the precipitate with the aid of 0.1 N sulfuric acid to a 50mL disposable centrifuge tube; centrifuge, and discard the supernatant liquid.

12.8.1 Sample centrifuging compacts the precipitate to the point where it should be possible to completely invert the tube during decanting to remove all of the supernatant chemical solution. When decanting, carefully tilt the tubes until it can be determined that the precipitate is compacted fully then invert the tube to fully decant. Adhere to this requirement for all subsequent decantations from centrifuge tubes.

12.9 Add 25mL EDTA solution to the sulfate precipitate. Vortex vigorously, and heat in a water bath to enhance dissolving of the precipitate. If the precipitate does not readily dissolve, add 10M NaOH solution dropwise. Do not add more than 7 drops of 10M NaOH.

12.9.1 If there is any undissolved material remaining, centrifuge the sample and transfer the solution to a clean disposable centrifuge tube. Submit the supernate solution for NaI detector counting for Ba-133 determination. If the resulting yield is within acceptable limits continue with the analysis. If it is not within the expected limits, combine the supernate and the solid material and reheat.

12.9.2 White precipitate is indicative of sulfate precipitates, and every effort must be made to dissolve as much as possible. Precipitates of any other color are more indicative of solids which may have been present in the sample prior to preparation and can be centrifuged from the sample and discarded in the appropriate waste receptacle.

12.10 To the dissolved sample in EDTA solution, add 1mL strontium-yttrium mixed carrier and stir thoroughly. Add a few drops of 10M NaOH if any precipitate forms.

12.11 Add 1mL $(NH_4)_2SO_4$ (200 mg/mL) and stir thoroughly. Add 2 mL of acetic acid (glacial), with stirring, until the white precipitate of barium (radium) sulfate has formed. Digest in a hot water bath until the precipitate settles and the solution clears. Centrifuge the sample and discard the supernatant.

12.12 Add 20mL EDTA solution to the sample, vortex vigorously, and heat it until the precipitate dissolves. If needed, add 10M NaOH dropwise to enhance dissolution. Do not add more than 7 drops.

12.13 Repeat steps 12.11 and 12.12 one additional time. **At the conclusion of step 12.12 note the time of the last barium sulfate precipitation. (This is the beginning of the Ac-228 ingrowth).**

12.14 Add 20mL of EDTA solution.

12.15 Add 1.0mL yttrium carrier (9 mg/mL standardized) and 1mL lead carrier (1.5 mg/mL). Vortex the sample and heat in a hot water bath for 10 minutes to ensure equilibrium between the yttrium carrier and the actinium in the solution and to dissolve any precipitate.

12.15.1 If the precipitate does not readily dissolve, add 10M NaOH solution drop wise. Do not add more than 7 drops. Cap the centrifuge tube and vortex to mix then store a minimum of 36 hours to allow complete ingrowth of Ac-228. It is important to note that no precipitate is present at this point.

12.16 Following ingrowth, add 0.3mL $(NH_4)_2S$ and stir well. Add 0.5 mL of 10M NaOH. Cap the tube and vortex until lead sulfide flocks. Wait a minimum of **10 minutes** then centrifuge and decant supernatant into a clean, labeled tube.

12.17 Add 1mL lead carrier (1.5 mg/mL). Add 0.1mL $(NH_4)_2S$ and four drops of 10M NaOH to repeat the precipitation of lead sulfide as before (step 12.1.13). Cap the tube and vortex until lead sulfide flocks. Wait a minimum of **10 minutes** then centrifuge and decant supernatant into a clean tube.

12.18 Filter the supernatant through a 11.0 cm diameter Fisherbrand type Q2 or equivalent filter paper into a clean labeled tube.

12.19 NOTE: It is important to proceed without delay to the final separation and count of the Actinium 228 activity to minimize decay. Optimally, the following steps will take approximately 3 hours to complete.

12.20 Add 5mL 18M NaOH, stir well, and digest in a hot water bath for **12 minutes** until yttrium hydroxide coagulates. Centrifuge samples and decant supernatant into a clean, labeled centrifuge tube. Save the supernatant for Ba-133 yield determination, step 12.33. **(Record the time that the 18M NaOH is added [time of separation]; this is the end of the actinium-228 ingrowth time and beginning of Ac-228 decay time)**.

12.21 To remove residual Ba-133 tracer which can cause erroneously high sample results, add 5 mL of ASTM Type II DI water to each sample, vortex, and centrifuge. Discard the supernate.

12.22 Carefully dissolve the precipitate in 0.5 mL 6N HNO_3. Cap and vortex the samples to ensure dissolution. Add 5mL ASTM Type II DI water. Reprecipitate yttrium hydroxide by adding 3mL 10M NaOH. Heat and stir in a hot water bath

for **12 minutes** until precipitate coagulates. Centrifuge sample and discard supernatant.

12.23 To remove residual hydroxide and Ba-133 tracer, add 5 mL of ASTM Type II DI water to each sample, vortex and centrifuge. Discard the supernate.

12.24 Dissolve the precipitate in 0.25 mL of 1N HNO_3. Cap the tubes and vortex. The samples should be cloudy.

12.25 Remove the caps from the tubes and add 1N HNO_3 dropwise to each sample until the samples are almost clear. Do not add excess, as this will result in fractionation of the Ac-228 from the yttrium carrier, biasing results low. Include a note in the preparation logbook for any samples that do not turn clear.

12.26 Cap the samples, vortex and return them to the hot water bath for **3 minutes**.

12.27 Add 3.5mL of DI and add 2mL 5% ammonium oxalate. Cap and vortex the samples.

12.27.1 The samples should appear “milky” at this step.

12.28 Heat the samples in a hot water bath for **3 minutes**. (Prolonged heating may result in significant dissolution of yttrium oxalate back into solution and reduced yield recovery.) Centrifuge the samples and discard supernate.

12.29 Add 10mL ASTM Type II DI water to the precipitate. Add 6 drops of 1N HNO_3 and 6 drops of 5% ammonium oxalate.

12.30 Vortex vigorously and heat in a hot water bath for **3 minutes**. (Prolonged heating may result in significant dissolution of yttrium oxalate back into solution and reduced yield recovery.) Centrifuge sample and discard supernatant.

12.31 Slurry the oxalate precipitate with 6.6mL of ASTM Type II DI water and transfer quantitatively to a tared, 2-inch stainless steel planchet. Dry under an infrared lamp, cool, and reweigh to determine yttrium recovery.

12.32 To determine Ra-228 (Ac-228) content, count samples in a low-background gas flow proportional counting system as detailed in the instrument SOP, PGH-R-002, “Gas Flow Proportional Counter Operation” current revision. The specific Gas Flow Proportional detectors utilized must have been calibrated following instructions in Section 11 of this SOP.

12.32.1 Following beta counting, perform calculations as detailed in Attachment 1 of this SOP. Note: Due to the short half-life of Ac-228 (6.02 hours), it is imperative to count sources as soon as possible following yttrium hydroxide precipitation as performed in Step 12.20 of this SOP.

12.32.2 The maximum practical count time for achieving the RL of 1.0pCi/L is 600 minutes. Samples which do not meet the RL within 600 minutes may require re-ingrowth or reprep depending on the possible reasons the samples could not meet the RL.

12.33 Barium-133 Yield Assessment

12.33.1 Barium-133 is diluted from a NIST traceable solution. Approximately 2500-3000 dpm is added to each sample. The amount added is based on minimizing the required count time for yield determination while ensuring the tracer does not carryover and contribute extraneous counts during the final sample count by GFPC counting.

12.33.1.1 Dilute the barium solution from step 12.20 to 30mL with ASTM Type II DI water. Cap the tube and shake it to mix the sample thoroughly.

12.33.1.2 Prepare a Ba-133 reference solution by adding a known quantity of barium-133 tracer to a 50mL centrifuge tube. Add 20mL of 0.25M EDTA solution and 5mL 18M NaOH. Dilute to 30mL with ASTM Type II DI water. Cap and shake to mix. (The proportion of the quantity added to the samples in 12.5 and the quantity added to the reference solution must be known in order for the yields to be determined.)

12.33.1.3 Count the reference solution and samples for Ba-133 gamma activity on a sodium iodide detector. Perform Ba-133 yield calculations as detailed in Attachment 1 of this SOP.

12.34 Barium yield by gravimetric assessment (used if Ba-133 is unavailable or for samples suspected to have interference from the use of Ba-133 tracer as indicated in Section 12.5 of this SOP.)

12.34.1 Perform Steps 11.5.4 through 11.5.10 on the solution saved from step 12.20.

12.34.2 Calculate the mass recovered on the planchet and calculate chemical recovery based on the target barium sulfate mass obtained during the barium carrier standardization.

13. Calculations

13.1 Refer to Attachment I of this SOP for Ra-228 associated calculations.

13.2 Any verified result for drinking water that exceeds the maximum contaminant level (MCL) established for (the sum of) Radium 228 and Radium 226 must be reported to the appropriate personnel and agencies according to the specific requirements of the state where the water was sampled. The directions for reporting any results that exceed the MCL limits are documented in the State Drinking Water Emergency Reporting Requirements Binder and Pace SOP PGH-C-025, current revision.

13.2.1 The MCL for radium in drinking water is Ra-226 + Ra-228 ≥5pCi/L OR either Ra-226 OR Ra-228 ≥ 5 pCi/L.

14. Quality Control

14.1 General guidelines for drinking water samples with results that exceed the Maximum Contaminant Level include the following: (All steps are to be conducted as soon as the exceedence has been identified.)

14.1.1 Verify the result(s) to ensure that there were no transcription or calculation errors and that all QC results are within the acceptable limits. Correct any problems and determine the new result. If there were no errors or the result still exceeds the MCL continue with the reporting process.

14.1.2 Immediately notify the Department Manager/Supervisor or specified designee, Department Manager/Supervisor and Quality department that a reportable result has been identified. Use telephone notifications to inform the contact people if the variance is identified after hours along with an e-mail follow up to document the event.

14.1.3 Refer to the State Drinking Water Emergency Reporting Requirements Binder for the state specific information regarding the proper course of action to take. Time is of the essence during this process with some of the state reporting requirements as short as 1 hour from the verification of an exceedence.

14.2 Each analyst who performs this test must satisfactorily complete a Demonstration of Capability Study as documented in Section 3.4 of the most recent revision of the Quality Assurance Manual.

14.2.1 The DOC study results are evaluated against the LCS acceptance limits.

14.2.2 IDOC and DOCs for drinking water methods must be done at a spiking level between the sensitivity level and MCL.

14.3 Daily instrument Quality Control checks for gas flow proportional counter must be completed following the instructions detailed in the SOP for Gas Flow Proportional Counter Operation, PGH-R-002 (current revision).

14.4 See Appendix II for performance indicator evaluation calculations and criteria. Numerical performance indicators may be used to assess QC for non-drinking water samples when the default assessment indicates a QC failure. The numerical performance indicator must be within +/- 3 for all other matrices. The z-score for precision assessment may be used for drinking waters with the approval of the Department Manager/Supervisor using the +/- 2 specification.

14.5 Method Blank (MB)

14.5.1 One MB must be prepared for each analytical batch. The purpose of the MB is to monitor for cross contamination during the analytical process. When available, the MB should be prepared from a similar matrix as samples contained in the analytical batch. If appropriate blank matrix material is not available, ASTM Type II DI water (Reagent Blank) must be carried through the procedure. A reagent blank may be used for sample correction purposes following approval of the Department Manager and affected clients.

14.5.2 The results of the method blank must be less than the reporting limit.

14.5.2.1 If the Method Blank is out of control, individual sample results may still be reportable if results are less than the CRDL (contract required detection limit) or greater than 10 times the blank result. Relative quantities of the sample and blank must be factored when making this determination (raw counts).

14.6 Laboratory Control Sample (LCS)

14.6.1 One LCS must be prepared for each analytical batch.

14.6.2 Typical detection limits are 1 pCi/L for Ra-228.

14.6.3 The Ra-228 spike activity must be between 2 and 10 times the detection limit.

14.6.4 A reference material containing a known concentration of radium-228 radioactivity in the same matrix as the batch is analyzed with the batch.

14.6.4.1 If this material is not available, a well-characterized material (WCM) may be used.

14.6.4.2 If neither of these are available, ASTM Type II DI water may be spiked with the appropriate radium-228 standard.

14.6.5 Calculations of LCS activity include source decay correction to account for decay between the LCS spike solution certificate reference date/time and the LCS sample analysis date/time.

14.6.6 Percent Recovery Calculation

$$\%REC = \frac{(LCSConc)}{TrueValue(decaycorrected)} * 100$$

Where:

LCSConc = Analytical result of the LCS
TrueValue = Known concentration of the LCS

14.6.7 LCS %REC acceptance limits are 60-135%.

14.7 Laboratory Control Sample Duplicate (LCSD)

14.7.1 An LCSD is not required for radium-228 analysis; however analysis of an LCSD must be utilized to measure batch precision whenever adequate sample quantity is not available for sample DUP analysis. The LCSD must be prepared in an identical fashion as the LCS and processed identically as for other samples.

14.7.2 The LCSD must pass the acceptance criteria for the LCS and the criteria established for duplicate precision (RPD 36% or less).

14.7.3 If the LCS and LCSD both pass %REC criteria however fail RPD criteria the batch results may be qualified and reported at the discretion of the analyst with guidance from the department manager.

14.8 Sample Duplicate (DUP)

14.8.1 One Duplicate Sample (DUP) must be randomly assigned within each batch. The purpose of the sample DUP is to measure precision of the analytical process. Laboratory duplicates are not intended to assess precision related to the sample collection process. Sample collection precision can only be assessed through collection of duplicate samples at the time of sample collection. The sample DUP is a duplicate quantity of sample processed identically as other samples in the analytical batch.

14.8.2 For batches with drinking water samples originating from the state of Arizona, Duplicate Samples (DUP) must be randomly assigned within each batch at a frequency of no less than 10%. A batch of 10 samples or fewer must contain at least one duplicate sample. A batch of greater than 10 samples up to 20 samples must contain a minimum of two duplicate samples if the batch contains samples originating from Arizona.

14.8.3 Relative Percent Difference Calculation (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

Where

R1 = Result Sample 1
R2 = Result Sample 2

14.8.4 Duplicate sample RPD acceptance limits are <36% for radium-228.

14.8.5 Sample duplicate criteria cannot be applied if results are below their associated MDC.

14.8.6 Due to the limits for acceptable LCS/LCSD recovery, it is possible to have an acceptable LCS and LCSD, and still not meet the duplicate samples RPD acceptance limit. In this case, if the individual LCS and LCSD recovery criteria are met, the sample data may be reported with narration.

14.9 Sample Matrix Spikes (MS)

14.9.1 Because this analytical method requires the use of carriers or radiotracers for yield determination, PASI's default QC policy is that a sample matrix spike (MS) is not required for radium-228 analysis with the exception of drinking water analysis.

14.9.2 Typical detection limits for Ra-228 are 1 pCi/L. The spike amount must be greater than 10 times the detection limit.

14.9.3 The MS is prepared by spiking a portion of radium-228 radioactivity solution into a portion of one sample in the batch and processing identically as for other samples.

14.9.4 The purpose of the MS is to assess the affect of sample components on the analytical process. The quantity of sample used for the MS must be equivalent to the quantity of sample used for sample analysis.

14.9.5 Calculations of MS activity include source decay correction to account for decay between the spike solution source certificate reference date/time and the MS sample collection date/time.

14.9.6 Percent Recovery Calculation

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue(decaycorrected)} * 100$$

NOTE: The SampleConc is zero (0) for the LCS

14.9.7 MS acceptance limits are 60-127% for radium-228.

14.10 Sample Matrix Spike Duplicates (MSD)

14.10.1 A sample Matrix Spike Duplicate (MSD) is not required for this analysis. When required by the customer/contract, a MSD must be prepared for each analytical batch. The MSD must be prepared as a duplicate of the MS.

14.10.2 The MSD must pass the acceptance criteria established for the MS recovery and the criteria established for duplicate precision.

14.11 PASI's default criteria for carrier and/or tracer yield are 30-110% of the expected value.

14.12 Summary of QC related Activities:

Method Blank	One per Batch
Reagent Blank	One per Batch (as required by client)

Duplicate Sample	One per Batch or a frequency of 10% for batches containing DW samples from Arizona.
Matrix Spike	One per Batch (for drinking waters or as required by client)
Matrix Spike Duplicate	One per Batch (or a frequency of 10% for batches containing DW samples from Arizona.or as required by client)
Laboratory Control Sample	One per Batch
Laboratory Control Sample Dup	One per Batch (in absence of Duplicate sample)

14.13 Corrective Actions for Out-Of-Control Data

14.13.1 Method Blank (Reagent Blank) (MB/RB) – Individual samples that do not meet the acceptance criteria must be reanalyzed. If there is no additional sample available for reanalysis and evaluate the usefulness of the data in the final report.

14.13.2 Duplicate (DUP) – DUP analysis that fails the replicate test must be reanalyzed to determine if analytical failure or sample heterogeneity was the cause of the problem.

14.13.3 Matrix Spike Recovery (MS) – MS recoveries that fail high and outside of control criteria with a sample result that is less than the reporting limit may be reported with narration. Additionally, MS recoveries that fail low and outside of control criteria for Drinking Water samples with a sample result that is greater than the MCL must be reported with comment as potentially biased high due to matrix interference. Otherwise, MS recoveries that do not meet the acceptance criteria must have that sample reanalyzed. If a Matrix Spike Duplicate is also analyzed and the recovery is comparable to the MS, the results are reported and noted in the final report. Matrix effect must be determined by re-analysis of the MS/Sample pair or demonstration of acceptable precision between a MS/MSD pair.

14.13.4 The analyst must evaluate the MS results to attempt to determine the cause of the failure and the appropriate action to take based on that evaluation. All decisions made must be documented.

14.13.5 Matrix Spike Duplicate (MSD) - If an MSD is analyzed and the recovery is comparable to the MS, the results are reported with qualification in the final report.

14.13.6 Laboratory Control Sample (LCS) – If an LCS analysis does not meet the acceptance criteria, the entire analytical batch must be re-prepped and reanalyzed.

14.13.7 The results of the batch may be reported, with qualification in the final report, if the LCS recoveries are high and the sample results within the batch are less than the reporting limit.

14.13.8 Laboratory Control Sample Duplicate (LCSD) – If an LCSD does not meet the recovery acceptance criteria, the entire analytical batch must be reanalyzed.

14.13.9 The results of the batch may be reported, with qualification, if the LCS recoveries are high and the sample results within the batch are less than their reporting limit, and duplicate precision meets the acceptance criteria.

14.13.10 If there is no available sample volume remaining for re-analysis and evaluate the usefulness of the data in the final report. Carrier Recovery: If analysis of samples other than drinking water matrices results in a carrier recovery outside of the default acceptance range, results may be reported with appropriate qualification and approval of the client. If analytical results are reported, they are noted in the case narrative. For all other analyses (including Drinking Water analysis), samples that result in a carrier recovery outside of the default acceptance range must be reanalyzed. If reanalysis results in a carrier recovery outside of the default acceptance range, the results are reported and qualified.

14.13.10.1 Default acceptance limits are 30-110% for gravimetric carriers. Results as high as 130% may be reported with permission of the Department Manager/Supervisor or manager-specified designee.

14.13.11 Tracer Recovery: If analysis of samples other than drinking water matrices results in a tracer recovery outside of the default acceptance range, results may be reported with appropriate qualification and approval of the client. If analytical results are reported, the results are noted in the case narrative. For all other analyses (including Drinking Water analysis), samples that result in a tracer recovery outside of the default acceptance range must be re-analyzed. If re-analysis results in a tracer recovery outside of the default acceptance range, the results are reported, and noted in the case narrative. For Ra-228 analysis using Ba-133 tracer, the uncertainty calculations for yield measurement include a maximum allowable 1 sig. uncertainty of 5% which correlates to 400 net tracer counts. If tracer yield measurement results in the acquisition of less than 400 net tracer counts, the result must be appropriately qualified.

14.13.11.1 Default acceptance limits are 10-110% for radioactive tracers. Results as high as 130% may be reported with permission of the department manager or manager-specified designee.

14.14 Contingencies for handling Out-of-Control or Unacceptable Data

14.14.1 Method Blank (Reagent Blank): If the sample is exhausted, evaluate the usefulness of the data and appropriately qualified and/or narrated.

14.14.2 Duplicates: If the sample is exhausted evaluate the usefulness of the data and appropriately qualified and/or narrated.

14.14.3 Matrix Spike Recovery: If a Matrix Spike Duplicate is analyzed and the spike recoveries are not comparable, and the sample is exhausted, evaluate the usefulness of the data and appropriately qualified and/or narrated.

14.14.4 Matrix Spike Duplicate: If a Matrix Spike Duplicate is analyzed and the spike recovery is not comparable to the Matrix Spike and the sample is

exhausted, evaluate the usefulness of the data and appropriately qualified and/or narrated.

14.14.5 Carrier Recovery: If the sample is exhausted evaluate the usefulness of the data and appropriately qualified and/or narrated.

14.14.6 Tracer Recovery: If the sample is exhausted, evaluate the usefulness of the data and appropriately qualified and/or narrated.

15. Method Performance

15.1 Each analyst must read and understand this procedure with written documentation maintained in their training file on the Learning Management System (LMS).

15.2 An initial demonstration of capability (IDOC) study must be performed. A record of the IDOC will be maintained on file in each analysts training file in the LMS.

15.3 On an annual basis, each analyst will complete a continuing demonstration of capability (CDOC).

15.4 Laboratory control samples are analyzed with each batch, the results are charted to monitor control limits and trending.

16. Pollution Prevention and Waste Management

16.1 Place radioactive waste into appropriate receptacles.

16.2 Discard acidified samples and unusable standards into proper waste drains.

16.3 Dispose of waste materials in accordance to type: Non-hazardous, hazardous, non-radioactive, radioactive or mixed.

17. References

17.1 Krieger, H. L. and Whittaker, E. L., *Prescribed Procedures for* Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, "Radium-228 in Drinking Water," Method 904.0, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH, August, 1980.

17.2 Eaton, A. D., et. al., editors, Standard Methods for the Examination of Water and Wastewater, 20th Edition, "Radium – Sequential Precipitation Method," Method 7500-Ra D., American Public Health Association, Baltimore, MD, 1998.

17.3 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW846), Volume 1C, Third Edition, "Radium-228," Method 9320, U. S. Government Printing Office, Washington, D.C., September 1986.

17.4 ASTM E181-93, Standard Test Methods for Detector Calibration and Analysis of Radionuclides, ASTM Standards, Vol. 12.02.

17.5 Table of Radioactive Isotopes, Brown and Firestone, Shirley editor, John Wiley & Sons, 1986.

17.6 Currie, L., Limits for Quantitative Detection and Quantitative Determination, Analytical Chemistry, Vol. 40. No. 3, Pg 586-593, 1968.

17.7 Currie, L., Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements, NUREG/CR - 4007, USNRC, 1984.

17.8 “American National Standard Calibration and Usage of Alpha/Beta Proportional Counters”, ANSI N42.25-1997.

17.9 "Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)", July 2004, Final.

17.10 "American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories", ANSI N42.23-1996.

17.11 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.

17.12 Department of Defense Quality System Manual (DoD QSM) version 4.2 (or most recent version).

17.13 "Manual for the Certification of Laboratories Analyzing Drinking Water" Fifth Edition, January 2005, EPA 815-R-05-004.

17.14 National Primary Interim Drinking Water Regulations (NIPDWR), Part 141.15.

17.15 Pace Pittsburgh SOP, PGH-R-002, current revision, Gas Flow Proportional Instruments Operation.

17.16 Pace SOP PGH-C-025, current revision (MCL Violation Reporting).

17.17 Pace SOP PGH-C-027, current revision (Deionized Water Quality and Suitability).

17.18 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (most recently approved revision).

17.19 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.

17.20 Pace Analytical Services, LLC. - Pittsburgh Laboratory Quality Assurance Manual, current version.

17.21 SOP PGH-C-032, Support Equipment, current version.

17.22 SOP PGH-Q-038, Laboratory Equipment, current version.

17.23 SOP PGH-Q-040, Internal and External Audits, current version

17.24 SOP PGH-Q-039, Corrective and Preventative Action, current version.

17.25 SOP S-ALL-Q-020, Training, current version,

17.26 SOP S-ALL-Q-028, Lab Track, current version

18. Tables, Diagrams, Flowcharts, Appendices, etc.

18.1 Attachment I: Calculations

18.2 Attachment II: Numerical Performance Indicators

19. Method Modifications.

19.1 For routine analysis of aqueous samples, PASI's default process for measuring the quantity of sample to be analyzed is to measure the mass of sample transferred and the mass of sample used is documented in the appropriate logbook. Subsequent calculations for analysis of aqueous samples assume the density of aqueous samples to be 1.0 g/mL. For these samples, analysis results are reported in volume units without density correction.

19.2 EPA method 904.0 specifies the use of 10mL of lead carrier with a concentration of 15 mg/mL, while this procedure uses 1 mL of lead carrier with a concentration of 150 mg/mL. The amount of lead added is equivalent. PASI uses a more

concentrated solution to reduce the chemical reagent footprint within the laboratory.

19.3 EPA method 904.0 specifies the use of 5 mL of Ammonium sulfate, 200mg/mL per liter of sample. This SOP requires the consistent addition of 5 mL of ammonium sulfate, 200mg/mL to samples independent of sample volume.

19.4 For Section 12.8 of this SOP, samples are siphoned and centrifuged to isolate the barium precipitate, whereas, the EPA method 904.0 specifies filtering the sample to collect the precipitate. Sample losses due to this difference are accounted for with the barium yield determination and do not adversely affect sample results.

19.5 In Section 12.21 and 12.23 of this SOP, a rinse of the yttrium hydroxide precipitate using ASTM Type II DI water has been added to enhance removal of residual Ba-133 tracer which could contribute to the beta count rate during sample analysis. Ba-133 tracer is not utilized in EPA method 904.0. Sample losses due to the rinses are accounted for with the yttrium oxalate yield determination and do not adversely affect sample results.

19.6 EPA method 904.0, section 8.13, specifies the addition of 10N NaOH dropwise with vigorous stirring until lead sulfide precipitates, then add 10 drops excess. Pace has quantified the addition of 10N NaOH to 0.5 mL in section 12.16 of this SOP.

19.7 EPA method 904.0, section 8.14, specifies washing the filter with a few mL of DI water. Pace does not rinse the filter. Sample losses due to this difference are accounted for with the barium and yttrium yield determination and do not adversely affect sample results.

19.8 EPA method 904.0 specifies the addition of 2mL of 6N HNO_3 for the second yttrium hydroxide precipitation and 1mL of 1N HNO_3 for the initial yttrium oxalate precipitation. The correlating steps in this procedure have reduced the volumes to 0.5mL of 6N HNO_3 and 0.25mL of 1N HNO_3. The reduction in acid is necessary, due to the ASTM Type II DI water rinses of Sections 12.21 and 12.23. Excessive acidity causes actinium and yttrium to fractionate, creating a chemical disconnect between the analyte (actinium-228) and the carrier (yttrium), contributing to a low bias for analytical results.

19.9 EPA Method 904.0 specifies to use 1,000 mL of sample for analysis. Pace analyzes approximately 800 mL of sample for analysis. All samples are measured by mass as specified within this SOP. Pace further restricts the analysis volume for samples known to contain matrix components that would limit analyte recovery. Pace restricts the analysis volume so as to limit the quantity of waste generated and to enhance productivity. The required detection limit of 1 pCi/L is achieved using a 800 gram analysis quantity.

19.10 EPA Method 904.0 calculations for activity do not show decay correction from the collection of samples to the analysis date of samples. Pace decay corrects Ra-228 results for all samples to the client specified collection date and time. This calculation is defined in Eq 5. of this SOP.

19.11 For the preparation of 0.25M EDTA, EPA Method 904.0 specifies to add sodium hydroxide to DI water then heat prior to adding the solid EDTA for dissolution. The heat generated from the dissolution of sodium hydroxide in water is sufficient to dissolve the EDTA and so, hot-plate heating is not performed.

20. Revisions

Document Number	Reason for Change	Date
PGH-R-003-13	1. Added note at step 12.5.1 to describe process for full decantation of sample in centrifuge tubes. 2. Revised section 12.12 to wait 10 minutes rather than five for lead flocking to occur. 3. Added steps 12.18 and 12.20 to perform a 5mL DI water rinse to remove Ba-133 tracer interference from samples. 4. Revised step 12.19 to use 0.5 mL 6N HNO3 instead of 2.0 mL to prevent fractionation of yttrium due to high acid content 5. Revised step 12.21 to use 0.25 mL of 1N HNO3 and to add dropwise to just slightly cloudy, to prevent fractionation of yttrium due to high acid content. 6. Revised step 12.22 and 12.23 to place samples in the hot water bath for 3 minutes following the addition of 1N HNO3 in following with the EPA written method. 7. Revised step 12.25 and 12.27 to limit the time the yttrium oxalate precipitate is in the hot water bath to 3 minutes to prevent yield losses. 8. Added 14.8.5 to include the ability to report samples results when a LCS/LCSD RPD failure occurs. 9. Modified section 14.5.1 to require approval of the Department Manager for approval to blank correct sample results. 10. Revised section 14 to address default criteria for gravimetric carriers and radioactive tracers. 11. Change wording in section 14 to address non-conformance with LabTracks and also to evaluate data and appropriately qualify or discuss in narrative for reports that do not include a case narrative.	25Apr2012
PGH-R-003-14	1. Updated Cover page, headers/footers for this revision 2. Added Arizona Drinking water batching requirements for Duplicate to Section 14.8.2.	11July2012
PGH-R-003-15	1. Updated Cover Sheet, Headers and Footers. 2. Section 4: Added noted interferences and the step in this SOP where they are addressed. 3. Section 6: Added reference to glossary section of QAM for definitions of terms and added section regarding recording observed measurements, prohibiting weight targeting, and not removing sample from beakers once added. 4. Section 10: Added ASTM Type II DI water preparation and reference, changed all DI water to ASTM Type II DI water throughout document. 5. Section 11: Added calibration frequency requirements, documentation required for removal of calibration points. 6. Section 12: Added comment regarding performing steps and chemical additions in order specified. 7. Section 12.2: Inserted steps to ensure the Ra-228 spike is added to LCS/LCSD samples after the initial sample acidification and prior to any other chemical additions.	23Sep2013

Document Number	Reason for Change	Date
	8. Section 12.3: Inserted to ensure Ra-228 spike additions to the MS are specified to occur prior to all chemical additions. 9. Section 12.5: Added information regarding determining if Ba-133 carryover has occurred and how to proceed. 10. Section 12.7: Added instructions on reducing excess sulfate precipitates due to calcium and sodium interferences. 11. Section 12.9.2: Clarified types of precipitates expected and which need to be dissolved. 12. Section 14: Added criteria for appropriate use of Numerical Performance Indicators. 13. Section 17: Updated references. 14. Section 19: Added comments regarding Pace's default sample measurement technique, density assumptions, and how to use the quantity measurement in the spreadsheet. 15. Attachment II: Updated combined standard uncertainties are assessed at 1 sigma.	
PGH-R-003-16	1. Annual SOP review and update. 2. Section 2.1 – Included reference to standard methods. 3. Section 2.4 – Moved the decay correction application discussion from Section 3 to be consistent with other Pace SOPs. 4. Section 8.1.2 – Added pH verification requirement and recording. 5. Section 8.4 – Added hold time requirement. 6. Section 9 – Added reference for instrument SOP and GFPC instrument components. 7. Section 9.7 and 9.9 – Changed filter paper to current brand and type being utilized by laboratory, added sample planchet dimensions. 8. Section 10 – Included preparation of Na_2CO_3 solution since it is used in calibration process. 9. Section 11.3 – Inserted to specify amount of strontium carrier added to calibration sources when preparing average efficiency calibration (not mass attenuation). 10. Section 11.3.3-11.3.5 – Added source preparation requirements for verification sources and calibration verification acceptance criteria. 11. Section 11.4.1 and 11.5.1 – Inserted comment for when carrier standardization is required. 12. Section 12.1 – Inserted instructions for diluting samples. 13. Section 12.5 – Changed order of chemical addition to match EPA method 904.0, inserted strontium carrier – inadvertently omitted in prior revision. 14. Section 12.9 – changed EDTA amount from 20 mL to 25 mL to match EPA method 904.0. 15. Section 12.33.1 – Included amount of Ba-133 added to samples and comment regarding concentration. 16. Included reference to actual GFPC Pace SOP, PGH-R-	13Jul2014

Document Number	Reason for Change	Date
	002, current rev where instrumentation discussed throughout document. 17. Section 14.12 – Included AZ DW QC requirements for duplicate and MSD. 18. Section 15.3 – Included CDOC requirements. 19. Section 17 – Added GFPC Instrument SOP, PGH-R-002, current revision. 20. Section 19 – Added notable deviations from methods including concentration of lead carrier, siphoning versus filtration, use of Ba-133 and ASTM Type II DI rinses, decreased acid volumes for 6N and 1N HNO_3 additions. 21. Reformatted document.	
PGH-R-003-17	1. Updated Section 4.3 to reference the correct SOP location which addresses Ba-133 carryover and bias. 2. Updated Section 4.4 to reference the correct SOP location which specifies the treatment option for samples generating excess calcium sulfate and sodium sulfate during the initial sample pre-concentration. 3. Section 19 updated to include the analysis volume difference between this SOP and the method, EPA 904.0.	17Dec2015
PGH-R-003-18	1. Section 2.4 – Clarified how decay correction is applied to samples. 2. Section 12.1.1 – Updated to include notating reasons for not using routine samples aliquots. 3. Section 14.6.5 and 14.9.5 – Included to specify how the spike true value for the LCS and MS are decay corrected. 4. Section 19.3 and 19.6 – Updated to include deviations in the amount of 10N NaOH and 200 mg/mL ammonium sulfate added to each sample. 5. Section 19.7 – Deviation for rinsing with filter DI water. 6. Section 19.10 – Added deviation for applying decay correction to sample collection date and time to sample results.	21Dec2016
S-PGH-R-003-rev.19	1. Updated section 8.1.2 samples must be held minimum of 24 hours. 2. Modified section 10.11 to remove the requirement to heat EDTA solution during preparation. Heating is not necessary to prepare the solution. 3. Sections 14.13.3 and 14.13.4 modified to clarify the corrective actions for failed sample matrix spikes and/or sample matrix spike duplicates. 4. Added notation at Section 19.11 to specify non-heating of EDTA solution during preparation as a modification.	08Feb2018

Attachment I – (Calculations)

The **radium-228 concentration** of a sample is calculated according to the following equations:

Eq. 1 $$A = \frac{(S - B)}{(Denom)}$$

Eq. 2 $$Denom = E * V * 2.22 * D * I * C * X * R_A * R_B$$

Eq. 3 $$D = e^{-\lambda t1}$$

Eq. 4 $$C = \frac{(1 - e^{-\lambda t3})}{\lambda t3}$$

Eq. 5 $$X = e^{-\lambda 2 t4}$$

Eq. 6 $$I = 1 - e^{-\lambda t2}$$

Eq. 7 $$\lambda = \frac{\ln 2}{T_{1/2}}$$

Eq. 8 $$\lambda 2 = \frac{\ln 2}{T_{1/2Ra}}$$

Eq. 9 $$R_A = \frac{M_Y}{T_Y}$$

Eq. 10 $$R_B = \frac{M_B}{T_B} \text{ OR } R_B = \frac{M_{Ba133}}{T_{Ba133}}$$

Where:

A	=	Ra-228 concentration in pCi/L.
S	=	Sample gross beta count rate (in cpm).
B	=	Background beta count rate (in cpm).
2.22	=	Conversion factor from dpm to pCi.
E	=	Detector efficiency (As determined in Section 11 of this SOP)
V	=	Sample volume (in liters).
D	=	Ac-228 decay factor between initial precipitation and start of count.
I	**=**	**Fractional ingrowth of Ac-228 into the purified radium sample**
C	=	Ac-228 decay factor from beginning of count to midpoint of count.

X	=	Ra-228 decay factor from sample collection date and time to count start date and time.
t1	=	Decay time in hours between final yttrium hydroxide precipitation and midpoint of count time.
t2	=	Ingrowth time in hours between first yttrium hydroxide precipitation and second yttrium hydroxide precipitation.
t3	=	Count time in hours.
t4	=	Decay time in days between the sample collection date and the sample count date.
$T_{1/2}$	=	Half-life of Ac-228 in hours (6.02 hours).
$T_{1/2Ra}$	=	Half-life of Ra-228 in years (5.75 years)
R_A	=	Fractional recovery of yttrium.
R_B	=	Fractional recovery of barium.
M_Y	=	Mass of yttrium oxalate recovered (in mg).
M_B	=	Mass of barium sulfate recovered (in mg).
M_{Ba133}	=	Measured sample Ba-133 Net Cts from the sodium iodide counter yield measurement
T_Y	=	Standardized yttrium carrier conc. (in mg yttrium oxalate per mL of yttrium carrier used)
T_B	=	Standardized barium carrier conc. (in mg barium sulfate per mL of barium carrier used)
T_{Ba133}	=	Measured Net Cts of the reference source for the specific sodium iodide detector used for the sample recovery determination.

The sample specific **counting uncertainty** C.U. is calculated as follows.

Eq. 11 $$C.U. = \frac{1.96 * \sqrt{((S/T_S)+((B/T_B)}}{Denom}$$

Where:

T_S	***=***	***Count time for the sample (in minutes)***
T_B	=	Count time for the background count (in minutes)

S, B, and Denom as previously defined.

As summed background and analyte count rates approach zero, assumptions underlying the uncertainty calculation are violated and it will return an unrealistic value of zero (0) uncertainty when zero summed counts are observed. The following equation provides a more accurate estimate of count uncertainty at zero and near-zero count rates.

Eq. 12 $$ZeroUnc=ZeroActFact/SmplTime$$

Note 1: Depending on sample type and contract requirements the zero activity factor may be either 3.0 or 2.71. PASI's default ZeroActFact is 2.71 consistent with the current version of ANSI N42.23. Bioassay samples must be calculated using 3.0 to be consistent with ANSI N13.30

Note 2: The Zero Count Uncertainty is compared to the count uncertainty above. The larger of the two is used as the counting uncertainty in subsequent total error calculations.

The error term is further evaluated to provide an estimate of total error hereafter referred to as the *Combined Standard Uncertainty* (CSU a.k.a. TPU). The CSU is calculated as follows:

Eq. 13 $$CSU\,(pCi/U) = \sqrt{(C.U.)^2 + (UE1 * A)^2 + (UE2 * A)^2 + (UE3 * A)^2 + (UE4 * A)^2}$$

Where:

UE1, UE2, UE3, and UE4 represent partial derivatives estimating the relative uncertainty at the ***95% confidence interval*** for various factors in the activity calculation as follows:

UE1 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with preparation (e.g., sample aliquot or transfers and splits prior to addition and equilibration of tracer).

UE2 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with analysis (e.g., peak integration, peak overlap, tracer contaminants).

UE3 represents combined factors estimating relative uncertainty (fractional) associated with yield correction (e.g., count uncertainty for tracer peak, SRM known value, tracer volume or mass aliquot, tracer equilibration efficiency).

UE4 represents the factor estimating additional uncertainty (activity) associated with an individual sample -- to be used in exceptional circumstances with approval of the Department Manager and appropriate documentation and narration only.

The Minimum Detectable Concentration (MDC) is calculated per guidance of ANSI N42.23 and N13.30 as:

Eq. 14 $$MDC = \frac{4.65 * \sqrt{B * T_S} + ZeroActFact}{T_S * Denom}$$

Where:

B, T_S, ZeroActFact, and Denom are as previously defined.

The critical level (Lc) is calculated per guidance of ANSI N42.23 as:

Eq. 15 $$Lc = \frac{1.65 * \sqrt{(B) * (1/Ts + 1/Tb)}}{Denom}$$

Where:

B, T_s, T_b, *ZeroActFact*, and Denom are as previously defined.

Attachment II - (Numerical Performance Indicators)

1. **Method Blank (MB)**

1.1 The numerical performance indicator for the method blank is calculated by:

$$Z_{Blank} = \frac{x}{u(x)}$$

Where:
x = measured blank activity
u(x) = combined standard uncertainty (1 sigma) in the blank measurement

1.2 MB performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. MB performance indicator values should be recorded on a control chart.

2. **Laboratory Control Sample (LCS)**

2.1 The numerical performance indicator for a laboratory control sample is calculated by:

$$Z_{LCS} = \frac{x - c}{\sqrt{u^2(x) + u^2(c)}}$$

Where:

x = Analytical result of the LCS
c = Known concentration of the LCS
$u^2(x)$ = Combined standard uncertainty (1 sigma) of the result squared.
$u^2(c)$ = Combined standard uncertainty (1 sigma) of the LCS value squared.

2.2 LCS performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. Performance indicator values should be recorded on a control chart.

3. Duplicates (DUP)

3.1 These criteria are applicable for the evaluation of the Duplicate, Matrix Spike Duplicate and Laboratory Control Sample Duplicates.

3.2 The numerical performance indicator for laboratory duplicates is calculated by:

$$Z_{Dup} = \frac{x_1 - x_2}{\sqrt{u^2(x_1) + u^2(x_2)}}$$

Where:
x_1, x_2 = two measured activity concentrations
$u^2(x_1)$, $u^2(x_2)$ = the combined standard uncertainty (1 sigma) of each measurement squared.

3.3 Duplicate sample performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. DUP performance indicator values should be recorded on a control chart for each QC sample type (Dup, MSD, LCSD)

4. **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

4.1 The numerical performance indicator for a matrix spike sample is calculated by:

$$Z_{MS} = \frac{x - x_0 - c}{\sqrt{u^2(x) + u^2(x_0) + u^2(c)}}$$

Where:
x = Measured concentration of the spiked sample
x_0 = Measured concentration of the unspiked sample
c = Spike concentration added
$u^2(x)$, $u^2(x_0)$, $u^2(c)$ = the squares of the respective combined standard uncertainties (1 sigma) of these values.

MS performance for all matrices is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. MS performance indicator values should be recorded on a control chart.

ATTACHMENT C-26

SAMPLE MANAGEMENT
PACE, PITTSBURGH

Document Information

Document Number: ENV-SOP-GBUR-0001 **Revision:** 01

Document Title: Sample Management

Department(s): Client Services

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ENV-SOP-GBUR-0001

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Nasreen Derubeis (009976)	Quality Manager II	20 Dec 2018, 05:43:40 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Penelope Westrick (005649)	Manager - Client Services	20 Dec 2018, 02:06:23 PM	Approved

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to outline the procedures involved with the receipt, login, storage, and disposal of samples received by Pace Analytical Services, LLC, (PASL) Pittsburgh.

2. Summary of Method

2.1. Samples are delivered to the laboratory via several delivery mechanisms. Samples received are checked for adherence to the Sample Acceptance Policy with any discrepancies noted. Discrepancies are communicated to the client for their acknowledgement and decision making.

2.2. The Laboratory Information Management System (LIMS, Epic Pro) assigns all samples with a unique sample number and manages the analyses assigned to each sample.

2.3. Samples are labeled with the appropriate information and staged in refrigerated sample storage coolers if temperature preservation is required or on open shelves for samples not requiring sub-ambient temperature preservation. Samples will remain under these conditions until prepared and/or analyzed.

2.4. Samples and associated sub-samples (digestates, extracts, etc.), with the exception of air cans, are maintained for a minimum of 45 days from receipt of samples unless otherwise requested by the client or other regulatory agency.

2.5. Samples are disposed of in accordance with local laboratory regulatory requirements, waste handling procedures and any USDA regulated soil requirements.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP apply to all personnel involved in the receipt, login, storage, and disposal of samples.

3.2. The Sample Acceptance Policy (Attachment I) contains the guidelines for acceptable sample conditions. Any deviation from these guidelines requires detailed documentation within the final test report (as required by 2009 TNI Standard V1M2 Section 5.8.7.2(b)(ii), usually as a footnote, or on the chain-of-custody (COC), Non-Conformance Form (NCF) or Sample Condition Upon Receipt (SCUR) form. Additionally, clients may be notified by email, phone, or other methods..

3.3. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. Not applicable to this SOP.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Samples may be prone to cross-contamination from others within the same delivery group (SDG) or from other client projects. The sample receiving personnel must make every effort to minimize cross-contamination.

6.2. Preservation checks are one of the most likely situations where cross-contamination may occur. Materials used in the process must be specific to each sample and may not used for multiple samples.

6.3. Samples are stored under specific conditions and in specific locations, typically by container type. However, consideration must be given to samples that are uniquely different from others. Samples that

are anticipated to be severely contaminated should be segregated from others in anticipation that the high levels of contaminants may cross-contaminate others in close proximity.

6.3.1. If a sample is identified to be potentially environmentally hazardous by either the client or by sample receiving, the sample(s) are segregated by containing them within a cooler that is clearly marked on the outside with the work order number and the suspected or known contaminant.

6.3.2. When samples arrive at the lab bearing the designation “UN 2910”, they are assumed to contain some level of radioactivity until otherwise determined by the lab by the use of a frisker or by gamma spec analysis. Any sample(s) that are determined to be radioactive are stored on shelving away from other samples that have not yet been analyzed. If a sample is determined to have a very high level of radioactivity, it is stored in a designated area. All such samples with known radioactivity levels are clearly marked with a sticker that shows the universal radioactivity symbol.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Acceptable sample preservation, containers, required volumes for tests completed locally, and hold times are listed in Attachment VI of this SOP. They may also be located in the PASL Quality Assurance Manual, the laboratory’s method SOPs or in the applicable test method. Samples are stored separately from all standards and reagents and any known highly contaminated samples.

7.2. **NOTE**: To avoid contamination, no food or drink products can be located near the areas where samples are unpacked, labeled, or staged.

7.3. Rad Aqueous Samples: Pace-Pittsburgh provides sample containers with the proper preservatives for each test at no additional cost to the clients who submit samples to the laboratory. Clients who subcontract work to Pace-Pittsburgh will be informed of this option when they arrange for sample analysis. For EPA region 4 work, Pace Pittsburgh provides all the sample containers with proper preservatives; therefore a field blank for sample containers is not required.

7.4. Sample Storage: See Section 12.3 for general storage guidelines.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services LLC. Quality Manual, Glossary Section.

8.2. **Chain of Custody (COC):** a form used to record the field identification of samples collected, analyses requested, date and time of collection, sample preservation used, and traceability of samples from time of collection until delivery to the laboratory. This is a legal document.

8.3. **Laboratory Information Management System (LIMS):** a computer system used to manage the flow and traceability of environmental samples and associated data within the laboratory.

8.4. **Matrix:** the bulk characteristics of a sample. See Table 8.1 below.

8.5. **Non-**Conformance Form (NCF): a form used to record the condition of samples received in the laboratory. This form is used with the 08-09-2018 version of the COC.

8.6. **Safety Data Sheet (SDS):** contains information on chemicals used in the laboratory.

8.7. **Sample Custody:** a sample is considered to be in someone’s custody if:

8.7.1. It is in one’s physical possession;

8.7.2. It is in someone’s view, after being in someone’s physical possession;

8.7.3. It is kept in a secured area, restricted to authorized personnel only.

8.8. **SCUR: - Sample Condition Upon Receipt:** a form used to record the condition of samples received in the laboratory. This form is used with client specific COC forms, and the forms that were in place prior to 08-09-2018.

8.9. **Sample Receipt Form (SRF):** form generated by LIMS system after a project is logged in. Contains sample and project information.

8.10. **UN Number:** identification numbers preceded by the letters UN are associated with proper shipping names considered appropriate for international and domestic transportation. These shipping names along with the identification numbers are located in the Federal Register (49CFR172.101).

Table 8.1

NELAC/TNI defined matrix	Corresponding EPIC Pro matrices
Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device.	Air (AR)
Aqueous: any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.	Water (WT)
Biological tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin. Would include tissue and plant samples.	Tissue (TS) or Tissue Dry (TD)
Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined. Includes any non-solid material not classified as waters.	Oil (OL) or Other (OT)
Drinking Water: any aqueous sample that has been designated a potable or potentially potable water source.	Drinking Water (DW)
Non-aqueous liquid: any organic liquid with < 15% settleable solids.	Other (OT)
Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.	Water (WT)- not assigned as a separate matrix.
Solids: includes soils, sediments, sludges and other matrices with > 15% settleable solids.	Solid (SL)
(No corresponding matrix to wipes; wipes would be included in with solids). Includes wipe samples or swabs taken to check for surface contamination	Wipe (WP) or Swab (SW)

9. Equipment and Supplies (Including Computer Hardware and Software)

Table 9.1

Equipment/Supplies	Description/ Comments
Sample Labels	
Thermometers	Infrared, digital, NIST traceable
Sample storage cooling units	Capable of holding required storage temperatures
Chain-of-Custody forms	Pace controlled document or client provided form
Sample Condition Upon Receipt (SCUR) Form	Pace controlled document
Non-Conformance Form (NCF)	Pace controlled document
pH Paper	Range 0-14

Equipment/Supplies	Description/ Comments
Label Printer	
LIMS computer system	EPIC Pro
Disposable pipettes	
Sample containers	Pre-cleaned and certified from an approved vendor

10. Reagents and Standards

10.1. All reagents used in this procedure must be labeled with:

10.1.1. Laboratory reagent identification number;

10.1.2. Unless otherwise noted, the name and concentration of the reagent;

10.1.3. Date the reagent was received, opened and, as needed, prepared;

10.1.4. Person preparing reagent;

10.1.5. Expiration date.

10.2. **Reagents: Table 10.1**

Reagent	Formula	Concentration
Sulfuric Acid	H_2SO_4	1:1
Nitric Acid	HNO_3	1:1
Hydrochloric Acid	HCl	1:1
Sodium Hydroxide	NaOH	50% or Pellets
Sodium Thiosulfate	$Na_2S_2O_3 \cdot 5H_2O$	Pill
Zinc Acetate Solution (for sulfide)	ZnOAc	1:1
Methanol	MeOH	Purge and Trap Grade
Ascorbic Acid (for cyanide)		
Sodium Bisulfate		
a,a,a-Trifluorotoluene	$C_6H_5CF_3$	2.5mg/L

10.3. For acids, bases and other reagents obtained from other laboratory departments, this information is located in the department reagent preparation log. In the event that these reagents are managed within the Sample Receiving group, the department must maintain its own reagent preparation log.

10.4. Some Pace labs use pre-preserved sample containers. In this case, documentation must be maintained for bottleware and preservation traceability.

11. Calibration and Standardization

11.1. Thermometers, IR-guns, and other equipment used for measuring temperatures must be calibrated according to SOP ENV-SOP-GBUR-0041, Support Equipment, or its equivalent revision or replacement.

11.2. Any maintenance to the equipment including calibration and battery changes must be recorded in the appropriate logbook.

12. Procedure

12.1. **Sample Receipt**

12.1.1. The laboratory receives client samples via three major methods: mail/commercial delivery service, PASL courier/field services and hand delivery.

12.1.2. **Courier COC Procedure**: PASL- Pittsburgh uses courier services that pick up client samples on either a regular schedule or on an as-needed basis as communicated by Project Managers or by the client.

12.1.2.1. When the client is present during courier pick-up, the client signs the chain-of-custody (COC) relinquishing custody to the courier. The courier signs the COC as accepting the samples and provides the client with a copy of the COC. When the courier returns to the lab with the client samples, the courier signs the COC as relinquishing the samples to the lab.

12.1.2.2. If the client is not present during courier pick-up, the courier signs the COC as accepting the samples and leaves a copy of the COC for the client. If a client also has a sample log in use, the courier must sign and date the log when the samples are picked up. When the courier returns to the lab with the client samples, the courier signs the COC as relinquishing the samples to the lab. The date/time of delivery to the lab by the courier is the official date/time received by the lab (analogous to the official date/time of receipt by an outside commercial carrier or courier).

12.1.2.3. To ensure the sample security, the PASL courier locks the vehicle at each client pick-up location, and any time the courier is not inside or in direct sight of the vehicle. IMPORTANT: PASL courier/field services personnel must open the sample coolers and verify there is adequate ice in the coolers before transporting or shipping to the laboratory. An exception to this policy would be for coolers already custody-sealed by the client. These coolers are not to be opened except by the receiving lab personnel.

12.1.2.3.1. If no custody seals are present, the courier checks the cooler to see if there is sufficient ice to keep the samples cool until they are returned to the lab. If there is not sufficient ice present, the courier will add ice and indicate by adding a comment to the COC listing the amount of ice that was added at the time of receipt from the client.

12.1.2.3.2. If the client does not have the samples packed into a cooler, the courier will pack the samples into a cooler and add ice if thermal preservation is required. The courier will note that ice was added at the time of receipt from the client, the amount of ice, and the date and time it was added.

12.1.2.4. The lab must provide pertinent information to the person or persons responsible for taking and transporting samples. This information includes sampling procedures (where applicable), and information on storage and transport of samples, including any information on factors that may influence the test results.

12.1.3. **Lab COC Procedures**: The chain of custody (COC) (see example Attachment I) is signed immediately upon receipt of the samples from the client. If the client drops off the samples or they are picked up by the Pace courier, a copy of the signed COC is given to the client at that time. If samples are received via commercial carrier or mail delivery, the COC should be signed immediately when the cooler or package is opened and ultimately placed in the project file. The delivery date and time is considered the date/time received.

12.1.4. **Samples Dropped Off**: Sample receiving personnel must review the COC for any evidence of rush turnaround requests and analyses with short hold times. Projects that fall under these conditions must be given immediate attention. The project manager responsible for that client must be alerted in the event that they have not already alerted the laboratory to the project as it may be possible that the client did not pre-schedule the project. Once the samples are received and logged into the LIMS, the sample technician and project manager will coordinate the notification and delivery of samples to the laboratory.

12.1.4.1. Internal Chain-of-Custody: If the lab uses an internal chain-of-custody (ICOC) procedure, the Project Manager must determine, prior to log-in, which projects require ICOC processing.

12.1.5. **Sample Acceptance Policy:** Copies of this policy must be provided, in the form of a letter, fax, or e-mail to each client or sampler, as necessary. Samples are considered acceptable if they meet the following criteria listed in the Sample Acceptance Policy (See Attachment IIA):

12.1.5.1. There is proper, full, and complete documentation (e.g., chain-of-custody) including:

12.1.5.1.1. Unique client sample identification. Sample containers are labeled using unique client sample identifications (traceable to the chain-of-custody or other documentation) on durable, waterproof labels or equivalent;

12.1.5.1.2. Location of sampling (site), time and date of sample collection;(COC and containers)

12.1.5.1.3. Sampler's name and signature;

12.1.5.1.4. Preservative used (if any);

12.1.5.1.5. Sample type (matrix);

12.1.5.1.6. Requested analyses;

12.1.5.1.7. Any special analysis requirements.

12.1.5.2. Appropriate sample containers have been used;

12.1.5.3. Holding times have not been exceeded upon receipt (holding times are available in Attachment VI and laboratory SOPs). If they have been exceeded, client permission to proceed and documentation in the final report are required;

12.1.5.4. Adequate sample volume has been received for all tests requested (if not, client permission to proceed is required). For data packages requiring quality control samples to be analyzed on client specific samples, the client must submit adequate sample volume to complete the QC samples as well;

12.1.5.5. When there is insufficient sample to complete the QC samples and the client does not wish to send more sample, or more sample cannot be obtained because of sample volume available or holding time issues, the lack of appropriate volume or mass is noted as a sample acceptance policy deviation on the final report. Batch Quality Control samples will be used in place of project-specific Quality Control samples;

12.1.5.6. Samples that require sub-ambient thermal preservation are considered acceptable if they are within ± 2.0 °C of the required temperature (for samples needing to be at 4.0 °C, the acceptable range is just above freezing to 6.0 °C, as defined by NELAC/TNI). Biological tissue samples are considered acceptable if they are received $\leq$ 0°C. The sample cooler (ice chest) temperature is recorded directly on the COC. Samples received outside of this criterion must have a notation on the COC, Non-Conformance Form (NCF) or Sample Condition Upon Receipt (SCUR) form, and qualified in the final report indicating that the temperature was outside of criteria;

12.1.5.6.1. Samples that are delivered to the lab on the same day they are collected may not meet the requirements of section 12.1.5.6. In these cases, the samples shall be considered acceptable if the samples were received on ice. If samples arrive at temperatures that are outside these requirements, the client will be notified and analysis will NOT proceed unless otherwise directed by the client, or if a holdtime is

close to expiration. Data will be appropriately qualified on the final report:

12.1.5.6.2. If sample analysis is begun within fifteen minutes of collection, thermal preservation is not required.

12.1.5.6.3. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen minutes of collection.

12.1.5.7. Data associated with any deviations from the above sample acceptance policy requirements will be appropriately qualified on the final report.

12.1.6. **Measuring temperature when temperature blank present:** Open the cooler and verify the temperature of the samples by taking the temperature of the cooler as described in section 12.1.7. Do not use the temperature blank.

12.1.7. **Measuring temperature when NO temperature blank present**: Open the cooler and verify the temperature of the samples by, measuring the temperature of representative sample bottles using the infrared (IR) thermometer gun. A representative sample will reflect an "average" condition of the containers in the cooler and, depending on the manner in which they are packed, may not necessarily be in direct contact with the cooling material. The temperature should be measured from 2 to 3 representative containers and averaged. If the containers in a cooler are from more than one client site location, the temperature must be measured for 2 to 3 representative containers from each site or group of samples collected at the same location.

12.1.7.1. **Temperature blanks are not acceptable for samples originating from West Virginia.**

12.1.7.2. **The temperature for samples originating from West Virginia must be verified on every container received. Measure the temperature of every container and note any non-compliance on the COC, NCF or SCUR document. The client must be notified and the non-compliance noted in the analytical section of the final report.**

12.1.7.3. NOTE: When an IR gun is used, the temperature should be measured on an opaque surface such as the bottle label. Measurements taken through a transparent surface (clear or amber glass) may not be reliable and should incorporate a specific temperature correction factor for that surface reading.

12.1.8. Record the uncorrected and corrected cooler temperatures temperature on the COC (example in Attachment I) and/or SCUR (example in Attachment II). In addition, record the type of "ice" used for packing the cooler (e.g., wet ice, "blue ice", gel packs, etc.) on the SCUR.

12.1.9. If samples within a project are spread over multiple coolers and one or more of the coolers are outside of the temperature criteria, then the contents of the cooler must be itemized and the samples and sample containers must be listed on a Cooler Temperature Breakdown form (Attachment IIIA) or NCF or SCUR. Sample containers affected by an out of control temperature must be included as a qualifier in the final report. This itemization must be retained in the project file for future reference.

12.1.10. Unpack the cooler and chain of custody (COC). Organize the samples, grouped by client sample ID, according to the order on the COC. Review COC against samples to make sure the bottles received match the analysis requested. All anomalies must be recorded on the COC, NCF or SCUR form and must be transferred to data qualifiers for the final report.

12.1.10.1. If a cooler will not be unpacked the same day that it is received, at a minimum the temperature of the samples as well as chemical preservation of the sample(s) (if required) must be checked and recorded on the NCF or SCUR. Also, the

presence/absence of ice, packaging material, custody seals, and method of delivery (e.g., Client, Courier, etc.) must be recorded on the COC, NCF or SCUR and must be transferred to data qualifiers on the final report.

12.1.10.2. Discard any ice or water that remains in the cooler and the packing material used to secure the samples. Water or ice should be discarded down a drain that connects to the local sewer. Packing materials should be placed in the garbage. If a sample container has broken, the contents remaining in the cooler MUST be discarded in a manner consistent with the hazardous waste handling standard operating procedure.

12.2. **Screening Rad Aqueous Samples**

12.2.1. Measure the emission rate of aqueous samples being tested for radiochemistry at the time of receipt when samples are unpacked from the cooler.

12.2.2. Using a MicroRem Doserate Meter, set the meter dial to X 0.1.

12.2.3. Pass the meter over or in front of the capped sample containers on any side.

12.2.4. If the meter needle reaches the limit, turn the dial to X1 and rescan the samples.

12.2.5. Document that the samples were scanned on the COC or SCUR. If any sample measures to be greater than 0.5 mrem/hr, place the sample on the shelf dedicated for the samples greater than 0.5 mrem/hr and notify the laboratory RSO or designee. Document on the NCF or SCUR.

12.3. **pH and Residual Chlorine Verification Instructions**

12.3.1. **Preservation must be checked for all sample containers to confirm that each container was correctly chemically preserved or correctly not chemically preserved.**

12.3.2. The pH of all sample bottles must be measured during sample receipt and check in. (see exceptions below in section 12.3.4).

12.3.3. The lot number of the pH paper used must be recorded on the NCF or SCUR in the box marked pH paper lot #.

12.3.4. Open each bottle (except as noted below in section 12.3.4). Do not dip the pH paper into the sample bottle or lid. Use a new disposable pipette, a stirring bar or another inert utensil to withdraw a small portion of the sample. Dispense the aliquot on a sample specific narrow-range pH strip by holding the pH strip over a waste container and allowing the sample to be dispensed on the paper. The sample is not to be allowed to re-enter the sample container after it comes in contact with the pH paper. Verify that the pH meets the requirements for that container type.

12.3.5. NOTE: Do not check the pH of samples for coliform, volatiles, TOC, WI-DRO (Wisconsin), oil and grease, phenolics, or hexane extractables (Method 1664A). These analyses will be checked by the analyst at the bench prior to or following analysis, and should not be opened by sample management personnel.

Table 12.3 – General pH Preservation Requirements by Preservative

Sample Preservatives	Sample pH Requirement
Hydrochloric Acid (HCl)	must be <2
Nitric Acid ($HN0_3$)	must be <2
Sulfuric Acid (H_2SO_4)	must be <2
Sodium Hydroxide (NaOH)	must be >12
Zinc Acetate & Sodium Hydroxide (NaOH)	must be >9
Unpreserved Sample	Unpreserved

12.3.6. If the pH is not within the required range, indicate the anomaly on the NCF, SCUR and/or on the COC and include the anomaly in a data qualifier on the final report. If there is any indication of a problem with a sample's preservation, the client must be contacted for instructions on how to proceed, and the laboratory must keep a record of this as per 2009 TNI standard V1M2 Section 5.8.3. In some cases the client may indicate to proceed despite the deviation, but the results must always be clearly qualified in the final report.

12.3.7. If an unpreserved sample appears to be preserved, the client must be contacted before proceeding with any analyses. The client can resample or instruct the lab to proceed with analysis. The lab must not adjust the pH of the sample. If the lab proceeds with analysis, this deviation must be documented and results qualified appropriately.

12.3.8. If a sample container does not meet the pH preservation required, the pH of the sample must be recorded on the NCF, COC or SCUR, and the anomaly must be reported as a data qualifier in the final report. Contact the Project Manager so that they can contact the client to verify if preservation should be completed at the lab. Additional preservative is added so that the preservative content is <1% of the sample container volume. The sample is mixed and the pH is measured again. The new pH reading is also recorded on the NCF, COC or SCUR along with the amount, type and lot number of the preservative added. In addition, the sample container is marked with the preservative added, volume added, date, time and initials of the technician.

12.3.9. Water samples received for radiochemistry testing by EPA 900 series methods must be preserved within 5 days of collection and held for 24 hours prior to analysis if not preserved in the field during sample collection.

12.3.10. **pH Preservation Adjustments:** Document on the NCF or SCUR if the pH preservation requirements are or are not met. Document that all containers have been checked, and that all containers are in compliance with EPA recommendation. If a sample container does not meet the pH preservation requirement, the pH of the sample must be recorded on the NCF or SCUR. Additional preservative is added so that the preservative content is < 1% of the sample container volume. For example:

A. For a 100mL container, a maximum of 1mL of preservative may be added;

B. For a 250mL container, a maximum of 2.5mL of preservative may be added;

C. For a 500mL container, a maximum of 5mL of preservative may be added;

D. For a 1L container, a maximum of 10mL of preservative may be added

12.3.10.1. The appropriate preservative is added to the sample container, the sample is mixed and the pH is taken again. The new pH reading is also recorded on the COC, NCF or SCUR along with the amount, type and lot number of the preservative added. In addition, the sample container is marked with the preservative added, volume added, date, time and initials of the technician.

12.3.10.2. For Metals analyses specifically, the lab must wait 24 hours after pH adjustment to pH < 2 before sample preparation can begin.

12.3.11. **Total Residual Chlorine Verification** – Total residual chlorine must be verified at the time of receipt or at the bench as required by the method or individual state regulatory agency for certain analyses (see Table 12.3). Sample receipt personnel must only check the sample bottles listed as YES in the check at receipt column.

12.3.11.1. Open the appropriate sample container. Utilizing a new disposable pipette, withdraw a 10 ml (approximate) portion of the sample and transfer it to a small clean container. Dispense an aliquot of DPD reagent into the aliquot of sample. The presence of residual chlorine is indicated by the presence of any pink color. The use of chlorine test strips is prohibited.

12.3.11.2. If any chlorine is detected, regardless of the amount, note the information on the NCF or SCUR, in the final report, and on the container.

12.3.11.3. Notify the laboratory department performing the scheduled test that the container needs to be dechlorinated as soon as practical.

Table 12.3A Analyses Requiring Residual Chlorine Verification

Analyses	Check at Receipt
Ammonia (NH3)	NO
Nitrate (NO3)	NO
Biochemical Oxygen Demand (BOD)	NO
Cyanides	NO
TKN	NO
Dioxin 1613B	NO
DRO by 8015	NO
EDB/DBCP by 8011	NO
PBDE 1614	NO
PCB's 1668A	NO
Volatile Organics (624) (8260) (GRO)	NO
Extractable organics (608) (625) (8081) (8082) (8270)	YES

12.3.12. Note any discrepancies pertaining to samples as defined by the sample acceptance policy detailed above on the COC, NCF or SCUR, and as a qualifier in the final report. Any discrepancies involving temperature, preservation, hold time, collection dates and times, sample volume, sample containers, and unclear requested analyses, must be reported to Project Management as soon as possible.

12.3.13. **Checking for Sulfide in Cyanide analyses:** Testing for sulfide by using the lead acetate paper is done in by the Wet Chemistry department.. Darkening of the paper indicates the presence of sulfide. Refer to laboratory's cyanide SOP ENV-SOP-GBUR-0137.

12.3.14. For short hold samples, the laboratory is notified and the samples are staged per section 12.1.4.

Table 12.3B – Analyses with Hold Times Less Than 72 Hours

Short Hold Time	Analyses	Details
15 minutes	Field Parameters	pH, Dissolved Oxygen, Residual Chlorine, ferrous iron
6 Hours/2 Hours	Total / Fecal Coliform (MPN, MF)	Must be received at lab within 6 hours of sampling. Analysis must begin within 2 hours of receipt.
24 Hours	Hexavalent Chromium	Aqueous Samples Only Field filtered within 15 minutes of collection
30 Hours	Total Coliform (Presence / Absence)	
48 Hours	Color	
48 Hours	MBAS	

Short Hold Time	Analyses	Details
48 Hours	Nitrate (unpreserved)	If Preserved, reported as NO_3+NO_2
48 Hours	Nitrite (unpreserved)	If Preserved, reported as NO_3+NO_2
48 Hours	Ortho –phosphate	Field filtered within 15 minutes of collection
48 Hours	Settable Solids	
48 Hours	Turbidity	
48 Hours	VOA - Soils by Unpreserved EPA5035	Jars, Encores®, Sleeves
48 Hours	Gross Alpha (NJ 48hr method)-waters	EPA NJAC 7:18-6
72 Hours	3030C Metals	
96 Hours	Radon	This parameter is included because travel time to the laboratory shortens the time for analysis to 72 hours.

12.4. **Sample Login**

12.4.1. All samples received by the laboratory must be logged into the LIMS. Rush projects and/or projects with short holds should be logged in first. After these projects have been addressed, projects should be addressed on a first in, first out basis. See table 12.4 for short hold tests.

12.4.2. Samples must be logged into the LIMS so the samples can be uniquely identified (Lab sample identification numbers). These lab sample ID numbers are used to track the prep and analysis activities of the samples, as well as identify the sub-samples, digestates, extracts, and other sample byproducts. This laboratory code maintains an unequivocal link with the unique client field sample ID code assigned to each sample.

12.4.3. Samples are logged into LIMS by using the associated profile and client information provided on the COC or by the Project Manager. Detailed information on the login process in EPIC-PRO can be found in Module 03: Epic Pro - Login Guide.

12.4.4. All samples are logged in with the client ID, collection date and time, received date and time and analysis requested. If a client does not provide a collection date the field is left blank. The project manager will contact the client to confirm a collection date as soon as possible. If the client provides a date but no time, the receiving personnel should enter 00:01 as the most conservative collection time.

12.4.5. For parameters with hold times measured in hours, identification of samples that were collected outside of the receiving laboratory's time zone must be qualified in LIMS and recorded on the NCF or SCUR. This information is used to ensure that hold times are met and that time zone information is taken into consideration.

12.4.6. Each sample is assigned one or more line items of a profile to assign tests to the sample within the LIMS system.

12.4.7. Generate sample labels and Sample Receipt Form (SRF) (see Attachment III).

12.4.8. Review the NCF or SCUR for errors and omissions, and initial the top right box of the SCUR or the bottom right box of the NCF

12.4.8.1. Create the SRF by selecting Systems, Submit Job. Enter the job type LLSRF Login Labels and SRF. Type the requested workorder ID into the Value box for Workorder. Select F10 or the save icon at the top of the screen to send the pdf to the Horizon system for archival. This will also print the sample labels and folder labels at the same time.

12.4.9. Attach the sample labels to the appropriate sample bottles.

12.4.10. Attach the bar code label to the chain of custody..

12.4.11. When labeling the samples, check to see that the IDs, date and time on the labels match the sample bottles. If there is a discrepancy, contact the person logging in the project or the appropriate project manager.

12.4.12. Initial the top right box of the SCUR or the bottom right box of the NCF to indicate the person placing the labels on the bottles.

12.4.13. Scan the chain of custody and NCF or SCUR to the network drive.

12.4.14. The Project Manager must review and verify the following information by comparing the COC to SRF. Some of this information may not be provided by the client and those fields should be left blank:

- Report Recipient,
- Invoice Recipient,
- Additional Report Recipient,
- PO#,
- Project Name,
- Project Number,
- Requested Due Date,
- Sample ID,
- Matrix,
- Collection Date & Time,
- Received Date & Time,
- Analysis: Double check compound lists,
- Price,
- Region Codes,
- Work Region % Split (for Pace internal subcontracted work),
- Has subcontracted work been shipped.
- Containers and preservation

12.4.15. If any samples require analyses performed outside of the laboratory, prepare the samples for subcontracting according to the procedures listed in the SOP describing the subcontracting of analytical services, ENV-SOP-GBUR-0002, Subcontracting Samples and SOP ENV-SOP-GBUR-0050, Evaluation and Qualification of Vendors.

12.5. **Sample Storage**

12.5.1. While awaiting login on the day received, samples may remain in the shipping cooler as received prior to login. Once unpacked, samples will be logged into the LIMS in a timely manner and returned to appropriate storage conditions as soon as possible. For the exceptional case where samples are not logged in the day they were received, they must be stored under appropriate temperature-controlled conditions until login takes place. In all cases, the same temperatures must be taken as soon after receipt as possible and the samples stored so as to maintain the required storage conditions while awaiting log-in.

12.5.2. Once logged into the LIMS and labeled, samples are placed in the appropriate storage areas. Specific temperature requirements are outlined in the analytical methods, but general guidelines are outlined below:

12.5.3. Biological tissue samples are staged by receiving date or project number on shelves in a freezer for all types of analyses.

12.5.4. Summa® canisters and Tedlar® bags are stored on designated shelving at ambient temperature.

12.5.5. Volatiles: Aqueous samples are stored by receiving date or by project number in a segregated volatiles cooler. Associated trip blanks are stored with the samples.

12.5.6. Volatiles: Soil and other solid samples received preserved in methanol are stored by receiving date or by project number in a segregated volatile cooler. Associated trip blanks are stored with the samples.

12.5.7. Volatiles: Soil and other solid samples received preserved with a stir bar, or deionized water and a stir bar, are stored by receiving date or by project number in a segregated volatiles freezer. Associated trip blanks are stored with samples.

12.5.8. Volatiles: Soil and other solid samples received in 4oz containers or similar bottleware must be preserved within 48 hours. In order to preserve these samples, it is necessary to collect a 5g aliquot of the sample and transfer it to a 40mL vial. One of the following preservation options must be utilized:

12.5.8.1. The 5g aliquot is preserved with a stir bar, 10 mL of deionized (DI) water and a stir bar, or 10 mL of sodium bisulfate and a stir bar and stored in a freezer until analysis, or;

12.5.8.2. Within 48 hours of collection in the field, the 5g aliquot must be immediately extracted with 5mL of methanol and stored in a segregated volatiles cooler until analysis, or;

12.5.8.3. Within 48 hours of collection in the field, the 5g aliquot can be preserved with 10mL of deionized water and a stir bar, stored in a segregated volatile cooler and analyzed within 48 hours of collection.

12.5.9. Volatiles: Soil and other solid samples received in Encore samplers should be managed within 48 hours of collection by freezing the Encore or extruding it into a 4 or 8oz jar.

12.5.9.1. If extruding the sample into a 40mL vial containing a stir bar or a stir bar and 10mL of deionized water, then the sample is stored in the segregated volatile freezer until analysis.

12.5.9.2. If extruding the sample into methanol, then the sample is extracted within 48 hours of collection and the sample is stored in a segregated volatile cooler until analysis.

12.5.10. NOTE: If samples are not received within 48 hours of collection or are not received with enough time to process the samples correctly within 48 hours of collection, this must be noted in a way that will be visible on the final report (e.g., footnote in LIMS).

12.5.11. General Chemistry/Semi-volatiles: Waters and other liquid samples are staged by receiving date or by project number on the shelves in the appropriate sample storage cooler.

12.5.12. General Chemistry/Semi-volatiles: Soils and other solid samples are staged by receiving date or by project number on the shelves in the appropriate sample storage cooler.

12.5.13. Metals Solids and Liquids: These samples are staged by receiving date or by project number on designated shelving in the laboratory or appropriate designated area. These samples may be stored at ambient temperature unless Mercury or Hexavalent Chromium analysis is needed. If Mercury or Hexavalent Chromium analysis will be performed, the samples are staged by receiving date or by project number in the appropriate sample storage cooler.

12.5.14. Internal Chain-of-Custody (if required by the client): When an analyst removes samples from a storage unit, the ICOC form must be completed in the applicable ICOC logbook. The following items must be documented: lab sample ID, analyst initials, date and time samples are removed, and sample container type. The project number is optional and only necessary when it is needed to uniquely identify a specific sample container. Once the analyst is finished with the sample, the sample must be returned to the applicable storage unit. The analyst must again document the necessary information in the ICOC logbook (date and time samples are returned to the storage unit and the analyst's initials). If the sample was entirely consumed, then document with the appropriate comment code.

12.5.15. Internal Chain-of-Custody (if required by the client): Similar steps must be taken for sample by-products such as extracts, digestates, and leachates. Once a sample is prepared for analysis, sample custody of the sample by-product must be transferred to the appropriate analytical group sample storage unit. Analytical staff must document in their ICOC logbook when removing and returning the sample by-products from and to the analytical sample storage location. If the sample by-product is entirely consumed during analysis, then document with the appropriate comment code.

12.6. **Sample Retention and Disposal**

12.6.1. Unused portions of samples are retained by the laboratory based on the program or customer requirements for retention and storage. The minimum sample retention time is 45 days from sample receipt. Samples requiring thermal preservation may be stored at ambient temperature when the hold time has expired; the report has been delivered, and/or allowed by the customer, program or contract. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

12.6.2. If samples must be returned to customers, the lab must take special care to ensure that the samples are not damaged during any handling, testing, storing, or transporting processes.

12.6.3. Disposal of Unconsumed Samples: Refer to the laboratory standard operating procedure for waste handling and disposal (ENV-SOP-GBUR-0006).

13. Quality Control

13.1. For any sample received at the laboratory that does not meet the sample acceptance, hold time or preservation criteria, the client must be contacted by project management and advised of the situation.

13.2. If the client instructs the laboratory to proceed with the analysis, all appropriate personnel/departments must be informed and the client approval must be documented on the NCF, SCUR or COC or saved to an electronic file in e-reports. Data will be appropriately qualified in the final report.

13.3. The client may also instruct the laboratory to preserve the samples at the laboratory prior to proceeding with analysis. This must be documented on the COC, NCF or the SCUR or saved to an electronic file in e-reports, and must be qualified in the final laboratory report.

13.4. All supporting documentation related to sample custody must be retained by the laboratory. This includes; memoranda, fax transmissions, all paperwork received with the COC and copies of email transmissions. Documenting Discrepancies during receipt of samples: The following are examples of client discrepancies that must be qualified in the final report.

13.4.1. Lost samples/insufficient sample volume,

13.4.2. Broken or missing bottles,

13.4.3. Missing COC,

13.4.4. Mislabeled bottles,

13.4.5. Preservation error,

13.4.6. Missing sample related details (date, time, sample type).

13.5. PASL sample management discrepancies will be documented on the SCUR form, NCF form, COC or within the project files and noted on the final report. Discrepancies attributable to errors and omissions on the part of the laboratory will be addressed and resolved through the lab's corrective action process.

14 Data Analysis and Calculations

14.1 Not applicable to this SOP.

15 Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 Not applicable to this SOP.

16 Corrective Actions for Out-of-Control Data

16.1 Not applicable to this SOP.

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 Not applicable to this SOP.

18 Method Performance

18.1 All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

19 Method Modifications

19.1 Not applicable to this SOP.

20 Instrument/Equipment Maintenance

20.1 Not applicable to this SOP.

21 Troubleshooting

21.1 Not applicable to this SOP.

22 Safety

22.1 Hazards and Precautions: Use extreme caution in handling samples and wastes as they may be hazardous. Each reagent and chemical used in this method should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats, safety glasses, and ventilation hoods. Safety Data Sheets (SDS) are on file and available to all personnel.

22.2 All personnel involved in sample management are responsible for complying with OSHA and DOT regulations. These regulations pertain to the safe handling and/or shipping of the chemicals specified in this procedure. Refer to the Sample Receiving Supervisor for any questions or concerns related to the safe handling and shipment of hazardous materials.

22.3 Other laboratory safety requirements are contained in the Chemical Hygiene Plan/Safety Manual. Immediate questions can also be addressed with the local Safety Officer.

23 Waste Management

23.1 Refer to the laboratory standard operating procedure for waste handling and disposal (ENV-SOP-GBUR-0006).

24 Pollution Prevention

24.1 Refer to Pace Safety Manual, COR-POL-0021.

25 References

25.1 Department of Defense (DoD) Quality Systems Manual - current approved version.

25.2 SW-846, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA, current revision.

25.3 American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1995, Standard Methods for the Examination of Water and Wastewater, A.E. Greenberg, L.W. Clesceri, A.D. Eaton and M.A.H. Franson, eds., 19th ed., American Public Health Association, Washington D.C.

25.4 U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

25.5 U.S. Environmental Protection Agency, 1988, Methods for Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

25.6 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (current approved revision).

25.7 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current approved version.

25.8 Pace Analytical Services, LLC. - Pittsburgh Laboratory Quality Assurance Manual, current version.

25.9 SOP ENV-SOP-GBUR-0041, Support Equipment, current version.

14.1 SOP ENV-SOP-GBUR-0047, Laboratory Equipment, current version.

14.2 SOP ENV-SOP-GBUR-0049, Internal and External Audits, current version.

14.3 SOP ENV-SOP-GBUR-0048, Corrective and Preventative Action, current version.

14.4 SOP ENV-SOP-GBUR-0059, Training, current version.

14.5 SOP ENV-SOP-CORQ-0007, Lab Track, current version.

15 Tables, Diagrams, Flowcharts, and Validation Data

15.1 Attachment I – Example Chain of Custody Form.

15.2 Attachment II – Example Sample Condition Upon Request (SCUR) Form and Sample Receiving Non-Conformance Form (NCF)

15.3 Attachment IIA - Sample Acceptance Policy

15.4 Attachment III – Example Sample Receipt Form (SRF)

15.5 Attachment IIIA - Example Cooler Temperature Breakdown

15.6 Attachment IV - Epic Container Code Key

15.7 Attachment V - Sample Receipt Form - Epic Container Codes

15.8 Attachment VI Tests, Sample Containers and Required Volumes

14 Revisions

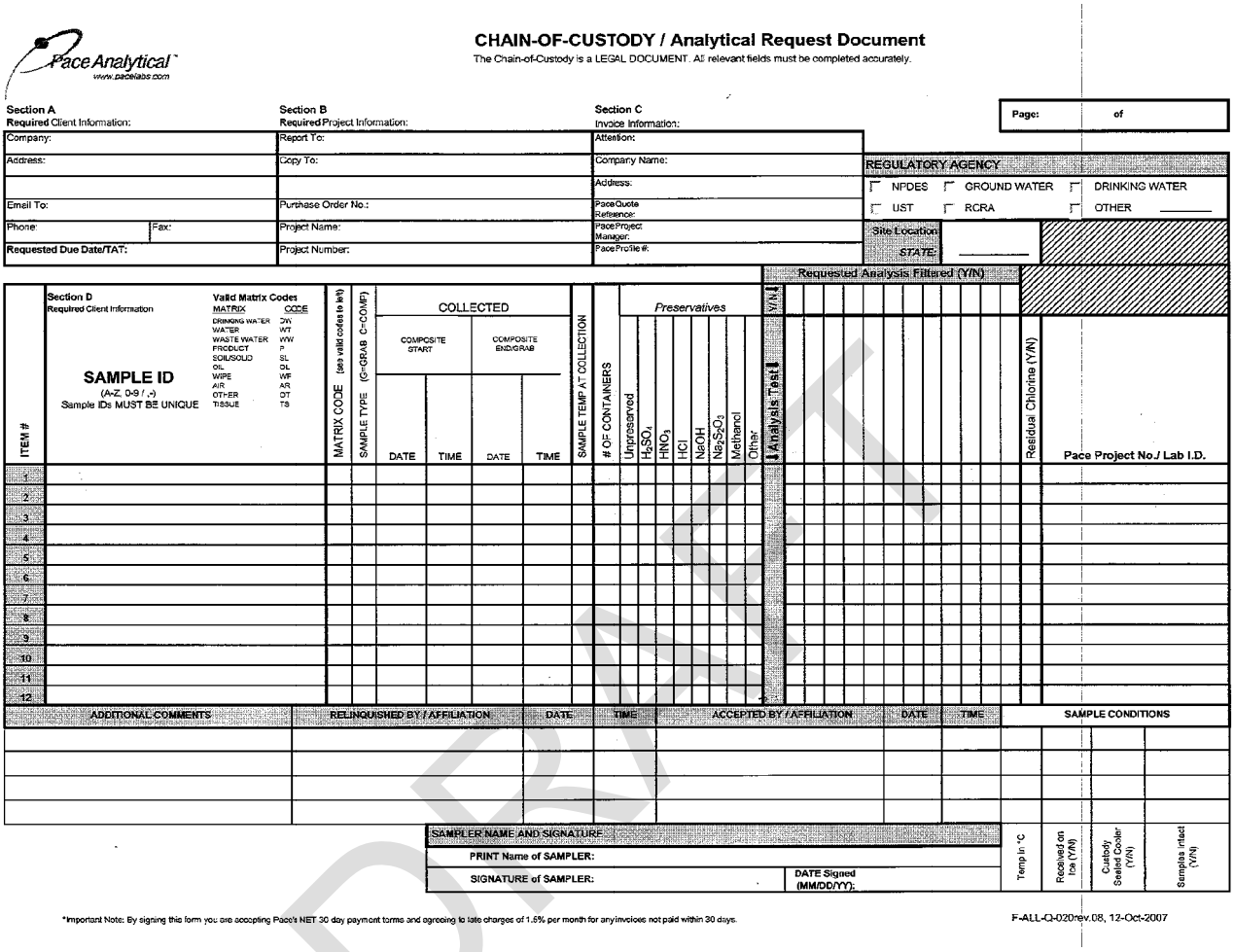
Document Number	Reason for Change	Date
PGH-C-001-4	1. Updated SOP to the new corporate template. Changes to the template are indicated above.	02Apr2013
PGH-C-001-5	1. Added to section 3.2: Any deviation from these guidelines requires detailed documentation within the final test report (as required by 2009 TNI Standard V1M2, Section 5.8.7.2(b) (ii). Additionally, clients may be notified by email, phone, or other methods. 2. Added throughout the SOP that samples received outside the sample acceptance policy are qualified in the final report. 3. Edited for grammar and spelling. 4. Added SOP references.	15Jul2014
PGH-C-001-6	1. Section 12.4 & 12.5 added if required by the client. 2. Updated section 12.6.1 to add sample retention time.	25Nov2015

Document Number	Reason for Change	Date
PGH-C-001-7	1. Section 12.3.3 NOTE: add phenolics. 2. Remove section 12.1.12 and 12.2 Total Residual Chlorine Verification Instructions and Table. 3. Renumber 12.2 thru 12.6. 4. Revised sections 12.4.8 - 12.4.14 to match the current terminology within LIMS, remove printing references. 5. Add 12.4.10. 6. Add 12.4.13. 7. Inserted revised SCUR Attachment II. Updated SCUR to add documentation of Rad aqueous scanning. 8. Add 7.3 Radiochemistry aqueous field blank. 9. Example Cooler Temperature Breakdown Form, C036-1 2June2016 added. 10. The SOP was renumbered through some sections. 11. Updated SOP references.	26Jun2016
PGH-C-001-8	1. Section 7.1 inserted required volumes for tests completed locally. 2. Section 7.3 revised wishes to prefers. 3. Table 9.1 – added Range 0-14. 4. Table 10.2 added ZnOAc. 5. Section 12.1.6 added If the containers in a cooler are from more than one client site location, the temperature must be measured for 2 to 3 representative containers from each site. 6. Add 12.3.1 Preservation must be checked on all sample. containers (see exceptions below). 7. Renumbered 12.3.2 through 12.3.9. 8. Table 12.2 – add Unpreserved samples to be checked. 9. 12.4.14 add Containers and Preservatives. 10. Added: Attachment VIII, Tests, Sample Containers and Required Volumes. 11. Updated the SCUR.	06Dec2016
PGH-C-001-9	1. Add sections 12.1.6.1 and 12.1.6.2 for specific temperature measurement requirements. 2. Renumber and add section 12.3.9 Total Residual Chlorine Verification. 3. Add Table 12.3 Analyses Requiring Residual Chlorine Verification. 4. Added an updated Attachment II.	27Dec2016
PGH-C-001-10	1. Updated and clarified the language in Section 12.3. 2. Corrected section 12.3.2: The pH of <u>all sample</u> bottles must be measured during sample receipt and check in. (see exceptions below in section 12.3.4).	18April2017

Document Number	Reason for Change	Date
PGH-C-001-11	1. Updated to SOT-All-C-001-rev.06. 2. Section 2.5 added USDA regulated soil reference. 3. Section 3.2 added reference to sample acceptance policy, removed TNI reference 4. Section 7.3, updated header to Rad Aqueous samples. Specified for EPA region 4 work, Pace Pittsburgh provides all the sample containers with proper preservatives, therefore a field blank for sample containers is not required. 5. Updated table 8.1 6. Section 12.1.2 added title Courier COC Procedure. 7. Section 12.1.2.3 added: An exception to this policy would be for coolers already custody-sealed by the client. These coolers are not to be opened except by the receiving lab personnel. 8. Added title Lab COC Procedures to section 12.1.3. 9. Added title Samples Dropped off to section 12.14. 10. Removed Table 12.1 and added to section 12.3.13. 11. Corrected section numbers in section 12.1.5.6.1-12.1.5.6.3. 12. Added header title to sections 12.1.6 and 12.1.7. 13. Added to section 12.1.8, recorded uncorrected and corrected temperature. 14. Updated section 12.1.9, use Above Cooler temperature breakdown form or SCUR. 15. Added section 12.3.9, pH Preservation Adjustments. 16. Added section 12.3.13, checking for sulfide in cyanide analyses, done in the Wet Chem department. 17. Added Attachment IIA, Sample Acceptance Policy. 18. Added the updated SCUR form.	19Sep2017
PGH-C-001-12	1. Updated section 12.1.6, do not use the temp blank to take the temperature of the cooler. Referred this section to section 12.1.7 2. Updated section 12.1.7 to use the IR gun to take the temperature of the cooler. Updated the sub sections numbering. 3. Removed references to using jacketed thermometer. Lab always uses IR gun.	10Oct2017
S-PGH-C-001-rev.13	Clarified section 12.3.9: Document on the SCUR if the pH preservation requirements are or are not met. Document that all containers have been checked, and that all containers are in compliance with EPA recommendation. If a sample container does not meet the pH preservation requirement, the pH of the sample must be recorded on the SCUR.	22Dec2017
S-PGH-C-001-rev.14	1. Section 12.3.3 updated to include documentation of the pH paper lot number on the SCUR. 2. SCUR updated to include space to document the pH lot number.	01Mar2018

Document Number	Reason for Change	Date
ENV-SOP-GBUR-0001 Rev.01	1. Updated all references to the SCUR document to include a reference to the Pace Non-Conformance Form (NCF). 2. Added definition for Non-Conformance Form and descriptions of when the SCUR and NCR are used. 3. 7.3 correct punctuation after preservatives. 4. Added NCF to Equipment and Supplies Table 9.1 5. 12.4.8 and 12.4.12, added "the bottom right box of the NCF" 6. 12.4.15 added Subcontracting Samples SOP reference.	20Dec2018

Attachment I – Example Chain-of-Custody Form



Pace Analytical
www.pacelabs.com

CHAIN-OF-CUSTODY / Analytical Request Document
The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Page: of

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:
Company:	Report To:	Attention:
Address:	Copy To:	Company Name:
		Address:
Email To:	Purchase Order No.:	Pace Quote Reference:
Phone: Fax:	Project Name:	Pace Project Manager:
Requested Due Date/TAT:	Project Number:	Pace Profile #:

REGULATORY AGENCY
NPDES GROUND WATER DRINKING WATER
UST RCRA OTHER
Site Location STATE:

Requested Analysis Filtered (Y/N)

ITEM #	Section D Required Client Information: SAMPLE ID (A-Z, 0-9 / , -) Sample IDs MUST BE UNIQUE	Valid Matrix Codes: MATRIX / CODE — DRINKING WATER DW; WATER WT; WASTE WATER WW; PRODUCT P; SOIL/SOLID SL; OIL OL; WIPE WP; AIR AR; OTHER OT; TISSUE TS	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test Y/N	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				
9																				
10																				
11																				
12																				

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS

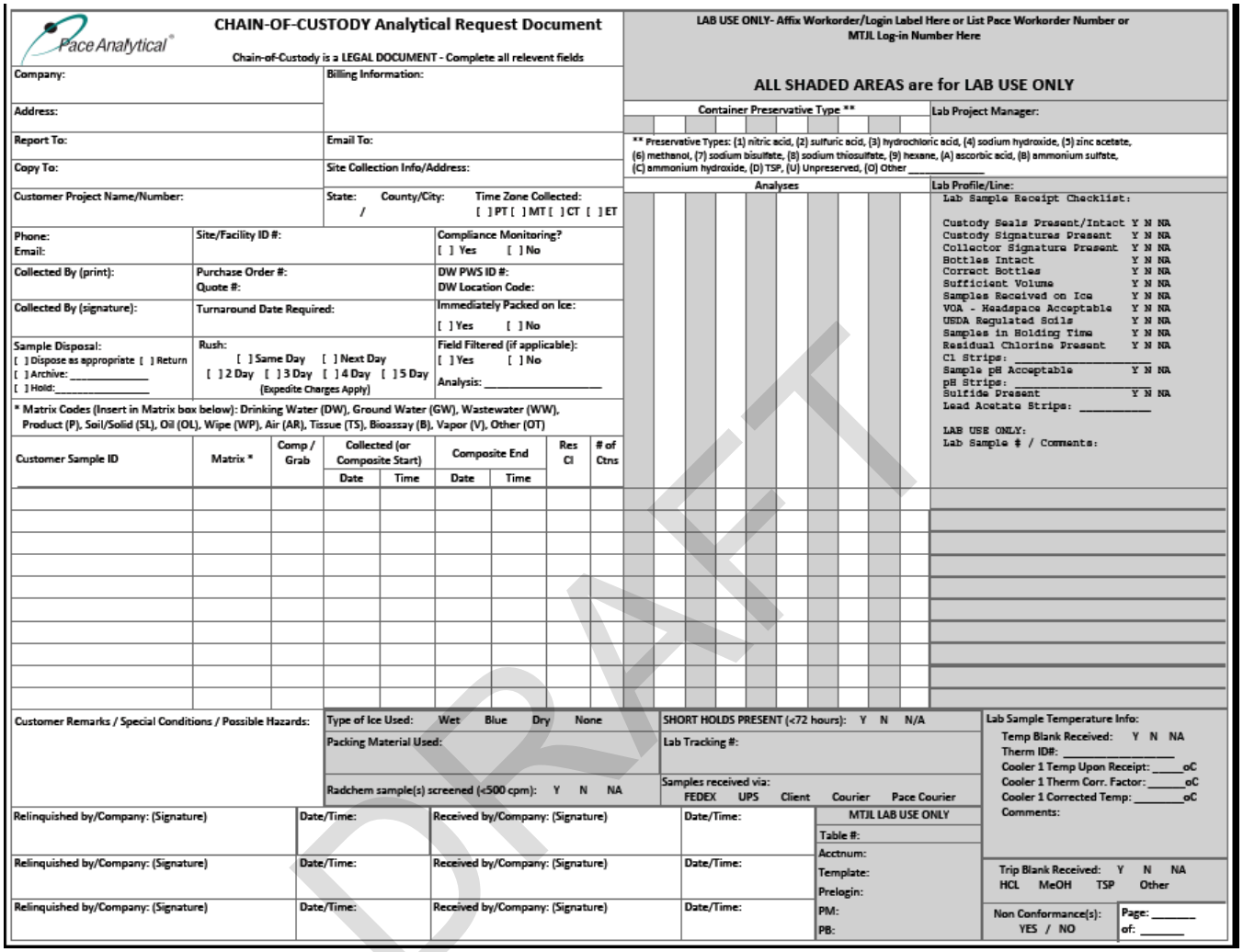
SAMPLER NAME AND SIGNATURE
PRINT Name of SAMPLER:
SIGNATURE of SAMPLER: DATE Signed (MM/DD/YY):

Temp in °C | Received on Ice (Y/N) | Custody Sealed Cooler (Y/N) | Samples Intact (Y/N)

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

F-ALL-Q-020rev.08, 12-Oct-2007

Attachment I – Example Pace Analytical Services LLC Chain of Custody 08-09-2018



Pace Analytical®

CHAIN-OF-CUSTODY Analytical Request Document

Chain-of-Custody is a LEGAL DOCUMENT - Complete all relevent fields

LAB USE ONLY- Affix Workorder/Login Label Here or List Pace Workorder Number or MTJL Log-in Number Here

ALL SHADED AREAS are for LAB USE ONLY

Company: | Billing Information:

Address:

Report To: | Email To:

Copy To: | Site Collection Info/Address:

Customer Project Name/Number: | State: County/City: Time Zone Collected: [ ] PT [ ] MT [ ] CT [ ] ET

Phone: Email: | Site/Facility ID #: | Compliance Monitoring? [ ] Yes [ ] No

Collected By (print): | Purchase Order #: Quote #: | DW PWS ID #: DW Location Code:

Collected By (signature): | Turnaround Date Required: | Immediately Packed on Ice: [ ] Yes [ ] No

Sample Disposal: [ ] Dispose as appropriate [ ] Return [ ] Archive: _____ [ ] Hold: _____ | Rush: [ ] Same Day [ ] Next Day [ ] 2 Day [ ] 3 Day [ ] 4 Day [ ] 5 Day (Expedite Charges Apply) | Field Filtered (if applicable): [ ] Yes [ ] No Analysis: _____

* Matrix Codes (Insert in Matrix box below): Drinking Water (DW), Ground Water (GW), Wastewater (WW), Product (P), Soil/Solid (SL), Oil (OL), Wipe (WP), Air (AR), Tissue (TS), Bioassay (B), Vapor (V), Other (OT)

Customer Sample ID	Matrix *	Comp / Grab	Collected (or Composite Start) Date	Collected (or Composite Start) Time	Composite End Date	Composite End Time	Res Cl	# of Ctns

Container Preservative Type ** | Lab Project Manager:

** Preservative Types: (1) nitric acid, (2) sulfuric acid, (3) hydrochloric acid, (4) sodium hydroxide, (5) zinc acetate, (6) methanol, (7) sodium bisulfate, (8) sodium thiosulfate, (9) hexane, (A) ascorbic acid, (B) ammonium sulfate, (C) ammonium hydroxide, (D) TSP, (U) Unpreserved, (O) Other _____

Analyses

Lab Profile/Line:

Lab Sample Receipt Checklist:

Custody Seals Present/Intact	Y N NA
Custody Signatures Present	Y N NA
Collector Signature Present	Y N NA
Bottles Intact	Y N NA
Correct Bottles	Y N NA
Sufficient Volume	Y N NA
Samples Received on Ice	Y N NA
VOA - Headspace Acceptable	Y N NA
USDA Regulated Soils	Y N NA
Samples in Holding Time	Y N NA
Residual Chlorine Present	Y N NA
Cl Strips: _____	
Sample pH Acceptable	Y N NA
pH Strips: _____	
Sulfide Present	Y N NA
Lead Acetate Strips: _____	

LAB USE ONLY:
Lab Sample # / Comments:

Customer Remarks / Special Conditions / Possible Hazards:

Type of Ice Used: Wet Blue Dry None | SHORT HOLDS PRESENT (<72 hours): Y N N/A

Packing Material Used: | Lab Tracking #:

Radchem sample(s) screened (<500 cpm): Y N NA | Samples received via: FEDEX UPS Client Courier Pace Courier

Lab Sample Temperature Info:
Temp Blank Received: Y N NA
Therm ID#: _____
Cooler 1 Temp Upon Receipt: _____oC
Cooler 1 Therm Corr. Factor: _____oC
Cooler 1 Corrected Temp: _____oC
Comments:

Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)	Date/Time:
Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)	Date/Time:
Relinquished by/Company: (Signature)	Date/Time:	Received by/Company: (Signature)	Date/Time:

MTJL LAB USE ONLY
Table #:
Acctnum:
Template:
Prelogin:
PM:
PB:

Trip Blank Received: Y N NA
HCL MeOH TSP Other

Non Conformance(s): YES / NO

Page: _____ of: _____

DRAFT

Attachment II – Example Sample Condition Upon Request Form

Pittsburgh Lab Sample Condition Upon Receipt

Pace Analytical Client Name: _____ Project # _____

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace Other _____ **Label** _____

Tracking #: _____ **LIMS Login**

Custody Seal on Cooler/Box Present: ☐ yes ☐ no Seals intact: ☐ yes ☐ no

Thermometer Used _____ **Type of Ice:** Wet Blue None

Cooler Temperature **Observed Temp** _____ °C Correction Factor: _____ °C Final Temp: _____ °C

Temp should be above freezing to 6°C

Comments:	Yes	No	N/A	pH paper Lot# / Date and Initials of person examining contents: _____
Chain of Custody Present:				1.
Chain of Custody Filled Out:				2.
Chain of Custody Relinquished:				3.
Sampler Name & Signature on COC:				4.
Sample Labels match COC:				5.
-Includes date/time/ID Matrix:_____				
Samples Arrived within Hold Time:				6.
Short Hold Time Analysis (<72hr remaining):				7.
Rush Turn Around Time Requested:				8.
Sufficient Volume:				9.
Correct Containers Used:				10.
-Pace Containers Used:				
Containers Intact:				11.
Orthophosphate field filtered				12.
Hex Cr Aqueous Compliance/NPDES sample field filtered				13.
Organic Samples checked for dechlorination:				14.
Filtered volume received for Dissolved tests				15.
All containers have been checked for preservation.				16.
All containers needing preservation are found to be in compliance with EPA recommendation.				
exceptions: VOA, coliform, TOC, O&G, Phenolics				Initial when completed / Date/time of preservation / Lot # of added preservative
Headspace in VOA Vials (>6mm):				17.
Trip Blank Present:				18.
Trip Blank Custody Seals Present				
Rad Aqueous Samples Screened > 0.5 mrem/hr				Initial when completed: / Date:

Client Notification/ Resolution:

Person Contacted: _____ Date/Time: _____ Contacted By: _____

Comments/ Resolution: _____

☐ **A check in this box indicates that additional information has been stored in ereports.**

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)

***PM review** is documented electronically in LIMS. When the Project Manager closes the SRF Review schedule in LIMS. The review is in the Status section of the Workorder Edit Screen.

J:\QAQC\Master\Document Management\Sample Mgt\Sample Condition Upon Receipt Pittsburgh (C056-7 16Feb2018)

Attachment II – Example Sample Receiving Non-Conformance Form (NCF)

Pace Analytical

Sample Receiving Non-Conformance Form (NCF)

Date:	Evaluated by:	Affix Workorder/Login Label Here or List Pace Workorder Number or MTJL Log-in Number Here
Client:		

1. If Chain-of-Custody (COC) is not received: contact client and if necessary, fill out a COC and indicate that it was filled out by lab personnel. Note issues on this NCF.

2. If COC is incomplete, check applicable issues below and add details where appropriate:

Collection date/time missing or incorrect	Analyses or analytes: missing or clarification needed	Samples listed on COC do not match samples received (missing, additional, etc.)
Sample IDs on COC do not match sample labels	Required trip blanks were not received	Required signatures are missing

Comments/Details/Other issues not listed above:

3. Sample integrity issues: check applicable issues below and add details where appropriate:

Samples: Past holding time	Samples: Condition needs to be brought to lab personnel's attention (details below)	Preservation: Improper
Samples: Not field filtered	Containers: Broken or compromised	Temperature: not within acceptance criteria (typically 0-6C)
Samples: Insufficient volume received	Containers: Incorrect	Temperature: Samples arrived frozen
Samples: Cooler damaged or compromised	Custody Seals: Missing or compromised on samples, trip blanks or coolers	Vials received with improper headspace
Samples: contain chlorine or sulfides	Packing Material: Insufficient/Improper	Other:

Comments/Details:

4. If Samples not preserved properly and Sample Receiving adjusts pH, add details below:

Sample ID:	Date/Time:	Amount/type pres added:
Preserved by:	Initial and Final pH:	Lot # of pres added:
Sample ID:	Date/Time:	Amount/type pres added:
Preserved by:	Initial and Final pH:	Lot # of pres added:
Sample ID:	Date/Time:	Amount/type pres added:
Preserved by:	Initial and Final pH:	Lot # of pres added:

5. Client Contact: If client is contacted for any issue listed above, fill in details below:

Client:	Contacted per:	
PM Initials:	Date/Time:	

Client Comments/Instructions:

F-ALL-C-011-rev.00, 05Jul2018

Attachment IIA – Sample Acceptance Policy (from F-ALL-C-006)

In accordance with regulatory guidelines, Pace Analytical facilities comply with the following sample acceptance policy for all samples received.

If the samples do not meet the sample receipt acceptance criteria outlined below, the Pace facility is required to document all non-compliances, contact the client, and either reject the samples or fully document any decisions to proceed with analyses of samples that do not meet these criteria. Any results reported from samples not meeting these criteria are appropriately qualified on the final report.

Sample Acceptance Policy requirements:

1. Sample containers must have unique client identification designations, and dates and times of collection, that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC);
2. There must be clear documentation on the COC, or related documents such as the Sample Condition Upon Receipt (SCUR) form, that lists the unique sample identification, sampling site location (including state; some regulations may require city, county, etc.), date and time of sample collection, and name and signature of the sample collector;
3. There must be clear documentation on the COC, or related documents, that lists the requested analyses, the preservatives used, sample matrix, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.);
4. Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval;
5. Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval;
6. Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval;
7. Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval;
8. All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2°C of the required temperature, or within the method-specified range. For samples with a required temperature of 4°C, samples with a temperature ranging from just above freezing to 6°C are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
9. For all compliance **drinking water** samples, analyses will be rejected at the time of receipt if they are not received in a secure manner, are received in inappropriate containers, are received outside the required temperature range, are received outside the recognized holding time, are received with inadequate identification on sample containers or COC, or are improperly preserved (with the exception of VOA samples- tested for pH at time of analysis and TOC- tested for pH in the field).
10. Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.

Attachment III – Example Sample Receipt Form

Sample Receipt Form

Pace Analytical Services, Inc.
Indiana

Pace Analytical®
www.pacelabs.com

Sample Acknowledgement Recipients:

Email:

Bill to:

Final Report Recipients:

Line Item Descriptions:
[4] ' Water Short VOC

Client P O No:
Phone: 1(317)875-5894
Project Manager: Phaedra Zucksworth
Client Project ID:

Lab Project No: 5018684
Project Deliverables Type: Standard Report
Project Report Due Date: 09/25/08
Profile: 907

Lab Smp ID: 5018684001 **Client Smp ID: RW-3:G090908** Collected Date: 09/09/08 11:25
Proj Smp No: 1 Matrix: Water Smp Type: PS Line Item: 4 Received Date: 09/11/08 10:19
Location: OH BUSTR

Auxillary Data:

Consultant Project Number:	NA	ENFOS Project (AAAAA-0000):	
Site ID/Facility Number:		Phase:	01
Gem Portfolio:	CENTRAL	SubPhase/Task:	03
State of Sample Collection:	OH	Cost Element:	05
Consultant Name:	URS	RCOP:	no
Rush Charges:	0		

PARAMETER	METHOD	UNIT PRICE	WR	SPL	%
8260 WUST - 8260 MSV UST	EPA 8260	$27.00			

COMPOUND	PQL	UNITS
Benzene	5	ug/L
Ethylbenzene	5	ug/L
Methyl-tert-butyl ether	4	ug/L
Toluene	5	ug/L
Xylene (Total)	10	ug/L

Sub Total - Sample 208364 $27.00

Lab Smp ID: 5018684002 **Client Smp ID: RW-4:G090908** Collected Date: 09/09/08 11:00
Proj Smp No: 2 Matrix: Water Smp Type: PS Line Item: 4 Received Date: 09/11/08 10:19
Location: OH BUSTR

PARAMETER	METHOD	UNIT PRICE	WR	SPL	%
8260 WUST - 8260 MSV UST	EPA 8260	$27.00			

COMPOUND	PQL	UNITS
Benzene	5	ug/L
Ethylbenzene	5	ug/L
Methyl-tert-butyl ether	4	ug/L
Toluene	5	ug/L
Xylene (Total)	10	ug/L

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Attachment IIIA - Example Cooler Temperature Breakdown Form

Pace Pittsburgh Laboratory

Temperature Cooler Breakdown

	Glass Jar 1L	Glass Jar 500mL	Glass Jar 250 mL	Glass Jar 120mL	Soil Kit	Chem 1-liter	Chem 500 ml	Chem 250	Organics	Nutrient 250	Nutrient 500	Phenolics	TOC	TOX	Total metals 250	Dissolved Metals 250	O&G	VOA	Cyanide	Sulfide	Bacteria	Wipes/swipe/smear/filter	Radchem	Cubitainer	Ziploc	Other

Cooler ID	Temp	Ice present Y/N

Comments

Initials _____

J:\QAQC\Master\Document Management\Sample Mgt\Cooler Temp Breakdown checksheet (C036-1 2June2016)

Attachment IV
EPIC Container Code Key

First Char	type
A	Amber
B	Borosilicate (clear)
C	Cassettes
D	VOA-water
E	Extract
F	Filter
G	Gallon
I	Wipe/Swab
J	Jar
K	Kit
M	Media
N	None
P	Polyurethane foam
R	Terra core
S	nondescript
T	nondescript
U	Summa
V	VOA-soil
W	wide mouth jar
X	XAD trap
Z	Ziploc

Second Char	material
G	Glass
P	Plastic
T	Tube
C	Cubitainer
N	General
S	Septa

Third Char	size
1	1 liter
2	500 nml
3	250 ml
5	120 ml
9	40 ml
F	4 oz

Fourth Char	preservative	
H	HCl	Hydrochloric acid
S	H2SO4	Sulfuric acid
T	NaThiosulfate	Sodium Thiosulfate
U	Unpreserved	Unpreserved
A	Ascorbic Acid	Ascorbic Acid
Z	Zn Acetate	Zinc Acetate
B	NaBisulfite	Sodium Bisulfite
0	NaOH	Sodium Hydrozide
N	HNO3	Nitric acid
M	Me0H	Methanol
X	Hexane	Hexane
W	Preweighed	Preweighed
C	Mea2	Methylene chloride
P	TSP	Trisodium phosphate

Wildcards	
AF	Air Filter
BTBE	Brass Tube
C	Air cassettes
DIG	Lab Digestate Container
EXT	Lab Extract Container
EZH	25g Encore
EZI	5g Encore
F	Large Air Filter
GCUB	1 Gallon Cubitainer
GJ	1Gallon Jug
GN	General Unpreserved
GNHC	General preserved with HCL
GNN	General preserved with Nitric Acid
I	Wipe/Swab
KE	Endotoxin kit
KL	Legionella kit
M	Plate media
NONE	No Container Needed
PUF	Polyurethane Foam
R	Terra core
STBE	Shelby Tube
T	Tedlar Bag
TCLP	Kit for Standard TCLP analysis
U	Summa Can
VSG	Headspace septa vial & HCl
VSGU	20mL scintillation vial
WK	WhirlPak Bag
XAD	XAD Trap
ZPLC	Ziploc Bag

Attachment IV
Common EPIC Container Codes

Contianer Code	Description
AF	Air Filter
AGIN	1L amber glass HCl
AG1S	1L amber glass H2SO4
AGIT	1L amber glass Na Thiosulfate
AG1U	1L amber glass unpreserved
AG2N	500mL amber glass HNO3
AG2S	500mL amber glass H2504
AG2U	500mL amber glass unpreserved
AG3S	250mL amber glass H2504
AG3U	250mL amber glass unpreserved
BG1H	1L clear glass HCl
BG1S	1L clear glass H2504
BG1T	1L clear glass Na Thiosulfate
BG1U	1L clear glass unpreserved
BP1A	1L plastic NAOH, Asc Acid
BP1N	1L plastic HNO3
BP1S	1L plastic H2504
BP1U	1L plastic unpreserved
BP1Z	1L plastic NaOH, Zn Ac
BP2A	500mL plastic Na0H,Asc Acid
BP2N	500mL plastic HNO3
BP2O	500mL plasitc NaOH
BP2S	500mL plastic H2SO4
BP2U	500mL plastic unpreserved
BP2Z	500mL plastic NaOH, Zn Ac
BP3A	250mL plastic Na0H, Asc Acid
BP3C	250m1 plastic NAOH
BP3N	250mL plastic HNO3
BP3S	250mL plastic H2804
BP3U	250mL plastic unpreserved
BP3Z	250mL plastic NaOH, Zn Ac
BTBE	Brass Tube
C	Air cassettes
DG9B	40mL amber VOA vial Na Bisulfate
DG9H	40mL amber VOA vial NCI
DG9M	40mL clear VOA vial Me0H
DG9P	40mL amber VOA vial TSP
DG9S	40mL amber VOA vial H2504
DG9T	40mL amber VOA vial Na Thio
DG9U	40mL amber VOA vial
DIG	Lab Digestate Container
EXT	Lab Extract Container
EZH	25g Encore
EZI	5g Encore

Contianer Code	Description
F	Large Air Filter
GCUB	1 Gallon Cubitainer
GJ	1Gallon Jug
GN	General Unpreserved
GNHC	General preserved with HCL
GNN	General preserved with Nitric Acid
I	Wipe/Swab
JGFM	4oz amber wide jar Me0H
JGFS	4oz amber wide jar H2SO4
JGFU	4oz amber wide jar
KE	Endotoxin kit
KL	Legionella kit
M	Plate media
NONE	No Container Needed
PUF	Polyurethane Foam
R	Terra core
SPST	120mL Coliform Na Thiosulfate
STBE	Shelby Tube
T	Tedlar Bag
TCLP	Kit for Standard TCLP analysis
U	Summa Can
VG9H	40mL clear VOA vial HCl
VG9T	40mL clear VOA vial Na Thiosulfate
VG9U	40mL clear VOA vial
VG9W	40mL glass VOA vial preweighted (EPA 5035)
VSG	Headspace septa vial & HCl
VSGU	20mL scintillation vial
WGFC	4oz wide jar and wipe with MeCl
WGFU	4oz wide jar unpreserved
WGFX	4oz wide jar and wipe Hexane
WGKU	8oz wide jar unpreserved
WK	WhirlPak Bag
WPDU	16oz clear wide mouth jar
XAD	XAD Trap
ZPLC	Ziploc Bag

Attachment V
Sample Receipt Form - EPIC Container Codes

Example of multiple containers for the same analysis on the same sample. 1/3, 2/3 and 3/3 indicate 3 samples each uniquely identified. This also appears on the labels applied to the samples.

Sample Receipt Form

Pace Analytical Services, Inc.
Pittsburgh

Pace Analytical®
www.pacelabs.com

Containers

Lab ID	Container ID	Type	Location	Preservative	Utilization
3017[redacted]001	3017[redacted]001 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]001 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]001 VG9H1/3	VG9H		NA	8260 W
	3017[redacted]001 VG9H2/3	VG9H		NA	SI-34MSV
	3017[redacted]001 VG9H3/3	VG9H		NA	
3017[redacted]002	3017[redacted]002 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]002 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]002 VG9H1/3	VG9H		NA	8260 W
	3017[redacted]002 VG9H2/3	VG9H		NA	SI-34MSV
	3017[redacted]002 VG9H3/3	VG9H		NA	
3017[redacted]003	3017[redacted]003 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]003 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]003 VG9H1/3	VG9H		NA	8260 W,SI-34MSV
	3017[redacted]003 VG9H2/3	VG9H		NA	
	3017[redacted]003 VG9H3/3	VG9H		NA	
3017[redacted]004	3017[redacted]004 VG9H1/3	VG9H		NA	8260 W,SI-34MSV
	3017[redacted]004 VG9H2/3	VG9H		NA	
	3017[redacted]004 VG9H3/3	VG9H		NA	
3017[redacted]005	3017[redacted]005 AG1U1/1	AG1U		NA	
	3017[redacted]005 BP3N1/1	BP3N		NA	7470 WD
	3017[redacted]005 VG9H1/3	VG9H		NA	8260 W,SI-34MSV
	3017[redacted]005 VG9H2/3	VG9H		NA	
	3017[redacted]005 VG9H3/3	VG9H		NA	6010 WD,8082 W
3017[redacted]006	3017[redacted]006 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]006 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]006 VG9H1/3	VG9H		NA	8260 W
	3017[redacted]006 VG9H2/3	VG9H		NA	SI-34MSV
	3017[redacted]006 VG9H3/3	VG9H		NA	
3017[redacted]007	3017[redacted]007 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]007 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]007 VG9H1/3	VG9H		NA	8260 W
	3017[redacted]007 VG9H2/3	VG9H		NA	SI-34MSV
	3017[redacted]007 VG9H3/3	VG9H		NA	
3017[redacted]008	3017[redacted]008 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]008 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]008 VG9H1/3	VG9H		NA	8260 W
	3017[redacted]008 VG9H2/3	VG9H		NA	SI-34MSV
	3017[redacted]008 VG9H3/3	VG9H		NA	
3017[redacted]009	3017[redacted]009 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]009 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]009 VG9H1/3	VG9H		NA	8260 W
	3017[redacted]009 VG9H2/3	VG9H		NA	SI-34MSV
	3017[redacted]009 VG9H3/3	VG9H		NA	
3017[redacted]010	3017[redacted]010 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]010 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]010 VG9H1/3	VG9H		NA	8260 W
	3017[redacted]010 VG9H2/3	VG9H		NA	SI-34MSV
	3017[redacted]010 VG9H3/3	VG9H		NA	
3017[redacted]011	3017[redacted]011 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]011 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]011 VG9H1/3	VG9H		NA	8260 W
	3017[redacted]011 VG9H2/3	VG9H		NA	SI-34MSV
	3017[redacted]011 VG9H3/3	VG9H		NA	
3017[redacted]012	3017[redacted]012 AG1U1/1	AG1U		NA	8082 W
	3017[redacted]012 BP3N1/1	BP3N		NA	6010 WD,7470 WD
	3017[redacted]012 VG9H1/3	VG9H		NA	8260 W,SI-34MSV
	3017[redacted]2 VG9H2/3	VG9H		NA	

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Attachment VI
Tests, Sample Containers and Required Volumes

Parameter	Method	Matrix	Container	Preservative	Volume Needed (mL or g)
Acidity	SM2310B	Water	Plastic/Glass	≤ 6°C	100 mL
Actinides	HASL-300	Water		pH<2 HNO_3	300 mL
Actinides	HASL-300	Solid		None	500 g
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	≤ 6°C	100 mL
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	600 mL
Total Alpha Radium (see note 3)	9315	Solid	Plastic/Glass	None	500 g
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	≤ 6°C; EDA if bromate or chlorite run	50 mL per ion being tested
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤ 6°C; $Na_2S_2O_3$	100 mL
Base/Neutrals and Acids	8270	Solid	8oz Glass	≤ 6°C	15 g
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1000 mL 3X volume for QC
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6°C	1000 mL
Cation Exchange	9081	Solid	8oz Glass	None	
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	50 mL
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	50 mL
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	10 mL
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 6°C $Na_2S_2O_3$	100 mL
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 6°C$Na_2S_2O_3$	100 mL
Coliform, Total and Escherichla (E. coli)	SM9223B	Water	100mL Plastic	≤ 10°C; $Na_2S_2O_3$	100 mL
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	100 mL

Parameter	Method	Matrix	Container	Preservative	Volume Needed (mL or g)
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	100 mL
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	30 g
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6ºC; ascorbic acid if Cl present	100 mL
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6ºC	15 g
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6ºC; $Na_2S_2O_3$ if Cl present	1000 mL 3X volume for QC
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6ºC; $Na_2S_2O_3$ if Cl present	40 mL X 3 plus Trip Blank
Ferrous Iron	SN3500Fe-D	Water	Glass	None	10 mL
Flashpoint/Ignitability	1010	Liquid	Plastic/Glass	None	50 mL
Flashpoint/Ignitability	1010	Solid	Plastic/Glass	None	50 g
Fluoride	SM4500Fl-C,D	Water	Plastic	None	500 mL
Gamma Emitting Radionuclides (see note 3)	901.1	Water	Plastic/Glass	pH<2 HNO_3	2 X 1000 mL
Gamma Emitting Radionuclides	901.1	Solid	Plastic/Glass	None	500 g
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	40 mL X 3 plus Trip Blank
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	Terracore kit plus Trip Blank
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	200 mL
Gross Alpha and Gross Beta (see note 3)	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	400 mL
Gross Alpha and Gross Beta	9310	Solid	Glass	None	500 g
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	50 mL
Hexavalent Chromium	7196/218.6/SM3500Cr-C,D	Water	Plastic/Glass	≤ 6ºC	250 mL
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6ºC	10 g
Mercury	7471	Solid	8oz Glass Jar	≤ 6ºC	2 g
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	25 mL
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	2 g
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	50 mL
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	50 mL
Nitrogen, Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6ºC	10 g
Nitrogen, Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6ºC	50 mL
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6ºC	50 mL

Parameter	Method	Matrix	Container	Preservative	Volume Needed (mL or g)
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	50 mL
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	10 mL
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 HCl ; ≤ 6°C	1000 mL 3X volume for QC
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	75 g
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass	≤ 6°C; NaOH or HCl if not extracted within 72 hours	1000 mL 3X volume for QC
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1000 mL 3X volume for QC
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6°C	15 g
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1000 mL 3X volume for QC
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6°C	15 g
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	1000 mL
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	100 mL
Paint Filter Liquid Test	9095	Solid	Plastic/Glass	None	100 g
pH	SM4500H+B/9040	Water	Plastic/Glass	None	50 mL
pH	9045	Solid	Plastic/Glass	None	20 g
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	50 mL
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; ≤ 6°C	100 mL This requires a separate containe for filtration
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	100 mL
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	5 g
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	15 g
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1000 mL 3X volume for QC
Radioactive Strontium (see note 3)	905.0	Water	Plastic/Glass	pH<2 HNO_3	200 mL
Radium-226 (see note 3)	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	600 mL
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	800 mL

Parameter	Method	Matrix	Container	Preservative	Volume Needed (mL or g)
Radium-228 (see note 3)	9320/901.1	Solid	Plastic/Glass	None	500 g
Radon	7500-Rn-B	Water	40 ml glass	None	40 mL X 2
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6ºC	
Solids, Settleable	SM2540F	Water	Glass	≤ 6ºC	1000 mL
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6ºC	100 mL
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6ºC	100 mL
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6ºC	100 mL
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6ºC	100 mL
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6ºC	20 g
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6ºC	100 mL
Sulfate	SM4500SO4/9036/ 9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6ºC	10 mL
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	100 mL
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	30 g
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤ 6ºC	250 mL
Sulfite	SM4500SO3	Water	Plastic/Glass	None	500 mL
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6ºC	250 mL
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	40 mL glass	pH<2 H_2SO_4 ; ≤ 6ºC	40 mL X 2
Tritium	906.0	Water	Glass	None	200 mL
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6ºC	50 mL
Total Uranium (see note 3)	ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO3	50 mL
Volatiles	8260	Solid	5035 vial kit	See note 1	Terracore plus Tr Blank
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6ºC; $Na_2S_2O_3$ if Cl present	40 mL X 3 plus Trip Blank
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6ºC; $Na_2S_2O_3$ if Cl present, unpreserved for specific compounds	40 mL X 3 plus Trip Blank

ATTACHMENT C-27

AM23G SCRUBBED-PACE ENERGY SERVICES PITTSBURGH

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STANDARD OPERATING PROCEDURE

ANALYSIS OF LOW LEVEL VOLATILE FATTY ACIDS

BY ION CHROMATOGRAPHY

Reference Methods: AM23G

Local SOP Number:	S-PAE-LLVFA-001-rev.02 (S)
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APPROVALS

Laboratory Manager — Date: 11/3/2017

Assistant General Manager — Date: 11/3/2017

Laboratory Quality Manager — Date: 11/3/2017

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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S-PAE-LLVFA-001-rev.02 (S)

TABLE OF CONTENTS

1. Purpose/Identification of Method

The purpose of this Standard Operating Procedure (SOP) is to describe a method of determination of low level volatile fatty acids (VFAs) in groundwater by ion chromatography. VFA's are also known as metabolic acids, stench acids, or low molecular weight organic acids (LMWOAs). VFAs are not a contaminant but a metabolic by-product and measurement of their concentration can aid remedial effort.

2. Summary of Method

2.1. Samples are pretreated to remove potential interference. The pretreated samples are then spiked with a mix of compounds that serve as preservatives and internal retention time markers. Samples are then analyzed by ion exchange in an ion chromatograph (IC). In the alkaline solutions of the IC, the acids ionize to their conjugate anions. The anions are separated on an ion chromatograph and chemically converted to their acid form in an electrolytic regenerated suppressor (ERS). The volatile fatty acids pass through an electrical conductivity detector. The instrument responds by producing peaks that correspond to the individual VFA concentration.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analysis of volatile fatty acids by IC.

3.2. **Parameters**: See table section 5, Limits of Quantitation.

4. Applicable Matrices

4.1. This method is applicable to groundwater and soil.

5. Limits of Detection and Quantitation

5.1. Studies are run annually in accordance with the Standard Operating Procedure for the Determination of Method Detection Limits (MDL/LOD) and Practical Quantitation Limits (PQL/LOQ), SOP-ADM 18. Results for these studies are retained by the Quality Manager. Reporting limits for the VFAs are shown below:

Limits of Quantitation

VFA	CAS Number	LOQ in mg/L
Lactic Acid	50-21-5	0.2
Acetic acid	64-19-7	0.1
Propionic acid	79-09-4	0.1
Formic acid	64-18-6	0.1
Butyric acid	107-92-6	0.1
Pyruvic acid	127-17-3	0.1
i-Pentanoic	503-74-2	0.1
Pentanoic acid	109-52-4	0.1
i-Hexanoic	646-07-1	0.2
Hexanoic acid	142-62-1	0.2

6. Interferences

6.1. The anion exchange chromatography used in this procedure produces considerable retention time shifts. The use of internal retention time markers can help eliminate any uncertainty in peak identification that these shifts introduce. Retention time shifts are due to variations in the pH and the ionic strength of the treated sample. Any increase in ionic strength will shift retention times and may degrade the chromatography. For this reason, surrogates are not used in this method because they would increase the ionic strength of the sample.

Dichloroacetic acid, nitrate and bromide co-elute. Inadvertent introduction of carbonate or carbon dioxide can adversely affect the samples. Additionally, nitrile gloves must be worn throughout the preparation procedure to minimize the introduction of sulfate and chloride.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Samples are collected in 40 ml amber glass VOA vials and preserved with 0.2mL of Benzalkonium chloride (BAK) Field Preservation Solution. Samples may be stored for up to 14 days at a temperature just above freezing but below 6° C.

8. Definitions

8.1. **Internal Retention Time Marker**: a spike of non-target compounds that is placed in a sample and used to determine the relative retention times. Internal Retention time markers are used to compensate for the inevitable retention time shift that occurs in this chromatography. The internal retention time marker is not to be used quantitatively, as a surrogate would be used.

8.2. **Analytical Batch:** a group of twenty client samples or fewer that are prepared and analyzed together within a 24 hour period.

8.3. **Laboratory Control Sample:** portion of reagent grade water that has been prepared in the same manner as a field sample, but has been spiked with a known amount of the contaminants being monitored. A LCS is used to assess the performance of the measurement system. A single laboratory control sample shall be analyzed with each analytical batch.

8.4. **Matrix Spike/Matrix Spike Duplicate:** prepared by adding a known concentration of target analyte to additional aliquots of field sample. Matrix spikes are used to determine the effect of sample matrix on a method's recovery efficiency. An MS and MSD shall be prepared with each analytical batch.

8.5. **Method Blank:** a portion of reagent grade water that is free from the analytes of interest that is processed through all the steps of the analysis procedure. A single method blank shall be prepared with each analytical batch.

8.6. **Initial Calibration Verification:** After initial calibration and prior to any sample analysis, the calibration must be verified by use of a standard at or near the mid-range of the calibration. The ICV standard is prepared from a source independent of the calibration standards.

8.7. **Continuing Calibration Verification:** In order to verify the calibration on a day-to-day basis, a CCV is run at the beginning and end of each analytical batch. This is a standard at or near the mid-range of the calibration. The CCV standard is prepared from a source independent of the calibration standards.

8.8. **Initial and Continuing Calibration Blanks:** The ICV or CCV must be immediately followed by an initial or continuing calibration blank (ICB or CCB). This standard is made up of a portion of reagent grade water.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Equipment

- Sample pre-treatment rack

9.2. Operating Conditions

- Proprietary

9.3. Materials

- Type II deionized water

10. Reagents and Standards

All reagents are prepared from Type II deionized water with a resistivity of at least 18-mega ohm/cm (Mohm). Reagent grade chemicals are used in preparing all reagents. All standards are labeled and documented in accordance with the Standard Operating Procedure for Standards and Reference Materials. The following are used:

- Quinic Acid/ Benzalkonium chloride (BAK) RT Marker Solution: Place 250 ml DI water in a 500 ml volumetric flask and add 2.0 g of BAK and let the BAK dissolve. Dissolve 0.1g Quinic acid into the solution. Dilute to volume with DI water.
- Benzalkonium chloride (BAK) Field Preservation Solution: BAK is an extremely viscous liquid that is completely water soluble (it is soap). Dissolve 12 grams of BAK into 1 liter of DI water, or purchase pre-preserved vials from an approved vendor.
- Potassium Hydroxide KOH (Eluent Generator Cartridge)
- UHP Nitrogen
- Calibration standard (10ppm) is obtained from Restek or other approved vendor.
- Working stock standard (100ppm) is obtained from Restek or other approved vendor.
- Initial and Continuing Calibration Verification (ICV/CCV): 2 ppm standard prepared by 0.2 mL of working stock standard solution to 9.8 mL of deionized water
- Laboratory Control Sample (LCS) and MS/MSD: 2 ppm standard prepared by 0.2 mL of working stock standard solution to 9.8 mL of deionized water or field sample

11. Calibration and Standardization

11.1. Standards are purchased as a custom mix from Restek or another vendor. All standards are stored in tightly sealed glass containers and cooled to just above freezing and below 6°C when not in use. Initial calibration standards are prepared at eight concentrations: 0.02, 0.05, 0.1, 0.2, 0.5, 1.0, 2.0, and 5.0ppm.

The standards are readied for analysis according to the following procedure:

- Insure that the instrument setup is correct.
- Prepare calibration standards and a blank by labeling nine vials with the concentrations specified in section 4.1.
- Add one drop of the internal retention time marker solution
- Cap the vials.
- Load the calibration standards into an autosampler rack and begin a sequence file that identifies each bottle and its contents. Each vial is assigned a tray position in the analytical sequence.

Note: Compounds are listed in order of elution. Quinic acid and BAK are used as internal retention time markers only.

Calibration Mix Concentrations

Acid	Conc. (ppm)	ICAL1 500x	ICAL2 200x	ICAL3 100x	ICAL4 50x	ICAL5 20x	ICAL6 10x	ICAL7 5x	ICAL8 2x
Quinic	-	-	-	-	-	-	-	-	-
Lactic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
Acetic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
Propionic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
Formic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
Butyric	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
Pyruvic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
i-Pentanoic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
Pentanoic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
BAK	-	-	-	-	-	-	-	-	-
i-Hexanoic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0
Hexanoic	10	0.02	0.05	0.1	0.2	0.5	1.0	2.0	5.0

12. Procedure

12.1. All samples, including quality control samples (MB, LCS, MS/MSD), must be field or lab preserved and pre-treated. The purpose of the pre-treatment is to remove sulfate, chloride, and heavy metals, and to minimize carbonate interference. Pre-treatment is accomplished through the use of pre-treatment cartridges. If the samples contain particulate material that may clog the IC system, an additional filter cartridge may be used to remove the particulates.

12.2. Samples are checked using Hach Quantab chloride titrator strips. It is documented on the sequence log whether the screening is greater or less than 250mg/L. All samples with chloride concentrations greater than 250ppm chloride are diluted by a factor of at least 10 prior to pre-treatment. In addition, some samples, based on historical information and client approval, are diluted at a factor of 10 or higher prior to pre-treatment.

- Pre-treatment cartridges are hydrated per manufacturer's instructions. The eluent from these hydrating flushes should be discarded in accordance with the Standard Operating Procedure for Waste Disposal (SOP-ADM 14).
- 10 ml of sample should then be placed into the syringe, and then forced through the hydrated cartridges. The first 6 ml of the eluent should be discarded, and the rest should be collected in 20 ml VOA vials containing 1 drop of the Internal RT Marker Solution.
- A portion of each sample is transferred into the autosampler vials. Cap the vials. Each vial is assigned a tray position in the analytical sequence file.

12.3. Acquisition of data for all standards, samples, blanks, and laboratory control samples is done using a Windows based personal computer outfitted with Dionex Chromeleon software. The peaks are maximized and the noise is minimized if the conversion is perfectly efficient. The conversion occurs in the suppressor (ERS, or electrolytic regenerated suppressor). The software collects data, plots the peaks, integrates the peaks, calculates the calibration curve associated with the target analytes, and calculates the concentrations of the analytes in mg/L. After review from the analyst, a standard report containing a chromatogram is printed.

13. Quality Control

The following quality criteria are monitored with each analytical sequence or daily batch of samples analyzed:

13.1. Calibration accuracy: The correlation of determination (which has the same meaning for a quadratic calibration as for a linear calibration) must be at least 0.99. If this criterion is failed, the system should be inspected and the calibration repeated.

13.2. Initial Calibration Verification (ICV): The acceptance criteria for the ICV standard must not vary more than ± 15% of its true value. If the calibration curve cannot be verified within the specified limits, the cause must be determined and the instrument recalibrated before samples are analyzed.

13.3. Continuing Calibration Verification (CCV): The acceptance criteria for the CCV standard must not vary more than ± 15% of its true value. If the calibration cannot be verified within the specified limits repeat the CCV analysis. If the CCV fails for a second time, discontinue sample analysis, determine the cause, and recalibrate the instrument. All samples analyzed after the last acceptable CCV must be reanalyzed.

13.4. Initial/Continuing Calibration Blank (ICB/CCB): The calibration blank must not contain target analytes above the reporting limits. For DoD analyses, the results for the CCB must be < ½ the reporting limit. If this criterion is not met, inspect all glassware, etc. and then prepare and analyze another blank. Blanks shall be run until this criterion is met. If three blanks are analyzed in succession and this criterion is still not met, the Laboratory Manager shall be notified.

13.5. Method Blank (MB/PBW): The method blank must not contain target analytes above the reporting limits. For DoD analyses, the results for the MB/PBW must be < ½ the reporting limit. If this criterion is not met, inspect all glassware, etc. and then prepare and analyze another blank. Blanks shall be run until this criterion is met. If three blanks are analyzed in succession and this criterion is still not met, the Laboratory Manager shall be notified.

13.6. Laboratory Control Sample (LCS): The acceptance criteria for the LCS must not vary more than ± 30% of its true value. If the percent recovery for this sample is not between specified limits, a fresh

LCS solution should be made and the LCS should be run again. If it fails again, all samples analyzed after the last acceptable LCS must be prepared again and reanalyzed.

13.7. Matrix Spike and Matrix Spike Duplicate (MS/MSD): The percent recovery must be between 70% and 130% and the relative percent difference (RPD) must be ≤ 30%. If this criterion fails, but all other instrument run criteria are passed, then it is simply noted in the case narrative for the instrument run and the analysis proceeds.

14. Data Analysis and Calculations

14.1. **Calibration Calculations**

The standards are analyzed as samples and then the Chromeleon software is used to combine the calibration and the method file. Because the conductivity of a weak acid is not directly proportional to the concentration, the calibration is done with a quadratic equation. Because this introduces two extra degrees of freedom, the large number of calibration levels is required. The calibration points (concentration vs. instrument response) are fit via a least-squared-error type regression routine in the software. For further procedural details, please see the Chromeleon software documentation.

The formula used for calculating concentrations is as follows:

Conc= $\frac{-b \pm \sqrt{b^2 + 4a(\text{area})}}{2a}$

Where: a=curve

b=slope

Percent recovery:

$$Percent\ \text{Recovery} = \frac{Measured\ Value}{True\ Value} X100$$

Duplicate Relative percent recovery (RPD):

$$\%RPD = \frac{\left|MS\ result - MSD\ result\right|}{\frac{\left(MS\ result + MSD\ result\right)}{2}} X100\%$$

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. **Peak Identification/Retention Time Determination**

Initially, the order of elution was determined from the analysis of single component standards. That order has been found to be the same order in which the acids are listed section 11.

With that information, the retention time and width of the window used to make identifications should be based on measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. The exact retention times will vary as a function of sample composition, column type, column age, and column history. For the instruments that use this method, true retention times and retention time windows are taken from the most recent standard analyzed. New retention times need to be re-evaluated each time a new column is installed or other maintenance is performed.

Retention time studies have been conducted for this analysis, however the experience of the analyst should weigh heavily in the interpretation of chromatograms. These studies are kept on file in the Quality Systems Office.

15.2. **Acceptance Criteria for Quality Control Measures**

Specifics related to acceptance criteria of quality assurance markers can be found in section 13, Quality Control.

15.3. **Secondary/Peer Data Review**

The analyst is responsible for insuring that all calibrations, calibration checks, and quality control samples are within the specifications outlined in this SOP. All data undergoes validation by the analyst and another analyst who is certified in this method. Both signatures are required on the case narrative sheets that are turned into the Laboratory Manager.

The analyst checks all raw data and calculations for reasonableness and accuracy, making sure that sample dilutions are taken into account. Quality control results are rechecked for compliance with acceptance criteria. If any acceptance criteria cannot be met or if any atypical conditions are encountered, a Case Narrative detailing the conditions is written and handed in with the results.

15.4. **Laboratory Manager Data Review**

The Laboratory Manager reviews 10% of all laboratory data and calculations. This review includes sample results, quality control acceptance limits, and a review of the level of quality control required for the project.

16. Corrective Actions for Out-of-Control Data

16.1. All samples associated with out of control quality control samples must be reanalyzed. Specific corrective actions are noted for each quality assurance marker in section 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If quality control acceptance criteria cannot be met using the corrective action within section 13, Quality Control, a detailed check of the de-ionized water and chemical purity is made. Reagents, standards, and other quality control samples are re-prepared and analyzed. If problems persist, sample analysis will be halted and the Technical Director or Laboratory Manager shall be contacted immediately to determine the cause and implement corrective action.

17.2. Any data submitted with unacceptable quality control sample results shall be qualified in a case narrative. The narrative should indicate the out of control event that occurred, the corrective action that was taken, and any other pertinent information to inform the client of exactly what occurred.

18. Method Performance

18.1. Analysts who use this method have been certified for the method by running Initial Demonstration of Proficiency (IDOP) Samples in accordance with the Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and Instrumentation. IDOP's are run any time there is significant change to an instrument, method, or in the training procedure for training a new analyst.

18.2. Performance evaluation samples are currently not available. When they become available, they will be analyzed twice annually using this method.

19. Method Modifications

19.1. The method used for this procedure is a modification of SW846-9056 in that this method uses an ion chromatograph (IC), a hydroxide eluent, an anion-exchange separation column, a hydronium based suppressor column and an electrical conductivity detector. However, due to the data quality objectives (DQOs) presented by the clients use of the data, and the inherent presence of matrix interference even in laboratory prepared samples, the quality control requirements of SW846-9056 are inappropriate for this method. Rather, the DQOs are met and quality is assured by the use of the quality control requirements of SW846-8000.

While SW846-8000 requires confirmation by either a second column or second detector, none is available for ICs that would provide either separation or sensitivity similar to that provided by the system here-in described. The current method is not used to monitor environmental contaminants, so confirmation is not part of the client DQOs. However, if the client requires confirmation we can achieve it by analysis of VFAs by gas chromatograph as per Pace Analytical Energy Services, LLC SOP AM21G.

20. Instrument/Equipment Maintenance

20.1. Instrument maintenance should be followed as suggested in the manufacturer's operation manual and as delineated in SOP S-PAE-Q-006, Equipment Maintenance.

20.2. Column "Clean Out"- Once the retention time of all analytes of interest has passed, the gradient program passes a high concentration of hydroxide onto the column.

The intent of this is to force even highly retained anions off of the column. However, the basic eluent used in this procedure strips the hydronium (H3O) from the suppressor membranes, and suppressor recovery from this clean-out procedure requires quite a lot of time. For 18.5 minutes after the clean-out procedure, the base line eluent is passed through the column, giving the chromatographic system a chance to re-equilibrate.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Safety glasses are required in all laboratory areas. Samples and reagents should always be handled with caution. For other safety concerns, consult the company Chemical Hygiene Plan. Safety Data Sheets (SDS) for all compounds used in this procedure is available in the laboratory.

23. Waste Management

23.1. Unused portions of samples are kept for thirty days following analysis. The samples are then removed from the laboratory and stored until disposal according to the Standard Operation Procedure for Waste Disposal.

23.2. Where possible, the laboratory takes steps to minimize the amount of waste generated by substitution and good chemical handling procedures. For specific information on waste minimization consult the Standard Operation Procedure for Waste Disposal.

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

Citing a reference does not imply that all of the recommendations and/or requirements in those cited methods are required in this Standard Operating Procedure. This section simply refers to sources that were consulted to gather information or knowledge in order to write an informed technical procedure.

25.1. Pace Quality Assurance Manual- most current version

25.2. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.3. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste. SW-846, 3rd ed. Method 8000., Office of Solid Waste and Emergency Response, Washington, DC. 1986.

25.4. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste. SW-846, 3rd ed. Method 9056., Office of Solid Waste and Emergency Response, Washington, DC. 1986.

25.5. Thermo Scientific, Dionex ICS-2100 Ion Chromatography System Operator's Manual, Document No 065291 Revision 3, October 2012.

25.6. Dionex ICS-2000 Ion Chromatography System Operator's Manual, Document No 031857, Revision 03, April 2006.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Instructions for priming the IC pump

27. Revisions

Document Number	Reason for Change	Date
Cover page	Revised footer language and removed the uncontrolled document numbering line.	3/6/2017
Section 4	Add soil to applicable matrices	3/6/2017
Section 12.2	Add wording to 250ppm chloride are “at least” 10 prior to pre-treatment.	3/6/2017
Section 12.2	Added that the chloride checks must be documented in the sequence log.	11/2/2017

Attachment I-Priming the IC Pump

Priming the IC Pump:

- Open the panel of the IC.
- On the right side, under IC system, click shutdown.
- Once the pump has stopped and the pressure is at 0psi, go to the left side under IC pump.
- Click on pump setting.
- Under Eluent Flow Valve, click open.
- Go to the IC and insert a syringe into the right pump head. Turn the knob on the pump head counter clockwise until water flows into the syringe. Repeat several times.
- While doing this, go back to the Pump settings and click Prime. (A message will pop up, instructing you to open the waste valve and click ok.)
- Go to the IC and turn the knob on the left pump head counter clockwise about 2 full turns.
- Go back to the computer and click ok in the message box.
- Let prime for about 30 minutes.
- Go to the panel and under Eluent Flow Valve, click close. Under Pump Setting, click off.
- Close the Eluent Flow Valve. (clockwise turn of right pump head until tight) Then close the waste valve (2 clockwise turns of left pump head-until tight)
- Close Pump Settings.
- Under IC system, click start up.

Quick Start Procedure of ERS 500 Electrolytically Regenerated Suppressor

- Refer to Thermo Scientific Document No. 031951-05, April 2013.

ATTACHMENT C-28

SAMPLE RECEIVING-PACE ENERGY SERVICES
PITTSBURGH

Document Information

Document Number: ENV-SOP-PITTS-0027 **Revision:** 00

Document Title: Sample Receiving

Department(s): Client Services

Previous Document Number: S-PAE-C-003-rev.03

Date Information

Effective Date: 26 Jul 2017

Next Review Date: 26 Jul 2019 **Last Review Date:**

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

STANDARD OPERATING PROCEDURE

SAMPLE RECEIVING

Reference Methods: N/A

Local SOP Number:	S-PAE-C-003-rev.03
Effective Date:	Date of Final Signature
Supersedes:	S-PAE-C-003-rev.02

APPROVALS

Client Services Manager — Date: 7/6/2018

Assistant General Manager — Date: 7/6/2018

Laboratory Quality Manager — Date: 7/16/2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

S-PAE-C-003-rev.03

TABLE OF CONTENTS

SECTION **PAGE**

1. Purpose/Identification of Method

The purpose of this Standard Operating Procedure is to outline the procedures for sample receipt and storage.

2. Summary of Method

2.1. This SOP describes all aspects of sample receipt, sample management and sample storage.

3. Scope and Application

3.1. **Personnel**: This Standard Operating Procedure applies specifically to the Sample Receipt Technician and/or his or her representative during activities of sample receipt. This Standard Operating Procedure also applies to the Client Service Office and bottle preparation personnel.

4. Applicable Matrices

4.1. Not applicable to this SOP.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Not applicable to this SOP.

8. Definitions

Aliquot: a portion of a sample

Background Radiation: naturally occurring radiation.

Chain of Custody Form: record that documents the possession of the samples from the time of collection to receipt in the laboratory. The record may include: number and types of containers; the mode of collection; time of collection; preservation; requested analyses; and sampler's printed name and signature.

Holding Time: the maximum time that samples may be held prior to analysis.

Non-Conformance: samples or sample documentation that are received with incorrect, incomplete, or inadequate information or properties.

Preservation: refrigeration and/or reagents added prior to sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Shall: denotes a requirement that is mandatory.

Short Holding Time: samples that must be analyzed within 48 hours or less of sample collection.

Temperature Blank: a bottle of water that accompanies the samples in each cooler. This blank is used to monitor cooler temperature upon receipt of samples in the laboratory.

Trip Blank: a set of 40mL VOA vials filled with deionized water that travel with samples to be analyzed for target analytes of concern. These samples are analyzed to determine if cross contamination occurred during transport.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Computer

9.2. Printer

9.3. LIMS

9.4. Sink

9.5. Storage Refrigerators with thermometers

9.6. Temperature Gun

9.7. pH Strips

9.8. Disposable pipettes

9.9. Disposable plastic containers

9.10. Safety glasses

9.11. Chemical protective gloves

9.12. Cut resistant gloves

9.13. Auto-retractable utility knives

9.14. Radiation Screening Instrument

The instrument used for scanning coolers and packages for radiation is an S.E. International Monitor 4 powered by a 9 volt battery.

The Monitor 4 Radiation instrument is calibrated annually by S.E. International by pulse generator and is typically ± 15% of full scale relative to Cesium 137.

There are many natural factors which affect background radiation levels at any given time the instrument is turned on.

10. Reagents and Standards

10.1. Methylene Chloride (DCM)

11. Calibration and Standardization

11.1. Not applicable to this SOP.

12. Procedure

The following policy and procedures are in place to ensure that all samples and chains of custody accepted at PAES are thoroughly inspected and all discrepancies are fully documented. A Client Service Representative shall contact the client in the event that there is any discrepancy involving the condition or the documentation of samples, upon receipt, that may affect the sample's integrity or the analytical process.

A permanent record of sample receipt shall be maintained electronically in the Laboratory Information Management System (LIMS). At a minimum, that record will include: (1) Client Name; (2) Project Name; (3) Date and time of sample receipt; (4) Unique laboratory identification code; (5) Name or initials of the person making the entries; and (6) requested analyses.

12.1. **Sample Acceptance Policy**

A printed copy of the Sample Acceptance Policy (see Attachment I) is forwarded to sample collection personnel as a part of their bottle order shipment.

12.2. **Sample Receipt Procedure**

- Prior to signing shipping documents from courier, ensure that the number of packages listed on the shipping documents corresponds with the number of packages actually delivered.
- The courier must document existing discrepancies before the Sample Receipt Technician accepts the packages. Document the discrepancy on a Non-Conformance Form and forward it to the Client Service Office for immediate action.
- Put on safety glasses and protective gloves before handling, opening, or unpacking packages and coolers that contain environmental samples.
- Prior to cooler inspection and opening, determine if any coolers were received from any DOE or DOD facilities. These coolers and packages must be scanned for radiation using the Radiation Alert Monitor 4 according to the following procedures:

  Turn the Monitor 4 Radiation Instrument to the ON position.

  Check the battery by sliding the range switch to the BATT position. The needle should move to the meter area marked battery at the right side of the meter display. If the meter indicates a low battery, outside of the battery area, turn the instrument off and replace the battery with a 9 volt battery and repeat steps 5 and 6.

  Set the range switch in the X1 position and take a background reading away from the proximity of the packages. The reading should be <0.1mR/hr, as indicated on the dial.

  Record the date, time, battery check, and background reading in the radiation instrument log. This documentation shall be done at least once a day when the radiation instrument is used.

  Hold the back of the instrument toward the cooler beginning at a distance of 1 foot and slowly close the distance to one inch. **If the instrument shows a reading above background, greater than 0.1mR/hr, leave the room and close both doors. The Technical**

Director will take appropriate action to remove and/or isolate the cooler.

- Inspect all coolers and packages for damaged or broken custody seals. Note any discrepancies on a Non-Conformance Form.

- Open the cooler and expose the sample bottles. Hold the back of the radiation instrument over the samples within the cooler to a proximity of 1 inch. If there is no reading above background, greater than 0.1mR/hr, continue with the sample receipt process. **If the instrument shows a reading above background, take the same steps as above.**

- Record the cooler temperature on the cooler receipt form as soon as possible upon opening the cooler. The temperature in the cooler should be above freezing but <6°C. If the temperature is not within those parameters, note the discrepancy on a Non-Conformance Form and indicate whether ice was present or not. Sign the chain of custody on the date of receipt.

  Special NPDES requirements: For samples requiring analyses for NPDES monitoring, a minimum of one bottle from each sampling location will be checked for temperature upon receipt. The temperature will be recorded either on the chain of custody form or the cooler receipt form. A list of NPDES-related clients will be maintained in sample login to assist with identification. This list will be reviewed and updated as necessary, annually at a minimum.

- Inspect each sample and sample label while removing it from the cooler. Samples containers should be intact. At a minimum, sample labels should be completed with the following information:

  Sample Number
  Date and time of Collection
  Site Name

  If samples cannot be properly identified by label inspection, note the discrepancy on a Non-Conformance Form.

- If samples were received in grouped sets, keep the sets grouped together as they are unpacked. If samples were not received in sets, organize them into sets while unpacking.
- Match the sample identifications to the Chain of Custody. Note any discrepancies on a Non-Conformance Form.
- If the project requirements specify data reporting above PAES' standard level, the technician must be aware that each group of 20 samples may have at least one duplicate and one spike set. Note any discrepancies on a Non-Conformance Form.
- Check to see if field and trip blanks are present and identified. Document all missing or potentially missing samples on a Non-Conformance Form.
- Check the Chain of Custody to ensure that all samples are entered and the specific analysis is listed for each bottle. Note all discrepancies on a Non-Conformance Form.
- Check appropriate samples for proper sample preservative using pH paper according to the procedure outlined in Section 12.5 of this Standard Operating Procedure. Samples that are improperly preserved are to be documented using a Non-Conformance Form.

- If samples for Petroleum Forensics are received, follow the instructions on Attachments IX and X to properly identify sample matrix and analysis.
- Complete Cooler Receipt form (Attachment V) for each package opened.
- If a request is made by the client to hold samples, a Sample on Hold Process Sheet (Attachment VII) will be generated and kept with the samples.
- Ensure that all sample receipt documentation is complete.

12.2.1. **Quarantined Soil Receipt Procedure**

This procedure is applicable to all soil samples received from a foreign country or the following US states and/or territories:

- Alabama
- Arkansas
- Arizona
- California
- Florida
- Georgia
- Louisiana
- Idaho
- Mississippi
- New Mexico
- New York
- North Carolina
- Oklahoma
- Puerto Rico
- South Carolina
- Tennessee
- Texas

Client Service personnel are responsible to determine if soil samples coming to the laboratory have originated from a restricted area as defined above.

Client Service will complete a Foreign and Domestic Soil Sample Processing form. (Attachment IV) This form will be printed on orange paper to make it easy to identify.

This form is forwarded to Sample Receiving to alert them of the possibility of sample receipt.

Once the project is received and verified through the Foreign and Domestic Soil Sample Processing form, the samples are logged into the LIMS as usual and a Regulated Domestic and Foreign Soils checklist (Attachment VIII) is completed and placed into the project folder.

When sample labels are applied to the containers, an orange dot is also applied to the sample container lids so that the analysts will know that the samples require special processing.

The orange form becomes part of the permanent file in the waste coordinators office and is used upon project completion to document proper disposal treatment of samples.

Refer to SOP S-PAE-S-002 Regulated Soil Handling for exact procedure for handling regulated soils.

12.2.2. **Weekend Sample Receipt Procedures**

If the Sample Receipt Technician is not present, the weekend analyst will follow the abbreviated sample receipt process below in order to comply with sample holding times. The complete sample receipt process will occur during normal business hours the following Monday.

- If coolers were received from DOD or DOE facilities, scan those coolers for radiation prior to and during opening following instructions above.
- Remove samples that have short holding times for immediate analysis.
- Leave a detailed message with Sample Receipt Technician on what bottles were taken. This may be done via email, voicemail, or written note.

12.2.3. **After Hours Receipt Procedures**

Because the Sample Receipt Technician is not typically present before or after normal business hours, samples that are received during those times shall be placed into the cooler in the Sample Receiving area until the Sample Receipt Technician processes them on the following business day. If a client wishes to have samples processed outside of normal business hours they must make those arrangements with PAES Client Service Office prior to sample delivery. The Client Service Manager must approve those arrangements in writing.

12.2.4. **Sample Receipt Discrepancies**

If there is any discrepancy, problem, or situation with the samples or the above steps that is out of the ordinary, a Non-Conformance Form must be completed immediately and submitted through the proper channels as specified in Section 12.4 of this Standard Operating Procedure.

After all sample receipt documentation has been completed and the samples have been thoroughly examined, the Sample Receipt Technician creates a batch file and begins the process of Sample Log-In in accordance with the LIMS Standard Operating Procedure.

12.3. **Non-Conformance Forms**

The Non-Conformance Form is PAES' primary documentation tool for sample receipt problems or discrepancies that require client contact or corrective action. It is imperative that Non-Conformance Forms are completed accurately and submitted to the Client Service Office in a timely manner.

Any non-standard requirements that were not negotiated in advance of sample receipt such as rapid-turnaround, particular reporting limits, or particular sample disposal instructions must be documented on a Non-Conformance Form.

Non-Conformance Forms are to be completed by the Sample Receipt Technician under the following conditions:

- Broken containers
- Label information inadequate to properly identify samples
- Information on COC inadequate to properly log-in samples
- Missing or extra samples
- Conflict between bottle labels and COC (except as noted below)
- Samples received past holding times (except as noted below)
- Improperly preserved samples
- Any other circumstances that are out of the ordinary

Exceptions: In the following situation, non-conformance forms are not required:

- Samples received for pH analyses are always received outside of the specified holding time; therefore a non-conformance form is not required. A notice in the narrative portion of the final report must indicate that samples for pH were received out of hold.

12.3.1. **Non-Conformance Form Submissions and Handling**

The Client Service Office is to be notified as soon as possible when a Non-Conformance Form is issued. Client Service is to expedite their completion of the Non-Conformance Process and return the documentation to the Sample Receipt Technician as quickly as possible. The original Non-Conformance Form is to be placed in the project file.

12.3.2. **Non-Conformance Completion**

A Non-Conformance Form is to be completed by Sample Receiving personnel as follows:

- Complete date, client name, name of person who received samples, and time of sample receipt.
- Using the lines on the form, ensure adequate information is provided to explain the non-conformance.
- Add additional information, if necessary, on the back of the Non-Conformance Form.
- Submit form to Client Service Office.

The bottom half of the form is to be completed by the Client Service Office as follows:

- Client Service shall document the action taken on the form.
- Client Service will then initial and date the form at the bottom.
- The form will then be returned to Sample Receiving personnel who will check it for completeness.
- Sample Receiving personnel will then log in the samples accordingly, and write the PAES project number on the top of the non-conformance form and place the original in the project file where it becomes a permanent part of the project file.

12.4. **Checking Sample Preservative**

In order for water samples to be considered valid, they must be either cooled and/or chemically preserved according to the type of analyses each sample will undergo. All appropriate samples for analyses that require chemical preservatives shall be checked upon log-in for proper preservative.

The following are not tested for a preservative prior to analysis:

- volatiles in 40 ml vials (either for SW-846 8260, EPA 624 or fuel oxygenates)
- dissolved gases (either light hydrocarbons, permanent gases or MEE; by AM20GAx or RSK175)
- volatile fatty acids (AM21 or AM23G)

All other water samples are to be tested through the use of the narrow range pH paper as per Table 12.4.

Table 12.4 Acceptable pH range by preservative

Preservative	Acid	Neutral	Base
Nitric Acid (HNO_3)	<2	-	-
Sulfuric Acid (H_2SO_4)	<2	-	-

Hydrochloric Acid (HCl)	<2	-	-
Benzyl alkonium chloride (BAK)	-	4-8	-
Unpreserved	-	4-8	-
Sodium hydroxide (NaOH)	-	-	>12
Sodium hydroxide and Zinc acetate (NaOH + ZnAc)	-	-	>12
Trisodium Phosphate	-	-	>10

12.4.1. **Procedures for Checking Sample Preservative**

- Ensure proper personal protective equipment is being worn, i.e. lab coat, safety glasses and protective gloves.
- Open sample container to be tested and pull a small amount of the sample into a disposable pipette.
- Squeeze the aliquot into a clean disposable plastic dish.
- Immerse the pH paper into the aliquot in the plastic dish. Narrow range pH paper will be used. The paper is selected based upon the preservative as presented in Table 12.5.
- Compare the pH paper to the chart to determine the pH range of the sample aliquot.
- The pH check shall be documented in detail by writing the full client sample id, the preservative, the pH and the pH strip lot number next to it on the pH screening form (Attachment VI). If a sample has more than one container with the same preservative, just put an A, B, … suffix after the sample name. For example, if there are 4 sulfide bottles for sample MW-1 the cooler receipt form would have written on it:
  MW-1A NaOH+ pH 12.9
  MW-1B NaOH+ pH 13.0
  MW-1C NaOH+ pH 13.0
  MW-1D NaOH+ pH 12.9
- Dispose of the used pipette and dish. Use a new set for each bottle tested.

12.4.2. **Preservative Check Documentation**

All samples that are checked for preservative will be documented manually on the Cooler Receipt Form and pH screening form. These forms become a permanent part of the record and are maintained for a minimum of five years.

12.5. **Sample Storage**

Samples shall be stored according to the conditions specified by preservation protocols. The storage conditions shall be maintained, monitored, and documented. Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in segregated areas to prevent cross contamination.

12.5.1. **Sample Storage Temperature Documentation and Responsibility**

Samples which require thermal preservation shall be stored under refrigeration or frozen. Digital thermometers that record maximum and minimum temperatures are used to insure that sample integrity is not compromised by transient temperature excursions between thermometer readings. It is the Sample Receipt Technician's responsibility to record that maximum and minimum and to reset thermometer on each work day. A temperature of just above freezing but below 6°C is acceptable for a refrigerator that has been closed overnight. A temperature of -10 to -20°C is

acceptable for a freezer. The temperature logs are to be maintained in the Sample Receipt Technician's Office.

12.5.2. **Corrective Action for Refrigerator Temperature Beyond Control Limits**

If the temperature is outside of the acceptable temperature range limits, the Sample Receipt Technician must immediately notify the Laboratory Manager. Maintenance will be arranged through the Laboratory Manager or his representative. If it is apparent that the proper sample temperature cannot be maintained in the area needing maintenance, then every effort will be made to move samples to another cooler that is functioning within the temperature control limits.

12.5.3. **Samples Requiring Extra Security**

If the Sample Receipt Technician has previously been notified by the PAES' project manager that the samples require high security, the Sample Receipt Technician is to complete an internal chain of custody form for each bottle type using our standard chain of custody form. Once the log-in process is complete the samples are to be placed in a cooler in a secure room. A sample tracking record with the appropriate bottle type circled shall be posted on the outside of the cooler. Only PAES personnel will have access to that area, and will sign out those samples when they are taken for analysis. Employees who have signed out samples will keep them in their possession at all times, or in a secure area, until such time as the analysis is complete or the remainder of the sample(s) and/or extract(s) is/are returned to the original locked location. A sample tracking record form is displayed in Attachment III.

13. Quality Control

13.1. Not applicable to this SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. Not applicable to this SOP.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. All personnel are required to wear appropriate personal protective equipment, which includes, but is not limited to, safety glasses, lab coat and protective gloves. Cut resistant gloves are to be used when using auto-retractable knives or broken glass. Coolers shall be opened in a well ventilated area. If odors are detected upon opening, the cooler shall be closed and moved to an area with a fume hood before proceeding with the sample receipt procedures.

23. Waste Management

23.1. Not applicable for this SOP.

24. Pollution Prevention

24.1. Not applicable for this SOP.

25. References

25.1. Radiation Alert, Operation Manual for the Monitor 4, Monitor 5, and MC1K, 1998.

25.2. The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Sample Acceptance Policy

26.2. Attachment II: Non-Conformance Form

26.3. Attachment III: Sample Tracking Record

26.4. Attachment IV: Foreign and Domestic Soil Sample Processing Form

26.5. Attachment V: Cooler Receipt Form

26.6. Attachment VI: pH Screening Form

26.7. Attachment VII: Sample on Hold Process Sheet

26.8. Attachment VIII: Regulated Domestic and Foreign Soils Checklist

26.9. Attachment IX: Petroleum-Sample Matrix Identification Instructions

26.10. Attachment X: Petroleum-Sample Login Table

27. Revisions

Document Number	Reason for Change	Date
Section 12.3	Added Sample on Hold Process Sheet	1/30/2017
Section 12.3.1	Added references to Regulated Domestic and Foreign Soils Checklist and SOP-S-002 for Regulated Soil Handling.	1/30/2017
Attachments	Added Attachments VII, VIII, IX, X	1/30/2017
Section 12.3.3	Changed Lab Manager to Client Service Manager	2/2/2017
Section 12.3	Added reference to the new Petroleum Forensics Attachments	2/2/2017
Table 12.5	Changed acceptable pH range to >10 for tri sodium phosphate to agree with notes put on the case narratives of final reports.	2/2/2017
S-PAE-C-003-rev.01 Attachment I	Updated with new sample acceptance policy	7/25/2017
S-PAE-C-003-rev.01 Section 12.1	Removed this section, as it is an attachment to the SOP	7/25/2017
S-PAE-C-003-rev.01 Section 12.5	Checking sample preservation-deleted anion and cation analyses	7/25/2017
S-PAE-C-003-rev.02	Added the documentation of pH strip lot numbers to Section 12. Updated the pH Screening Form to include the documentation of the pH strip lot numbers.	7/6/2018

Attachment I
Sample Acceptance Policy

Pace Analytical® Energy Services™	**Document Name:** **Sample Acceptance Policy**	**Document Revised: July 24, 2017** **Page 14 of 1**
	Document No.: **F-PAE-Q-017-rev.01**	**Issuing Authority:** **PAES Quality office**

PAES Sample Acceptance Policy

In accordance with regulatory guidelines, PAES complies with the following sample acceptance policy for all samples received.

If the samples do not meet the sample receipt acceptance criteria outlined below, the Pace facility is required to document all non-compliances, contact the client, and either reject the samples or fully document any decisions to proceed with analyses of samples that do not meet these criteria. Any results reported from samples not meeting these criteria are noted in the case narrative of the final report.

Sample Acceptance Policy requirements**:**

1. Sample containers must have unique client identification designations, and dates and times of collection, that are clearly marked with indelible ink on durable, water-resistant labels. The client identifications must match those on the chain-of-custody (COC);
2. There must be clear documentation on the COC, or related documents such as the Cooler Receipt form, that lists the unique sample identification, sampling site location (city, state), date and time of sample collection, and name and signature of the sample collector;
3. There must be clear documentation on the COC, or related documents, that lists the requested analyses, the preservatives used, sample matrix, and any special remarks concerning the samples (i.e., data deliverables, samples are for evidentiary purposes, field filtration, etc.);
4. Samples must be in appropriate sample containers. If the sample containers show signs of damage (i.e., broken or leaking) or if the samples show signs of contamination, the samples will not be processed without prior client approval;
5. Samples must be correctly preserved upon receipt, unless the method requested allows for laboratory preservation. If the samples are received with inadequate preservation, and the samples cannot be preserved by the lab appropriately, the samples will not be processed without prior client approval;
6. Samples must be received within required holding time. Any samples with hold times that are exceeded will not be processed without prior client approval; Clients are requested to notify PAES client services if samples with short holding times are being shipped;
7. Samples must be received with sufficient sample volume or weight to proceed with the analytical testing. If insufficient sample volume or weight is received, analysis will not proceed without client approval;
8. All samples that require thermal preservation are considered acceptable if they are received at a temperature within 2ºC of the required temperature, or within the method-specified range. For samples with a required temperature of 4ºC, samples with a temperature ranging from just above freezing to 6ºC are acceptable. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. Any samples that are not received at the required temperature will not be processed without prior client approval.
9. Some specific clients may require custody seals. **For these clients**, samples or coolers that are not received with the proper custody seals will not be processed without prior client approval.
10. Samples must pass the radiation screening according to the criteria set forth in the SOP for Sample Receiving.
11. Coolers that arrive with hazard labels on them, for which PAES is not equipped or certified, will not be accepted. A chart listing these hazards is posted in Sample Receiving.

Attachment II

NON-CONFORMANCE FORM

PAES Work Order #: _____

Date: _____ Time of Receipt: _____ Receiver: _____

Client: _____

REASON FOR NON-CONFORMANCE:

_____

_____

_____

_____

_____

_____

_____

_____

_____

ACTION TAKEN:

Client name: _____ Date: _____ Time: _____

_____

_____

_____

_____

_____

_____

_____

Client Service Initials: _____ Date: _____

Attachment III

Sample Tracking Record

Client Name: _____ **Page** _____

Client Project Number: _____

Bottle Type **Circle or Highlight** (Sample Receiving Only)						
	VOA	VFA	TIC	Hydrogen		
	G. Chem.	LLVFA	Soils	CSIA		
	TOC/DOC	Cations	Diss. Gas	Petroleum		
	Sulfide	Anions	Vapor	Molecular		

Sample Receiving only to mark above dotted line

Sample Numbers	**Removed from Storage**			**Bottle**	**Returned or Place**	
	By	**Date**	**Time**	**Type**	**By**	**Date**

Enter Bottle Type From List Above In Proper Column

Sample Tracking Record

Client Name: _____ **Pag**

Client Project Number: _____

Sample Receiving only to mark above dotted line

Sample Numbers	Removed from Storage			Bottle Type	Returned	
	By	Date	Time		By	

Enter Bottle Type From List Above In Proper Column

Attachment IV

Foreign and Domestic Soil Sample Processing

Client Name: _____
Client Contact Name: _____
Project Name: _____
Expected Date of Arrival: _____
State/Country of Origin: _____
PAES WO#: _____
Subcontracted? Yes / No Lab _____
Heat treated/Disposal Date: _____

Comments:
_____
_____
_____
_____
_____
_____
_____

Soil samples received for the above mentioned project, must be disposed of as per USDA specifications.
These samples must be tracked through the lab by using the NEON ORANGE dot stickers. Upon completion of analytical testing, soil samples that have the NEON ORANGE dot stickers, must be kept in the waste room, separated from the others.

After completion of the analytical program and generation of the final report, please return this form to the Waste Coordinator for filing and disposal purposes.

Attachment V

Cooler Receipt Form

Client Name: _____ **Project:** _____ **Lab Work Order:** _____

Shipping/Container Information (circle appropriate response)
Courier: FedEx UPS USPS Client Other:_____
Air bill Present: Yes No
Tracking Number:_____
Custody Seal on Cooler/Box Present: Yes No **Seals Intact:** Yes No
Cooler/Box Packing Material: Bubble Wrap Absorbent Foam Other:_____
Type of Ice: Wet Blue None **Ice Intact:** Yes Melted
Cooler Temperature:_____ **Radiation Screened:** Yes No
Chain of Custody Present: Yes No
Comments:_____

A. **Laboratory Assignment/Log-in** (check appropriate response)

	YES	NO	N/A	Comment Reference non-Conformance
Chain of Custody properly filled out				
Chain of Custody relinquished				
Sampler Name & Signature on COC				
Containers intact				
Were samples in separate bags				
Sample container labels match COC Sample name/date and time collected				
Sufficient volume provided				
PAES containers used				
Are containers properly preserved for the requested testing? (as labeled)				
If an unknown preservation state, were containers checked? Exception: VOA's coliform				If yes, see pH form.
Was volume for dissolved testing field filtered, as noted on the COC? Was volume received in a preserved container?				

Comments:_____
_____

Cooler contents examined/received by : _____ **Date:**_____

Project Manager Review: _____ **Date:** _____

Attachment VI

pH SCREENING FORM

Client _____

Client
PAE
Comp

No.	Sample	Preservative							pH	pH
		HNO_3	H_2SO_4	HCl	None	BAK	NaOH	NaOH+ ZnAc		

Attachment VII

SAMPLE ON HOLD (SAVE) PROCESS SHEET

Requested by: _____ Date Requested: _____

Client Name: _____

Contact person(s): _____

Phone Number: _____

Client project name/number: _____

Sample name(s)/number(s): _____

Save in walk-in cooler: Y or N (circle one) Save till date: _____

Samples will be held for 30 days unless otherwise stated. After 30 days, the sample(s) will be discarded or returned to the client. (If requested in writing by the client.)

Comments: _____

Sample custodian signature: _____ Date: _____

Copy of original COC with Project Number must accompany this form.

F-PAE-Q-025-rev.00, 15JAN2015

Attachment VIII

Regulated Domestic and Foreign Soils Checklist

Project #: _____ **Time:** _____

Initials: _____ **Date:** _____

Origin (Circle One):	**Domestic** **Foreign**
If "Domestic", State of Origin (Circle One)	AL AR AZ CA FL GA ID LA MI MS NC NM NY OK OR SC TN TX WA
If "Foreign", Country of Origin:	_____

Note: Soils from Hawaii and Puerto Rico are of Foreign Origin

Sample analysis will take place at (Circle all that apply):

PAES Subcontract Laboratory

Name of Subcontract Laboratory: _____

	Action	**Completed**
1) Did "Regulated" sticker get placed on Samples?	Regulated sticker must be placed onto each sample container.	Yes / No
2) If samples were sent to a subcontract laboratory, do they hold a valid Soil Permit and Compliance Agreement from the USDA? If not being subcontracted please circle NA.	Subcontract Laboratories are required to hold a valid Soil Permit and Compliance Agreement before we can send soil samples to them. Verify validity by contacting and getting a copy of it	Yes / No / NA
3) Were Samples placed in designate container in Walk-In Cooler?	Regulated samples retained at PAES Laboratory must be stored in designated containers in the Walk-In Cooler.	Yes / No
4) Were there signs of breakage or leakage? If no please complete 5, circle NA for 6 and move to 7. If yes please circle NA for 5, and move to 6.	Check for broken glass or loose soil in the cooler.	Yes / No
5) Were ice and melt water separated from cooler and disposed of properly? (No signs of breakage or leakage)	Foreign and Domestic Sources: Ice and melt water can be disposed of by dumping down the sink.	Yes / No / NA
6) Were ice and melt water separated from cooler and disposed of properly? (Signs of breakage or leakage)	Foreign and Domestic Sources: Ice and melt water must be baked at 140°C then cooled and dumped down the sink. Soils must be disposed of by baking and then placing in appropriate waste barrel.	Yes / No / NA
7) Was the cooler decontaminated?	Soak cooler for 30 minutes with 1:10 bleach solution, drain in sink, let cooler air dry.	Yes / No

Comments:

Attachment IX

How to test if sample matrix is PRODUCT or WATER?

Petroleum Samples

1. Look at the chain-of-custody (COC) to see what analyses are being requested and what matrix is listed on the COC by the client
2. Look at the sample containers to see what the client sent
3. Follow table to determine if 'TEST' needs to be completed
4. If 'TEST' is required, grab a clean 2mL glass vial
5. Grab clean glass pipette and DCM container vial
6. Pipette ~1mL of DCM in the clean 2mL vial
7. Grab a disposable glass pipette (do not use same pipette for different samples)
8. Pipette ~1mL of sample in the clean 2mL vial with the DCM already in it, shake it slightly
9. Determine if there is a separation of phases
10. If there is a line, the sample is a water (bring folder up to PM so that the correct analyses are chosen)
11. If there is no line, the sample dissolved with DCM and matrix is a product
12. If still unsure of matrix, ask analyst or PM to come down and analyze further
13. Place 2mL vial with sample mixture in disposal container when finished

Attachment X

MATRIX TYPE

ANALYSIS

	PRODUCT (Oil/Gasoline/Diesel)	SOIL (Soil/Dirt/Sediment/Wipe)	AQUEOUS (Water)
C3-C36 or Whole Oil or ASTM D3328	TEST	X	X
Fuel Oxygenates Or EPA 1625M	TEST	X	X
Organic Leads Or OrgPb/EDB/ MMT	TEST	X	X
Simulated Distillation Or ASTM D2887	TEST	X	X
Full Scan Or C8+ Hydrocarbon Or ASTM D5739	TEST	OK	TEST
C3-C10 Or PIANO GC/MS	TEST	OK	TEST
PAH	TEST	OK	TEST

ATTACHMENT C-29

WASTE HANDLING AND MANAGEMENT-PACE ENERGY SERVICES PITTSBURGH

Document Information

Document Number: ENV-SOP-PITTS-0023 **Revision:** 00

Document Title: Waste Handling and Management

Department(s): Waste

Previous Document Number: S-PAE-W-002-rev.00

Date Information

Effective Date: 14 Jun 2016

Next Review Date: 19 Oct 2020 **Last Review Date:** 19 Oct 2018

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-PITTS-0023 **Revision:** 00

Title: Waste Handling and Management

All dates and times are in Central Time Zone.

Review: ENV-SOP-PITTS-0023 00 Waste Handling and Management

Review

Name/Signature	Title	Date	Meaning/Reason
Charlotte Washlaski (003467)	Quality Manager	19 Oct 2018, 03:10:29 PM	Reviewed

STANDARD OPERATING PROCEDURE

WASTE HANDLING AND MANAGEMENT

Reference Methods: N/A

Local SOP Number:	S-PAE-W-002-rev.00
Effective Date:	Date of Final Signature
Supersedes:	SOP-ADM14
SOP Template Number:	SOT-ALL-W-002-rev.06

APPROVALS

Charlotte Weidlich — 6/14/2016
Laboratory Quality Manager — Date

Charlotte Weidlich — 6/14/2016
Laboratory Hazardous Waste Coordinator — Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Charlotte Weidlich	Waste Coordinator	2/16/2017

S-PAE-W-002-rev.00

TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. Pace Analytical Services, Inc. (Pace) acknowledges its obligation to the responsible management of the environment and its resources. Pace Management is committed to operating in such a way that meets or exceeds the state and federal laws governing waste management and encourages the use of best practices to reduce, reuse and recycle waste material where possible. This Standard Operating Procedure (SOP) documents the systems, processes and procedures that Pace Analytical Energy Services uses to manage generated wastes.

1.2. It is Pace's policy to minimize the amount of hazardous waste it produces and to reduce the hazardous properties of those wastes whenever practical within regulatory compliance. This can be achieved by periodic auditing of all processes producing hazardous waste; reduction of sample volume delivered by the client; return of excess sample material to clients whenever practical and economical; investigation of new technologies that might require smaller volumes of sample, or produce fewer or less hazardous by-products; implementation of lab cleaning procedures that reduce the volume of cleaning residue; recycling of hazardous materials; and investigation of new treatment technologies that are comprehensively destructive or are effective in reducing the volume or hazardous qualities of the wastes produced.

2. Summary of Method

2.1. Pace facilities that generate waste must initially contact the EPA to obtain an ID number. Each unique type of generated waste is classified and characterized into waste streams according to procedures in 40 CFR 261. The amount of waste the facility generates determines the Generator Status of a lab, which in turn determines how long and how much waste can accumulate. Pace Analytical Energy Services is ultimately responsible for the waste it generates, and is required to obey any and all regulations during the process of creating, accumulating, disposing, and releasing waste to a TSDF for final disposal. Documentation is kept to prove all regulations have been obeyed.

3. Scope and Application

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel responsible for all aspects of waste handling and management.

3.2. This SOP is applicable to all processes at Pace Analytical Energy Services that involve generated waste, and is designed to assist its operations in adhering to regulations set forth in the following federal statutes: Resource Conservation and Recovery Act (RCRA), Clean Water Act (CWA), Toxic Substances Control Act (TSCA), and DOT Title 49, and Transportation (parts 100-199). Particular attention is given to local pretreatment standards covering discharges to publicly owned treatment works (POTW) when performing elementary neutralization on acidic and basic waste. The local standards are based in part upon provisions in the National Pretreatment Standards and Prohibited Discharge Standards.

3.3. The degree to which RCRA regulations apply to Pace facilities is dependent upon the generator status of the operation. Under the federal rules (state requirements may be more stringent or give the classes a slightly different name) there are three different classes of hazardous waste generators based upon the amount of waste generated in a month to month time frame.

3.4. **Waste Generator Class Limits:**

Hazardous Waste Generator Class	Quantity of Hazardous Waste Generated per Month	Generated Monthly Acute Hazardous Waste	Maximum Allowable Hazardous Waste Quantity on-site	Maximum Permitted Waste Accumulation Time
Cond. Exempt Small Quantity	<100kg	<1 kg	<1000kg	Unlimited
Small Quantity	100-1000kg	<1 kg	<6000kg	180 days (270 days if the waste must be sent >200 miles to TSDF)
Large Quantity	>1000kg	>1kg	Unlimited	90 days

3.5. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. Not applicable to this SOP.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Not applicable to this SOP.

8. Definitions

8.1. **Acutely Hazardous Waste -** A waste which is hazardous as identified with an (H) Hazard Code in the lists of Hazardous Waste in 40 CFR Part 261, Subpart D, Sections 261.30, 261.31 and 261.33.

8.2. **Animal and Plant Health Inspection Service (APHIS) -** an agency of the USDA responsible for protecting animal health, animal welfare, and plant health. APHIS is the lead agency for collaboration with other agencies to protect U.S. agriculture from invasive pests and diseases.

8.3. **Clean Air Act -** The Federal Clean Air Act, 42 U.S.C. 7401, and amendments thereto amending 42 U.S.C. 1857 et.seq.

8.4. **Conditionally Exempt Small Quantity Generator -** A generator who produces no more than 100 kilograms of hazardous waste or one kilogram of acutely hazardous waste (or a total of 100 kilograms of any residue or contaminated soil, waste or other debris resulting from the cleanup of a spill, into or on any land or water, or any acute hazardous waste) in a calendar month. The total amount of hazardous waste which may be accumulated on-site is 1000 kilograms.

8.5. **Confined Space –** A space that is large enough and so configured that an employee can bodily enter and perform assigned work; and has limited or restricted means for entry or exit (for example, tanks, vessels, silos, storage bins, hoppers, vaults, and pits are spaces that may have limited means of entry); and is not designed for continuous employee occupancy.

8.6. **Container -** Any device material is stored, transported, treated, disposed of, or otherwise handled.

8.7. **Contingency Plan -** A document setting out an organized, planned, and coordinated course of action to be followed in case of fire, explosion, or release of hazardous waste or hazardous waste constituents which could threaten human health or the environment.

8.8. **Designated Hazardous Waste Storage Area -** Area used to hold hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere. This is the storage area into which hazardous waste from the laboratory (e.g., satellite waste) is moved.

8.9. **DOT -** The United States Department of Transportation.

8.10. **DTSC –** Department of Toxic Substances Control.

8.11. **Elementary Neutralization Unit –** A device which: (1) is used for neutralizing wastes which are hazardous only because they exhibit the corrosivity characteristic defined in 40 CFR 261.22 or are listed in Subpart D of Part 261; and (2) meets the definition of tank, container, transport vehicle, or vessel in 40 CFR 260.10.

8.12. **EPA -** The United States Environmental Protection Agency.

8.13. **EPA Hazardous Waste Number -** The EPA number assigned to each EPA hazardous waste identified in 40 CFR Part 260, Subpart D - Lists of Hazardous Wastes.

8.14. **EPA Identification Number -** The site-specific number assigned to each generator, transporter, and TSDF upon approval of a notification form.

8.15. **Federal Clean Water Act -** 33 U.S.C. 1251, et. Seq.

8.16. **Foreseeable Emergency -** Any fire, explosion, or sudden or non-sudden release of hazardous waste or hazardous waste constituents to the air, soil, or surface water, which could threaten human health or the environment.

8.17. **Generator -** Any person, by site who owns or operates a facility where hazardous waste is generated, i.e. Pace Analytical Services (Pace).

8.18. **Hazardous Waste Coordinator -** The Pace employee responsible for creating, guiding, and implementing all hazardous waste management operations.

8.19. **Hazardous Waste -** As defined in 40 CFR Part 261, Subparts B and C, a solid, semi-solid, liquid or contained gaseous waste, or any combination of these wastes.

8.19.1. Which, because of either quantity, concentration, physical, chemical, or infectious characteristics may:

8.20.1.1. Cause or contribute to an increase in mortality or an increase in irreversible or incapacitating reversible illness; or

8.20.1.2. Pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed of or otherwise mismanaged.

8.19.2. Or which has been identified as having a characteristic of hazardous waste by the EPA using the criteria established under 40 CFR Part 261, Subpart C, or as listed under Sections 261.31, 261.32, 261.33, and 261.34. Such wastes include, but are not limited to, those which are reactive, toxic, corrosive,

ignitable, irritants, strong sensitizers or which generate pressure through decomposition, heat or other means. Such wastes do not include radioactive substances that are regulated by the Atomic Energy Act of 1954, as amended. A waste is considered hazardous if it is listed or it fits into one of four categories. These categories are as follows:

8.20.2.1. Ignitable – (40 CFR 261.21, Waste Code D001) A flash point of less than 60°C/140°F.

8.20.2.2. Corrosive – (40 CFR 261.22, Waste Code D002) A pH of less than 2.0 or greater than 12.5.

8.20.2.3. Reactive – (40 CFR 261.23, Waste Code D003) Reactive wastes exhibit one or more of the following characteristics:

8.20.2.3.1. It is unstable and can undergo a violent change without detonating.

8.20.2.3.2. It can react violently with water.

8.20.2.3.3. When mixed with water it can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

8.20.2.3.4. It is cyanide or sulfide bearing waste that, when exposed to pH conditions between 2.0 and 12.5, can generate gases, vapors, or fumes that can present a danger to human health or the environment.

8.20.2.3.5. It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

8.20.2.3.6. It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

8.20.2.3.7. It is a forbidden explosive as defined in 49 CFR 173.51, or a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.88.

8.20.2.4. Toxic – (40 CFR 261.24, Waste Codes D004-D043) A solid waste that contains a toxic concentration of a contaminant listed in 40 CFR 261.24, Table 1. A toxic waste is given any and all D-codes that apply to the particular material.

8.20. **Hazardous Waste Constituent -** A substance, compound, or element listed as hazardous waste in EPA 40 CFR 261.

8.21. **Lab Pack Material -** A hazardous waste that does not match a listed Pace waste stream category.

8.22. **Large Quantity Generator (LQG) -** Any generator who generates at a rate greater than 1000 kilograms of hazardous waste per month.

8.23. **Manifest -** As defined in 40 CFR Part 262, Subpart B, namely “the form used for identifying the origin, quantity composition, routing and destination of hazardous waste”.

8.24. **Plant Protection and Quarantine (PPQ) –** A program within APHIS which attempts to safeguard agriculture and natural resources in the U.S. against the entry, establishment, and spread of animal and plant pests and noxious weeds.

8.25. **Regulated Soil –** Soil from foreign countries, U.S. territories and areas within states that are under Federal quarantine that can be moved into or through continental U.S. only if conditions and safeguards prescribed by the USDA and APHIS are met.

8.26. **Sample -** Except as provided below in 8.27.7.2.3, any solid waste, water, soil, or air that is collected for the sole purpose of being tested to determine its characteristics or composition.

8.26.1. Samples are not subject to any requirements of 40 CFR Part 261.5 or Parts 262 through 267 or Part 270 or Part 124 or to the notification requirements of Section 3010 of RCRA, when:

8.27.1.1 The sample is being transported to a laboratory for the purpose of testing; or

8.27.1.2 The sample is being transported back to the sample collector after testing; or

8.27.1.3 The sample is being stored by the sample collector before transport to a laboratory for testing; or

8.27.1.4 The sample is being stored in a laboratory before testing; or

8.27.1.5 The sample is being stored in a laboratory after testing but before it is returned to the sample collector; or

8.27.1.6 The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary).

8.26.2. In order to qualify for the exemption in 8.27.1.1 and 8.27.1.2 above, a sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must:

8.27.7.1. Comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements; or

8.27.7.2. Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:

8.27.7.2.1. Assure that the following information accompanies the sample:

8.27.7.2.1.1. The sample collector's name, mailing address, and phone number;

8.27.7.2.1.2. The laboratory's name, mailing address, and phone number;

8.27.7.2.1.3. The quantity of the sample;

8.27.7.2.1.4. The date of shipment; and

8.27.7.2.1.5. A description of the sample.

8.27.7.2.2. Package the sample so that it does not leak, spill, or vaporize from its packaging.

8.27.7.2.3. This exemption does not apply if the laboratory determines that the waste is hazardous but the laboratory is no longer meeting any of the conditions stated in 8.27.1 above.

8.27. **Satellite Waste or Laboratory Satellite Waste -** Hazardous waste generated by Pace that is at or near any point of generation and under the control of the operator. Satellite accumulation provisions allow generators to accumulate up to 55 gallons of hazardous waste (or 1 quart of acute hazardous waste) in containers without starting the storage clock as described in Section 3.4.

8.28. **Satellite Waste Container -** Any portable device used to accumulate laboratory generated waste prior to transfer to the hazardous waste storage area.

8.29. **Small Quantity Generator (SQG) -** A generator who produces no more than 1000 kilograms of hazardous waste (or a total of 1000 kilograms of any residue or contaminated soil, waste or other debris resulting from the cleanup of a spill, into or on any land or water, or any acute hazardous waste) in a calendar month. The total amount of hazardous waste which may be accumulated on-site is 6000 kilograms.

8.30. **TSDF -** A Treatment/Storage/Disposal Facility.

8.31. **Universal Waste –** Commonly used items that are hazardous but can be recycled. These include fluorescent lights, computer monitors, etc.

8.32. **Waste Stream -** The generic profile of chemical and physical properties that satellite wastes exhibit.

9. Equipment and Supplies (Including Computer Hardware and Software)

The following equipment is mandatory under RCRA guidelines unless otherwise denoted. Periodic review (not to exceed monthly) of availability of equipment and supplies below should be conducted to maintain an adequate and viable supply.

9.1. **Chemical Spill Control Neutralizers**: The waste room stores three types of bulk dry spill neutralizers: solvent, acid and base. They may be utilized by placing the dry neutralizer onto a liquid chemical spill. Neutralization is indicated by a prevalent color change.

9.2. **Communication Device**: Required for emergency notification of spill, fire, etc.

9.3. **Drums**: Common types of waste drums used for storing and shipping hazardous wastes are polyethylene, steel-polyethylene lined, and steel. Sizes are typically 5gal, 15gal, 30gal, and 55gal. Drums used for liquids typically are closed top with an opening to pour the solvent through a funnel, while drums used for solids or lab packs are open-top.

9.4. **Fire Alarm Pull Station**: A fire alarm pull station must be in close proximity to the hazardous waste room. The alarm may be activated by pulling the switch. Other alarm systems may be utilized as long as all personnel are trained on the procedures.

9.5. **Fire Extinguisher**: An extinguisher with a rating appropriate to the waste being stored in the area must be in close proximity to the hazardous waste room.

9.6. **Labels**: A multitude of labels are provided to ensure compliant labeling. They may be purchased or prepared manually.

9.7. **Liquid Chemical Neutralizers**: Liquid chemical neutralizers (base and acid) may be used to neutralize a contained hazardous liquid. This may be done by slowly adding the neutralizer to the liquid.

9.8. **Spill Control Pads**: Spill pads are used to soak up hazardous liquids. They do not neutralize spills. They are especially effective for cleaning up oily materials. Various pads are available for aqueous and petroleum based liquids.

9.9. **Spill Control Pillows**: Spill pillows may be used to soak up large amounts of liquid chemical spills. No neutralization occurs.

9.10. **Spill Dikes**: vary depending on the size and type of room: Their purpose is to encircle a spill, barring the spread of a hazardous chemical. They will also absorb liquids, but do not neutralize spills.

10. Reagents and Standards

10.1. Not applicable to this SOP.

11. Calibration and Standardization

11.1. Not applicable to this SOP.

12. Procedure

12.1. All Pace facilities that generate hazardous waste must have a Generator's US EPA Identification Number. The ID number is obtained through the applicable EPA region's office by completing EPA form 8700-12, and must be completed before generating any hazardous waste.

12.1.1. Pace only utilizes transporters and treatment, storage, or disposal facilities (TSDFs) that have EPA identification numbers for hazardous waste handling and meet the TSDF transfer requirements.

12.1.2. A new ID number is necessary when changing locations as the number is tied to the facility address.

12.1.3. This facility's US EPA Identification Number is PAD987397239.

12.2. The laboratory generates wastes originating from several source types: materials and chemicals used to prepare and analyze samples (e.g., solvents, acids), unconsumed liquid and solid samples, certain types of batteries, mercury from lamps and broken thermometers and automobile waste. Unconsumed samples may include laboratory-contaminated sample residue (both liquid and soil) generated as part of digestion, extraction, etc., procedures used to prepare samples for analysis.

12.2.1. This facility is classified as a Conditionally Exempt Small Quantity Generator.

12.3. Hazardous waste classification is the most critical step in establishing an effective, compliant waste-handling program. Laboratory wastes are classified using the criteria set forth under RCRA for ascertaining non-hazardous versus hazardous status, and this criterion is listed in the definition of hazardous waste in 8.20.

12.4. The following are the waste streams resulting from materials and chemicals used in the laboratory operation. Applicable information for each is given pertaining to packing, labeling, or listing on a manifest. A description of how the wastes are created, and the preferred method of final disposal for each, is included. The overriding principle in hazardous waste classification is application of a conservative formula based on all known or suspected hazards related to a waste material. While this formula may result in some materials being disposed as hazardous when in fact, they are non-hazardous (e.g., false positive), the formula will not be compromised in the interest of reducing the amount of waste produced. This will minimize any risk of a material being disposed of erroneously as non-hazardous when it, by definition, is a hazardous waste.

12.4.1. **Corrosive waste** is generated in the majority of the departments in the laboratory. This waste stream consists primarily of spent or excess aqueous reagent solutions generated from preservatives, acid digestions of metals, impinger solutions or other corrosive solutions generated in the course of analysis. The predominant corrosives include hydrochloric acid, nitric acid and sulfuric acid, but corrosives also include bases. Varying concentrations of metals may be present dependent upon the composition of the reagents added. This waste stream only has the hazardous quality of being corrosive; therefore, if a waste has any additional hazardous waste quality (e.g., Toxic or Ignitable) it cannot be mixed with this stream. This stream is most commonly treated onsite.

Corrosive Waste	
DOT Shipping Name	RQ Waste, Corrosive Liquid, N.O.S (i.e. corrosive material)
EPA Waste #	D002
Container	Various
Average pH	<2.0, >12.5
Disposal Method	Treatment by Neutralization
Label	Corrosive

12.4.2. The **Chlorinated Waste Stream** consists primarily of methylene chloride with a very small amount of other organic solvents derived from extraction procedures performed on samples and from rinsing glassware.

Chlorinated Solvents	
DOT Shipping Name	RQ Hazardous Waste, Toxic Liquid, N.O.S (i.e. dichloromethane, acetone, methanol)
EPA Hazard Codes	U080, F001/F002
Container	Various
Average pH	7.0
Disposal Method	Haz Waste Hauler
Label	Toxic, Chlorinated

12.4.3. . The **Product Sample Waste Stream** consists primarily of unused client product samples. This stream is containerized and placed with a TSDF for disposal.

12.5. Some waste can become complicated when attempting to classify as non-hazardous or hazardous due to the list of hazardous constituents contained in sections 40 CFR 261.30-261.35 including a majority of analytes of interest routinely analyzed in Pace laboratories. Definitions have been established for each of the F, K. P, and U lists covering hazardous waste originating from non-specific sources, specific sources and discarded commercial chemical products, off-specification species, container residues, and spill residues. The application of listed hazardous wastes and substances is intended for manufacturing processes involving pure products, by-products, wastes generated as part of the production process and cleanup of materials contaminated from a spill of the listed commercial chemical product or manufacturing chemical intermediate. See Attachment I for common F-listed wastes.

12.5.1. Hazardous waste classification of unconsumed samples by listed hazardous waste criteria is not commonly applied in laboratory operations. Examples of sample types which would be identified as listed hazardous wastes include the following:

12.5.1.1. Samples containing 5% or more (by volume) of halogenated and non-halogenated “spent solvents:” (e.g., drum sample with > 10% TCE);

12.5.1.2. Pure product and two phase solution samples containing a listed chemical product or manufacturing intermediate (e.g., drum sample);

12.5.1.3. Samples from specific sources listed in section 261.32 (e.g., bottom sediment sludge from the treatment of wastewaters from wood-preserving processes that use creosote and/or pentachlorophenol - K001);

12.5.1.4. Samples representing any residue or contaminated soil, water or other debris resulting from the cleanup of a spill into or on any land or water of any commercial chemical product or manufacturing chemical intermediate having a generic name listed in section 261.33, or any residue or contaminated soil, water or other debris resulting from the cleanup of a spill, into or on any land or water, of any off-specification chemical product and manufacturing chemical intermediate which, if it met specifications, would have the generic name listed in section 261.33.

12.5.2. For the wastes listed in 12.5.1.1 and 12.5.1.2, disposal can be achieved by individually lab packing them or combining with other compatible hazardous wastes.

12.5.3. The remaining two sample types in 12.5.1.3 and 12.5.1.4 would also require lab packing for disposal. However, it is important to note that in order for the laboratory to ascertain that the samples were derived from a specific listed source or from a spill of a listed chemical, they must be so informed by the industrial concern or lead agency (e.g., EPA, state regulators) submitting the sample for analysis. If a water or soil sample contains a listed hazardous waste substance whose origin is unknown or

uncertain to the lead agency, then that sample is not classified as a listed hazardous waste. Rather in this case, determination of a hazardous waste classification can only be obtained by the waste exhibiting a characteristic of hazardous waste (e.g., ignitability, corrosivity, reactivity).

12.5.4. Due to the fact that the majority of samples analyzed by Pace do not meet the well-defined criteria for identifying "listed" hazardous waste, disposal classification of unconsumed samples will be based upon characteristics of hazardous waste:

12.5.4.1. Non-Hazardous - Analysis results indicate an absence of contaminants; unless contaminants listed under the hazardous disposal categories are parts of the requested sample analysis.

12.5.4.2. Hazardous – Analysis results indicate presence of contaminants (Attachment III) or sample analysis requires hazardous materials and contaminants. Samples in this category are segregated from others and disposed of as hazardous according to laboratory procedures.

12.5.4.3. PCB Waste - Generated exclusively by samples contaminated with greater than trace levels of polychlorinated biphenyls ($\geq$ 50ppm). Samples containing 50ppm (total) or higher of PCBs must be segregated and disposed of as PCB waste.

12.5.4.4. Waste Oil/Paint - Samples which are predominantly of an oil matrix (e.g., highly viscous organic liquid) or paint (solvent and pigment blend) are segregated and disposed in a separate container. Though these samples are defined as nonhazardous, oil samples are a special case and never disposed as nonhazardous.

12.5.5. USDA-APHIS-PPQ Regulated Soils (Regulated Soils) are a special case of sample strictly controlled under quarantine regulations 7 CFR 330 because they can readily provide a pathway for a variety of dangerous organisms throughout the United States. The movement of soil into the United States from foreign sources and from certain regulated areas within the continental U.S. is restricted unless permitted by APHIS under specific conditions and safeguards.

12.5.5.1. Any laboratory that plans to handle Regulated Soils must have an approved Soil Permit or Regulated Soil Agreement from USDA-APHIS-PPQ.

12.5.6. Though Pace is obligated to ensure nonhazardous discharge complies with requirements set by applicable publicly owned treatment works (POTW), Pace is not obligated to run every available analysis on every sample for proper waste classification. Consequently, samples are characterized according to the preservatives added, the requested analytical testing data, and any knowledge of the sample provided by the client. When sample analysis is canceled/not completed, those untested samples only need be characterized by the preservatives added and any knowledge of the sample that is obtained by the client.

12.6. Consolidation of wastes from the laboratory proceeds via two distinct routes covering either laboratory-generated hazardous wastes or excess unconsumed samples.

12.6.1. Laboratory Accumulation and Satellite Waste Containers

12.6.1.1. Waste materials from routine lab procedures are collected in containers of appropriate construction, placed in convenient locations at the point of generation. Under RCRA guidelines, these are defined as satellite containers.

12.6.1.2. The amount of hazardous waste stored in the laboratory at the individual satellite areas cannot exceed 55 gallons (liquid) or 550 lbs (solid) per waste stream, for non-acute hazardous waste.

12.6.1.3. Satellite waste containers must be labeled in accordance with all regulations, including:

12.6.1.3.1. Designation of the contents to be hazardous waste with the words "Hazardous Waste" clearly legible.

12.6.1.3.2. The waste stream description (e.g., acid waste).

12.6.1.3.3. A hazard label (e.g., corrosive).

12.6.1.4. The satellite containers must be maintained such that evolution of chemical vapors is precluded. This requires that the container be closed at all times, except when adding or emptying hazardous waste to and from the container.

12.6.1.5. The most critical point in the waste handling system is when a person (e.g., analyst, technician) places a waste material into a satellite container. Here, the characteristics or listing of the waste and the waste stream must both be known to match. For this reason, only material from approved procedures should be placed in the compatible satellite containers. All materials from experimental procedures, unknown or out of the ordinary sources, or from spill cleanups must be characterized and described to the Hazardous Waste Coordinator, who determines the proper method of disposal.

12.6.1.6. Full satellite containers must be transferred to the proper accumulation drum within 3 calendar days. Lab collection containers must not be filled to the top of the opening. Space must be left to prevent splashing of hazardous material when containers are emptied and to allow for expansion and contraction within the drum during transport.

12.6.1.7. Satellite containers for liquid hazardous waste must have secondary containment made of material that could successfully contain the entire satellite container's contents.

12.6.2. Unconsumed Sample Disposal

12.6.2.1. Client samples are stored on-site for a defined period of time after the final analytical report is generated and prior to sample disposal. The purpose of sample storage is to provide the client time to review the analytical report and determine if the samples require additional testing or need to be returned to the client. Samples are not considered a waste during this time according to 40 CFR 261.4(d)(vi).

12.6.2.1.1. Sample storage time is 30 days from final report. Other sample storage hold times may be assigned for specific contractual requirements.

12.6.2.1.2. During sample storage, the process and sample status must be obvious to employees, customers and auditors. This transparency is imperative to ensure samples are considered active test specimens to be retained until they are categorized as a waste for disposal.

12.6.2.2. Samples which cannot be returned to the client for disposal are characterized according to section 12.4. Samples are characterized by one of three methods:

12.6.2.2.1. Analytical results are evaluated against characterization criteria established for the sample waste stream. The samples which exhibit waste characteristics as previously outlined are segregated and denoted per laboratory/facility policies. OR:

12.6.2.2.2. Samples are scanned out of EPIC Pro (LIMS), if available, as RCRA nonhazardous to be disposed as the waste stream is normally handled unless the sample tested over RCRA

limits, in which the LIMS will prompt the employee that the sample is scheduled for Hazardous disposal, and is segregated from the nonhazardous samples. OR:

12.6.2.2.3. Samples of a certain type are all "assumed" to be hazardous, and all are placed into an accumulation drum with all required RCRA labeling for that waste stream.

12.6.2.3. Samples are pulled from storage and disposed of according to local lab processes. The analysts are responsible for the disposal of analyzed samples that are non-hazardous.

12.7. Transferring Satellite Waste to the Waste Storage/Accumulation Area

12.7.1. All transfers of satellite waste to waste drums must be made by the Hazardous Waste Coordinator or designated, trained personnel. When a satellite waste container is full, the Hazardous Waste Coordinator, or designee must be notified. Regular disposal events may be scheduled to dispose satellite waste on a continuous basis.

12.7.2. Find the correct waste drum by referring to the Hazardous Waste placard and hazard label. Mixing solvents that are not compatible could result in a hazardous reaction.

12.7.3. Ensure there is enough capacity in the drum to hold all the content that will be dispensed.

12.7.4. Check to make sure there is a ground connection before opening a solvent waste drum.

12.7.5. Open and slowly pour the contents of the satellite container into the proper waste drum using an appropriate solvent resistant funnel.

12.7.6. Replace the cap on the bunghole and carefully screw the cap on but do not tighten the cap.

12.8. Unconsumed Soil Samples

12.8.1. All soil samples are to be placed into the soil drum in the waste room after the 30 day after final report storage requirement. Separate according to USDA requirements and heat to 105C if necessary.

12.9. Elementary Neutralization

12.9.1. Dilute corrosive solutions (e.g., preserved metals samples) which do not exhibit any hazardous characteristics other than being corrosive, may be neutralized. Elementary neutralization is exempt from RCRA permitting requirements for on-site hazardous waste treatment. While exempt under RCRA guidelines, before utilizing this practice to reduce off-site treatment or disposal of wastes, local pretreatment and discharge standards must be met for publicly owned treatment works (POTW).

12.9.2. The discharges listed below are prohibited under the National Pretreatment Standards and Prohibited Discharge Standards:

12.9.2.1. Pollutants causing fire or explosion (waste with a flashpoint < 60^{o}C);

12.9.2.2. Corrosive wastes with pH less than 2 or greater than 12.5;

12.9.2.3. Solid or viscous pollutants that could potentially block the system;

12.9.2.4. Oxygen-demanding pollutants;

12.9.2.5. Wastes which generate toxic gases.

12.9.3. Wastes that are generated by the laboratory that have a pH of less than 2 and greater than 12.5 shall be neutralized to a pH of not less than 6 and not greater than 8 prior to sewer disposal using the following procedure. Acids and bases shall always be neutralized separately.

12.9.3.1. Put on safety glasses, inner gloves and over gloves (PVC).

12.9.3.2. Place a large plastic bucket into a fume hood and turn the fume hood on.

12.9.3.3. Set the sash height according to the arrows on the front of the hood.

12.9.3.4. Place approximately 1 liter of tap water into the bucket.

12.9.3.5. Slowly pour the acid or base into the bucket.

12.9.3.6. Add soda ash (acids) or sodium bicarbonate (acids or bases) approximately a tablespoon at a time, allowing the reaction to stop prior to adding more.

12.9.3.7. Check the pH frequently.

12.9.3.8. When the pH meets the above specifications, the solution is amenable to sewer disposal according to the following instructions.

12.9.4. The only laboratory chemicals and waste that are amenable to sewer disposal are the ones that are deemed non-hazardous, or corrosive wastes that have been treated. Non-hazardous aqueous samples are amenable to sewer disposal. Any time samples or neutralized wastes are disposed using the sanitary sewer; the tap shall be run for a minimum of fifteen minutes.

12.9.4.1. Turn on the cold water in the sink where the waste will be poured 5 minutes prior to the start of disposal to flush the lines.

12.9.4.2. While keeping the cold water on, slowly pour the waste into the sink. Triple-rinse the waste container. The container can then be placed in the trash.

12.9.4.3. When all of the corrosive wastes have been disposed, keep the water running for an additional 15 minutes to flush the lines before turning off the water.

12.10. Waste Storage Container Requirements

12.10.1. Drums in the hazardous waste storage area are labeled consistent with both DOT and EPA regulations concerning hazardous materials and wastes.

12.10.2. Labels must be easily visible and legible (e.g., a drum must not be labeled and then placed in such a way that the label cannot be seen).

12.10.3. The Accumulation Start Date must be recorded on the drum. The date should reflect the first time waste was added to the drum and not the date when the waste was generated in the laboratory.

12.10.3.1. Once a waste is removed from the point of generation to a hazardous waste staging area, the clock is started for storage time prior to disposal.

12.10.3.2. Drums must be picked up by TSDF for disposal before accumulation time exceeds RCRA requirement for lab's generator status.

12.10.4. The hazardous waste staging room must be arranged in such a fashion to assure direct access pathways in the event of foreseeable emergency and for safe waste transfer. A minimum aisle space of three feet must be maintained at all times to access hazardous waste containers.

12.10.5. All hazardous waste drums and containers must be securely closed when not in use. All volatile and flammable hazardous waste liquid containers must be securely grounded at all times. Drums containing these liquids should also be manipulated with non-sparking tools and fitted with a drum venting bung, to assure that excess pressure build-ups are safely released.

12.10.6. All liquid waste stream containers must be provided with secondary containment devices. Such containment devices must be made of materials compatible with each waste, and they must be free of leaks. The waste storage room may act as secondary containment as long as the room has been constructed to safely and effectively contain a hazardous waste spill.

12.10.6.1. Secondary containers must exceed the total volume of the largest container stored in each containment device for indoor storage.

12.10.7. Compatibility of wastes must be considered in arranging storage areas. For example, acid waste should never be stored adjacent to basic waste, particularly cyanide wastes. Further examples are outlined in 40 CFR 264, Appendix V.

12.10.8. The hazardous waste staging area is controlled so unauthorized personnel are not able to access the room or contents.

12.10.9. The maximum volume of acutely hazardous waste (e.g., P-listed wastes) that can be accumulated in the laboratory is one quart. The volumetric measurement of one quart is based upon container size in which the waste is stored and not the actual amount (volume) of waste present. An example of how this one quart limit can inadvertently be exceeded involves the disposal of a neat standard of 2,4-dinitrophenol into a one gallon bottle. While the neat standard itself may only constitute 1-2mL, the volume as defined under RCRA would be one gallon, thus the laboratory would be out of compliance.

12.11. Waste Documentation and Reporting

12.11.1. All drums containing hazardous waste are recorded in a logbook or database. The information contained in this log is useful when filling out EPA biennial reports and for retaining an accurate description of how much waste has been accumulated. The following information is entered into the logbook/database;

12.11.1.1. The drum number;

12.11.1.2. The date filling the drum was started;

12.11.1.3. The drum capacity (e.g., 55-gallon, etc.);

12.11.1.4. The manifest number associated with the drum's disposal.

12.11.2. The following hazardous waste records must be maintained a minimum of five years and should be retained indefinitely:

12.11.2.1. Drum tracking logs;

12.11.2.2. Sample Reports;

12.11.2.3. Sample disposal information and waste records on computer disc;

12.11.2.4. Analytical records relating to sample waste stream profiling and characterization;

12.11.2.5. Labpack inventory logs;

12.11.2.6. Biennial Reports, Exception Reports, or other reports filed for compliance reasons;

12.11.2.7. Records related to unresolved enforcement action must be retained indefinitely until such a time that the matter is resolved;

12.11.2.8. Facility Certificates of Destruction or Recycling.

12.11.3. A Waste Manifest is the documentation form that must accompany all shipments of hazardous waste while in transit.

12.11.3.1. A Hazardous Waste Manifest Cover Sheet (*F-ALL-W-DRAFT-rev.00 or local replacement*) will be utilized to ensure waste transfer from generator to TSDF fulfills all legal requirements.

12.11.3.2. The manifest is be signed and dated by a DOT trained Pace employee responsible for the shipment and the transporter. The transporter will leave 2-3 of these "two-signature page" copies of the manifest.

12.11.3.3. Within 35 days you will receive a three-signature page (generator, transporter, facility) showing the waste reached its intended destination.

12.11.3.3.1. If you do not receive the three signature page within 35 days of shipment, call the facility to find out why you have not received it. If you do not receive the three-signature page within 60 days you must file an exception report.

12.11.3.4. All manifests must be kept for a minimum of three years.

12.11.4. The central accumulation staging room must have a documented inspection weekly and satellite waste containers must have documented inspection as part of the monthly laboratory inspection. The inspections should ensure all regulations are obeyed; see sections 12.10 and 12.6.1 for accumulation storage and satellite rules.

12.11.4.1. A record of the inspections must be kept in an inspection log or summary.

12.11.4.2. Records must be maintained for at least three years from the date of inspection. At a minimum, the records must indicate:

12.11.4.2.1. The date and time of the inspection;

12.11.4.2.2. The name and signature of the inspector (typically will be Hazardous Waste Coordinator);

12.11.4.2.3. A notation of the observations made (can be in a check-off format, e.g., fire extinguisher: charged _X_ requires recharging __);

12.11.4.2.4. The date and nature of any repairs or other remedial actions.

13. Quality Control

13.1. Not applicable to this SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. The analyst must read and understand this procedure with written documentation maintained in his/her training file.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A formal safety plan is also available to all employees. Gloves and eye protection should be routinely worn when handling reagents or samples. When qualified employees are transferring wastes, additional protection such as goggles, face shield, and lab apron are recommended.

22.2. Safety Data Sheets are located in a central location and should be consulted prior to handling samples and standards.

22.3. A hazard assessment must be completed for waste areas to ensure proper PPE are utilized.

23. Waste Management

23.1. Not applicable to this SOP.

24. Pollution Prevention

24.1. Not applicable to this SOP.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. 40CFR261-268, Code of Federal Regulations Chapter 1, Subchapter I – Solid Waste.

25.3. 29CFR171-174, Code of Federal Regulations, Transportation.

25.4. Federal Plant Pest Regulations, Part 330.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: RCRA Requirements for Labs as a Function of Generator Status.

26.2. Attachment II: Hazard Codes for Common F-List Wastes (solvents).

26.3. Attachment III: TCLP Contaminant List with Concentration Limits.

26.4. Attachment IV: Hazardous Waste Label for Accumulation Drum (example).

26.5. Attachment V: Satellite Container Inspection Form (example).

26.6. Attachment VI: Waste Accumulation Room Inspection Form (example).

26.7. Attachment VII: Hazardous Waste Manifest Cover Sheet.

27. Revisions

Document Number	Reason for Change	Date
SOT-ALL-W-002-rev.05	Section 8.20.2.4: Added definition of Toxic. Section 12.5.5: Added more detail to USDA Regulated Soils. Section 12.6.2.1: Defined sample storage hold times. Added Section 12.11.3.1 and Attachment VIII– Hazardous Waste Manifest Cover Sheet info. Clarity added throughout that liquid waste containers need secondary containment, not all waste containers. Referenced sections and regulations corrected.	14Nov2013
SOT-ALL-W-002-rev.06	Section 8.27: reorganized for clarity and fixed reference section errors. Section 8.28: updated definition. Sections 12.5.5 and 12.5.5.1: updated to reference new, local version of Regulated Soil Handling SOT. Section 12.5.6: clarification added that Pace labs must obey discharge rules. Section 12.6.2.2.4: added red text paragraph regarding removal of sample labels for ESI clients. Removed old Attachment IV (soil regulations map) because it was outdated and this topic is covered in Regulated Soil SOP.	24Mar2015

Attachment I: RCRA Requirements for Labs as a Function of Generator Status

Requirement (40CFR)	CESQG	SQG	LQG
Waste Determination (262.11)	Applicable	Applicable	Applicable
Generation Rate Limits (261.5 and 262.34)	<100 kg/mo	100-1,000 kg/mo	1,000 kg/mo or greater
Accumulation Quantity Limit w/o Permit (261.5 and 262.34)	Not to exceed 1,000 kg at any time. Not to exceed 1 kg acute at any time	not to exceed 6,000 kg at any time	No limit
Accumulation Time (261.5 and 262.34)	No limit	180 days or 270 if waste is to be transported over 200 miles.	90 days
EPA ID Number (262.12)	Not required***; possible state requirement	Required	Required
Mark Containers with Start Date (262.34)	Not applicable	Applicable	Applicable
Mark Containers “Hazardous Waste” (262.34(a))	Not applicable	Applicable	Applicable
Air Emission Standards 40 CFR 265 Subpart CC	Not applicable	Not applicable	Applicable
Satellite Accumulation (262.34(c))	Not applicable	Applicable	Applicable
Use Manifests (262, Subpart B)	Not required; possible state requirement	Required	Required
Exception Reporting (262.42)	Not required	Required after 60 days. No TSDF notification requirement.	Required after 45 days. Notification of TSDF within 35 days.
Biennial Report (262.41)	Not required	Not required; possible state requirement	Required
Contingency Plan (265, Subpart D)	Not required, but OSHA (29 CFR 1910.38) requires emergency planning	Basic planning required in accordance with the standards in 262.34(d)(4) and (5) and 265, Subpart C as well as OSHA regulations	Full written plan in accordance with 265 Subpart D, is required by 262.34(a)(4) and OSHA regulations
RCRA Personnel Training (262.34 and 265.16)	Not required, but recommended	Basic training required by 262.34(d)(5)(iii)	Full compliance with the training requirements in 265.16 is required by 262.34(a)(4)

Requirement (40CFR)	CESQG	SQG	LQG
Storage Requirements (without permit) (262.34 and 265)	None, but OSHA regulations under 29 CFR 1910, Subparts H and N, apply, particularly 29 CFR 1910.106	Compliance with technical standards in Part 265, Subparts I and J; for containers and tanks is required by 262.34(d)(2) and (3) and OSHA regulations	Compliance with technical standards in Part 265, Subparts I, J, W, and DD, is required by 262.34(a)(1) and OSHA regulations
Recordkeeping Requirements (262.40)	Waste determinations and generation log required (notification of regulated waste activity, training records, manifests, and land disposal restriction notifications recommended)	Notification of regulated waste activity, waste determinations, generation log, manifests, land disposal restriction notifications, exception reports, and correspondence with local emergency responders (written contingency plan, weekly container inspection & periodic equipment maintenance logs, and RCRA training records recommended)	Notification of regulated waste activity, waste determinations, generation log, manifests, land disposal restriction notifications, exception reports, biennial reports, correspondence with local emergency responders, RCRA training records, and written contingency plan required (weekly container inspection is required & periodic equipment maintenance logs is recommended)
Waste “Designated Facility”	State-approved or RCRA permitted facility or legitimate recycler	RCRA-permitted facility or legitimate recycler	RCRA-permitted facility or legitimate recycler
Land Disposal Restrictions (268.7)	Possible state requirement	Applicable	Applicable

Attachment II: Common F-Listed Solvents

Waste Name	Hazardous Waste Code(s)	Waste Name	Hazardous Waste Code(s)
Acetone	F003	Methylene Chloride	F001, F002
Benzene	F005	Methyl ethyl ketone (MEK)	F005
iso-Butanol	F005	Methyl isobutyl ketone	F003
n-Butyl alcohol	F003	Nitrobenzene	F004
Carbon Disulfide	F005	2-Nitropropane	F005
Carbon Tetrachloride	F001	Orthodichlorobenzene	F002
Chlorobenzene	F002	Pyridine	F005
Chlorinated fluorocarbons (CFC)s	F001	Tetrachloroethylene	F001, F002
Cresols	F004	Toluene	F005
Cresylic acid	F004	1,1,1-Trichloroethane	F001, F002
Cyclohexanone	F003	1,1,2-Trichloeoethane	F002
2-Ethoxyethanol	F005	1,1,2-Trichloro-1,2,2-trifluoroethane	F002
Ethyl acetate	F003	Trichloroethylene	F001, F002
Ethyl benzene	F003	Trichloroflourormethane	F002
Ethyl ether	F003	Xylene	F003
Methanol	F003		

Attachment III: TCLP Contaminant List

Waste ID #	Contaminant	Conc (mg/L)
D004	Arsenic	5.0
D005	Barium	100.0
D006	Cadmium	1.0
D007	Chromium	5.0
D008	Lead	5.0
D009	Mercury	0.2
D010	Selenium	1.0
D011	Silver	5.0
D012	Endrin	0.02
D013	Lindane	0.4
D014	Methoxychlor	10.0
D015	Toxaphene	0.5
D016	2,4-D	10.0
D017	2,4,5-TP Silvex	1.0
D018	Benzene	0.5
D019	Carbon Tetrachloride	0.5
D020	Chlordane	0.03
D021	Chlorobenzene	100.0
D022	Chloroform	6.0
D023	o-Cresol	200.0
D024	m-Cresol	200.0
D025	p-Cresol	200.0
D026	Cresol	200.0
D027	1,4-Dichlorobenzene	7.5
D028	1,2-Dichloroethane	0.5
D029	1,1-Dichloroethylene	0.7
D030	2,4-Dinitrotoluene	0.13
D031	Heptachlor	0.008
D032	Hexachlorobenzene	0.13
D033	Hexachlorobutadiene	0.5
D034	Hexachloroethane	3.0
D035	Methyl ethyl ketone	200.0
D036	Nitrobenzene	2.0
D037	Pentachlorophenol	100.0
D038	Pyridine	5.0
D039	Tetrachloroethylene	0.7
D040	Trichlorethylene	0.5
D041	2,4,5-Trichlorophenol	400.0
D042	2,4,6-Trichlorophenol	2.0
D043	Vinyl Chloride	0.2

Attachment IV: Hazardous Waste Label for Accumulation Drum (Example)

Attachment V: Satellite Container Inspection Form

Waste Container ID	Clearly Labeled as "Hazardous Waste" with Waste Stream	Liquid Waste has Secondary Containment	Closed when not in use
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No

If any of the above fields are a "NO", please document how the container was brought back into compliance.

Comments: ______________________________

Inspector Signature ____________________ **Date:** ____________

Reviewer Signature ____________________ **Date:** ____________

ATTACHMENT VI: WASTE ACCUMULATION ROOM INSPECTION FORM

Containers closed when not in use	**Labels Easily Seen and Legible**	**Drums have Accumulation Start Date**	**Storage Amounts and Limits Obeyed**[1]	**Secondary Containment** for Liquid Waste	**Adequate Aisle Space**	**Available Emergency Equip. and Materials**	**Signature and Date of Inspection**	**Corrective Action for NO Answers**
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
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Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		

1: Accumulation limits are 90 days for LQG, and 180 days for SQG. SQG may have no more than 6000kg waste at any time.

ATTACHMENT VII: HAZARDOUS WASTE MANIFEST COVER SHEET

HAZARDOUS WASTE PICKUP DATE: _____

MANIFEST NUMBER(S)	3-SIGN. MANIFEST RETURN DATE
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

2-SIGNATURE FORM/PICK-UP CHECKLIST

- ☐ DRUMS REQUESTED FOR PICKUP/PRESENT MATCHES MANIFEST
- ☐ HAZARD CODES ARE CORRECT FOR EACH WASTE STREAM
- ☐ PACE REPRESENTATIVE AND TRANSPORTER SIGNATURE PRESENT

35 DAYS FROM PICKUP DATE: _____

If the three signature page has not been received within 35 days, contact the disposal facility to determine where the shipment is and request a copy of the three signature page.

If the three signature page has not been received within 45 days, you are required to file an exception report with the local regulating authority.

ALL 3-SIGNATURE MANIFEST(S) RECEIVED DATE _____

- ☐ FILE COVER SHEET, 3-SIG AND 2-SIG FORMS TOGETHER IN FOLDER, BINDER OR BY PAPER-CLIP. RETAIN FOR AT LEAST 3 YEARS

WASTE COORDINATOR: _____

DATE COMPLETED: _____

ATTACHMENT C-30

MEASUREMENT OF PERCENT MOISTURE IN SOILS AND SOLIDS
PACE INDIANAPOLIS

Document Information

Document Number: ENV-SOP-GBAY-0004 **Revision:** 00

Document Title: Measurement of Percent Moisture in Soils and Solids

Department(s): Client Services

Previous Document Number: S-GB-C-008-rev.05

Date Information

Effective Date: 20 Dec 2016

Next Review Date: 31 Oct 2020 **Last Review Date:** 31 Oct 2018

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-GBAY-0004 **Revision:** 00

Title: Measurement of Percent Moisture in Soils and Solids

All dates and times are in Central Time Zone.

Review: ENV-SOP-GBAY-0004 00 Measurement of Percent Moisture in Soils and Solids

Review

Name/Signature	Title	Date	Meaning/Reason
Kate Verbeten (007119)	Quality Manager	31 Oct 2018, 03:40:22 PM	Reviewed

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Green Bay, WI 54302

Phone: 920 469-2436
Fax: 920 469-8827

STANDARD OPERATING PROCEDURE

MEASUREMENT OF PERCENT MOISTURE IN SOILS AND SOLIDS

Reference Methods: ASTM D 2974-87

LOCAL SOP NUMBER:	S-GB-C-008-Rev.05
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-C-008-Rev.04
SOP TEMPLATE NUMBER:	SOT-ALL-L-004-rev.00

LOCAL APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/20/16

Kate Verbeten, Laboratory Quality Manager — Date: 12/19/16

Alee Her, Department Manager — Date: 12/19/16

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

This is **COPY#** 1 Distributed on **12/27/16** by JLS and is X **CONTROLLED**

S-GB-C-008-Rev.05

Table of Contents

1. Purpose/Identification of Method

1.1 This is Standard Operating Procedure (SOP) describes procedures used to measure percent moisture of soils and solid samples based on ASTM D 2974-87 Standard Test Methods.

2. Summary of Methods

2.1 A sample aliquot is weighed before and after heating to dryness at 103-105° C. The weight loss is calculated as % Moisture.

3. Scope and Application

3.1 **Personnel**: The policies and procedures contained in this SOP are applicable to all analysts experienced with the used of laboratory balances, desiccators, and ovens. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.2 **Parameters:** This SOP applies to percent moisture typically used to correct results of inorganic and organic parameter analysis to dry weight basis.

4. Applicable Matrices

4.1 This SOP is applicable to soil and solid samples (including sludges) containing at least 0.1% moisture.

5. Limits of Detection and Quantitation

5.1 Not applicable to this SOP.

6. Interferences

6.1 Non-representative materials, e.g., leaves and sticks should be removed from the sample prior to measurement.

6.2 Measurements are subject to negative bias for samples containing significant quantities of ammonium carbonate, volatile organics, or other volatile materials that could be lost during drying.

7. Sample Collection, Preservation and Handling

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil/ solid	Wide mouth glass or plastic 4-oz container	N/A	≤6°C	30 Days. Note: Analyze as soon as possible to minimize microbiological decomposition of organic solids.

8. Definitions

8.1 Refer to Glossary section of the Pace Quality Manual.

9. Equipment and Supplies

9.1 Instrumentation:

Equipment	Vendor	Model / Version	Laboratory Identification	Description / Comments
Analytical Balance	Mettler Toledo	PB602-S	40BAL9	Electronic with RS-232 output, capable of weighing 0.01g
Analytical Balance	A&D	EK200I	40BALN/ 40BALQ	Electronic with RS-232 output, capable of weighing 0.01g
Drying Oven	VWR	1370 FM	40OVN7	Capable of maintaining temperature at 103-105°C
Drying Oven	VWR	1370 GM	40OVNH	Capable of maintaining temperature at 103-105°C
Computer for Electronic Prep Log				Automated sample weight upload into LIMS

9.2 Supplies

Supplies	Vendor	Model / Version	Description / Comments
Desiccators	Fisher	Fisher p/n 08-644	Labconco Model
Indicating Desiccant	Fisher	Fisher p/n 07-578-4B	
Non-indicating Desiccant	Fisher	Fisher p/n 07-577-3B	
Disposable Aluminum Weighing Dishes	Fisher	Fisher p/n 08-732	
Spoonula Lab Spoons	Fisher	Fisher p/n 14-375-10	
Trays, plastic or metal	NA	NA	

10. Reagents and Standards

10.1 Not applicable to this SOP.

11. Calibration and Standardization

11.1 Analytical Balance Calibration

11.1.1 Annual Calibration - The balance must be calibrated at least annually by an outside agency and checked daily before each use using Class 1 or 2 weights. Refer to Pace SOP, S-GB-Q-030 *Support Equipment* (most current revision or replacement).

11.2 Daily Calibration Check

11.2.1 Clean the balance and surrounding area prior to starting the daily calibration check.

11.2.2 Check the sight level on the balance. If it needs adjusting, level the balance.

11.2.3 The weight set ID indicated in the logbook is used as the primary set. If an alternate weight set ID is used, that ID must be recorded in the comment section of the balance calibration logbook for that day.

11.2.4 Tare the balance before weighing the NIST certified weights.

11.2.5 Use forceps or other means to lift each weight (Do not touch the weights with fingertips as the residue may artificially adjust the true value of the weights). Record the date of the calibration check, the true value of the weight, and the actual measured weight in the logbook. Repeat this procedure for the other certified weights. If calibration weights differ from the certified weights by more than specified in the balance calibration logbook, corrective action must be taken (see 11.3).

11.3 Corrective Action

11.3.1 Clean the balance and balance pan. Check the sight level on the balance and adjust if necessary. Re-tare and re-weigh all the certified weights.

11.3.2 The internal calibration function (if available) of the balance may be used as a means of corrective action.

11.3.3 Utilize the internal calibration function and diagnostics. Refer to instrument manual.

11.3.4 Contact the QA office for assistance if the balance does not meet the calibration tolerances.

11.3.5 If the above action does not correct the problem, the balance should be taken out of service and appropriately labeled to avoid improper usage. A service technician should be contacted.

11.3.6 Record any corrective action. Initial and date all entries in the logbook.

12. Procedure

12.1 Locate the samples to be analyzed, place on a cart and allow samples to warm to ambient temperature prior to processing.

12.2 Review location of samples. Soil samples that are collected in regulated domestic areas or that are of foreign origin must be handled in accordance with the Pace SOP: S-GB-S-001, *Regulated Soil Handling* (most current revision or replacement).

12.3 Determine the number of aluminum weighing pans required for the number of samples to be analyzed plus one for a duplicate.

12.4 The samples scheduled for analysis are batched in the PMST QUEUE in groups of 20. The QC batch will also include a duplicate for one of the project soil samples.

12.5 After batching samples in EPIC Pro print the work list.

12.6 Open the Electronic Prep Log. To start a new worksheet, select the template from the list of active templates. You may also search for them by clicking the triangle expanded button and entering in criteria for your search. Then press enter (or click the search button) to perform the search. Once you have the needed template, double click on it.

12.7 Now that the template is loaded you need to enter the Batch for your test. You can either drag over the Batch Samples in the order you need them, or you can drag them over and then reorder them using the drag and drop method. Once you have them in the order you need them click the arrows to the right of "Search by Batch" to minimize the space the "Search Samples" section takes up.

12.8 Save your data by pressing the disc icon next to the search template button. If this is your first time saving it will request you to enter a prep group description. This is used by your group to find the Electronic Log you are making. Enter the name of the queue, batch number and lab group (example for Sample Receiving enter "SR").

12.9 Verify balance calibration - refer to section 11 for balance check procedures and corrective actions. Refer to the balance logbook for the acceptance criteria for the designated balance.

12.10 Not that you have your run set-up, you can start entering results.

12.11 To use the balance, verify the balance matches the instrument ID and click the balance icon in between the search button and the Autopost button.

12.12 For each sample, label and record the tare weight (to the nearest 0.01 g) for an aluminum weighing dish by placing your curser in the field you wish to put the weight in and press the print button on the balance.

12.13 Using a clean spoonula lab spoon, stir the material in the sample container. Transfer at least 10g of the remaining sample to the tared weighing dish

12.14 Weigh the sample and dish, recording to the nearest 0.01g.

12.15 Place the samples to **dry in the** oven overnight at 103-105°C. Check that the oven temperature i**s recorded on th**e Electronic Prep Log benchsheet and is within required specifications before placing samples into oven as per Pace SOP S-GB-Q-030 *Support Equipment* (most current revision or replacement).

12.16 Overnight is a period of time ≥ 8 hours.

12.17 Record the oven temperature prior to removal of the samples and verify it is still within the required specifications of 103-105 °C. Remove the sample from the oven place in a desiccator to cool. The desiccator should contain mostly non-indicating desiccant with enough indicating desiccant to demonstrate that the desiccant is still active.

12.18 After the sample has cooled, weigh the dried residue to the nearest 0.01g. If the sample has been oven dried for at least 8 hours, proceed to section 12.20. If dried less than 8 hours, proceed to the next section.

12.19 Return the samples to the oven for one additional hour. At the end of the hour**, remove the** samples once again; allow them to cool to room temperature and reweigh. If the weight is within 0.01g or 0.1% of the previous weight, record the weight and proceed to 12.20. If the weight has changed by more than 0.01g or 0.1%, repeat step 12.15 until a constant weight (<0.01g or 0.1% change) is achieved.

12.20 As weights are entered Percent Difference and Weight Differences will be calculated when using multiple weighings.

12.21 You can manually select a weight to use by entering an “M” in the Use test box to override the automatic weight chosen. If you want to manually de-select a weight, enter an “m”. The weights are chosen using weight differences being less than 5mg.

12.22 Once all of your weights have been taken and additional information has been entered your results will be calculated and you will be ready to autopost the data into EpicPro.

12.23 Save your data by pressing the disk icon next to the search template button.

12.24 If required you may print or create a PDF of your data by selecting Menu -> Print Landscape.

12.25 Verify all of the samples you wish to autopost have a Y for select, then press the AutoPost Button.

12.26 Select a few samples randomly and verify that the % Moisture final result is being calculated correctly following Section 14 for the calculation.

12.27 Once data is autoposted, review for precision. See Section 14 for the RPD Calculation.

13. Quality Control

13.1 **Duplicate Sample –** Measure one duplicate sample with each batch of 20 samples. The Relative Percent Difference (RPD) for duplicate results must be ≤ 10%. If this is not met the entire batch must be re-analyzed.

13.2 Documentation of Equipment Operation and Calibration – The balance calibration check and oven temperature should be recorded on the lab datasheet for each sample batch. In addition, the oven temperature should be read each day it contains active samples and the temperature recorded in the oven log. If balance checks and oven temperatures are not within acceptable limits, all effected samples must be re-analyzed.

14. Data Analysis and Calculations

14.1 % Moisture is calculated and in the Electronic Prep Log worksheet using the following equation.

$$\% \text{ Moisture} = (W_w - W_d)*100\%/W_w$$

Where:

W_w = Wet weight of the sample (Dish + sample weight before drying – dish tare weight)
W_d = Dry weight of the sample (Dish + sample weight after drying – dish tare weight)

14.2 Relative Percent Difference (RPD) is calculated as follows:

$$\%RPD = (S1-S2)*100\%/((S1+S2)/2)$$

Where: S1 = %Moisture for Sample
S2 = %Moisture for Sample Duplicate

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See Section(s) 11.3 and 13.

16. Corrective Actions for Out-of-Control or Unacceptable Data

16.1 See Section 13.

17. Contingencies for Handling Out-of-Control Data

17.1 See Section 13.

18. Method Performance

18.1 All applicable personnel must read and understand this SOP with documentation of SOP reviewed maintained in their training files. Additionally, staff must read and understand the Pace SOP: S-GB-S-001 *Regulated Soil Handling* (most current revision) in addition to receiving Regulated Soil Training upon hire and annually thereafter.

18.2 **Demonstration of Capability (DOC) –** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) described in S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement). All results must be ± 10% of the mean to qualify the analyst for reporting sample results. Results of DOC studies for each analyst shall be retained in the lab quality assurance office. Each analyst must successfully repeat this study annually to maintain qualification.

19. Method Modifications

19.1 Method modifications for ASTM D2974-87 are as follows:

19.1.1 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.

19.1.2 All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.

19.1.3 Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace Laboratories.

19.1.4 ASTM D2974-87 states to use 50 g of the test specimen; Pace Analytical Services, Inc – Green Bay uses a 10 g aliquot.

19.1.5 ASTM D2974-87 states to dry the sample for 16 hours, Pace Analytical Services, Inc. – Green Bay defines the drying time as overnight, which is a drying time of ≥8 hours.

20. Instrument/Equipment Maintenance

20.1 Maintain the analytical balance, ovens, and furnaces according to the most current revision of SOP S-GB-Q-030, *Support Equipment.*

21. Troubleshooting

21.1 Refer to Section 11.3. If additional assistance is required refer to the operations manual for the oven or balance as needed**.**

22. Safety

22.1 **Standards and Reagents**: Not applicable to this SOP.

22.2 **Samples -** Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.

22.2.1 Regulated soil samples are to be handled in accordance with Pace SOP: S-GB-S-001, *Regulated Soil Handling* (most current revision or replacement).

22.3 **DO NOT WEAR LATEX OR NITRILE GLOVES WHILE HANDLING HOT TRAYS.**

23. Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

24. Pollution Prevention

24.1 The Pace Chemical Hygiene Plan/Safety Manual contains additional information on pollution prevention.

25. References

25.1 Pace Quality Assurance manual (most current revision or replacement.

25.2 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (most current revision or replacement).

25.3 ASTM D 2974-87 Test Method A "Standard Test Methods for moisture, Ash and Organic Matter of Peat and Other Organic Soils", American Society of Testing and Materials, Reapproved 1995.

25.4 EPA Contract Laboratory Program SOW for Inorganic Analysis Doc. ILM 1.03 March 1990.

25.5 ASTM D 2216-98 "Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass", American Society of Testing and Materials, 1998.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1 Not applicable to this SOP**.**

27. Revisions

Document Number	Reason for Change	Date
S-GB-C-008-Rev.04	Throughout Document: Updated SOP to new format following SOP: S-GB-Q-017 *Preparation of SOPs* (most current revision or replacement). Updated SOP references throughout document. Section 7: Added 30 day hold time. Section 9.1: Updated to current oven and balance equipment listings. Section(s) 12.2, 18.1, and 22.2.1: Added pertinent information on the requirements for Regulated Soil Handling Procedures. .	19Dec2014
S-GB-C-008-Rev.05	Cover Page: Updated QM name, name change.	12Dec2016

ATTACHMENT C-31

DETERMINATION OF TOTAL ORGANIC CARBON USING THE WALKLEY-BLACK PROCEDURE
PACE INDIANAPOLIS

Document Information

Document Number: ENV-SOP-GBAY-0032 **Revision:** 00

Document Title: Determination of Total Organic Carbon Using the Walkley-Black Procedure

Department(s): Wet Chemistry

Previous Document Number: S-GB-I-037-rev.06

Date Information

Effective Date: 15 Jan 2018

Next Review Date: 15 Jan 2020 **Last Review Date:**

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

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STANDARD OPERATING PROCEDURE

The Determination of Total Organic Carbon Using the Walkley-Black Procedure

Reference Methods: Methods of Soil Analysis, Walkley-Black

SOP NUMBER:	S-GB-I-037-Rev.06
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-037-Rev.05

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 01/15/18

Kate Verbeten, Laboratory Quality Manager — Date: 1/12/18

Chad Rusch, Department Manager — Date: 1/12/18

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL

Signature	Title	Date

This is **COPY#** ____ Distributed on _____ by _____ and is ____ **CONTROLLED**

S-GB-I-037-REV.06

Table of Contents

1 Purpose

1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the method used to determine the concentration of Total Organic Carbon (TOC) in soil samples compliant with the Walkley-Black procedure.

2 Summary of Method

2.1 An aliquot of a dried and homogenized solid sample is put into a COD vial and caped. The sealed tubes are heated in a hot block at 150°C. After two hours, the tubes are removed, cooled and measured spectrophotometrically at 620 nm.

3 Scope and Application

3.1 **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2 **Parameters**: This SOP applies to Total Organic Carbon (TOC).

4 Applicable Matrices

4.1 This SOP is applicable to sediments, soils and sludges.

5 Limits of Detection and Quantitation

5.1 Current LOD and LOQ can be found in the Laboratory Information Management System (LIMS) - EpicPro.

5.2 Level of Detection (LOD): The LOD is determined by the 40CFR Part 136B MDL study. Once the 40CFR Part 136B MDL is determined it may be elevated, if deemed unrealistic as demonstrated using method blank evaluations.

5.3 Level of Quantitation (LOQ): The LOQ is calculated as 3 times the LOD. A realistic LOQ is typically near the lowest non-zero calibration point and higher than typical blank measurements. If 3 times the LOD is less than the low standard, the LOQ is set as the low standard.

6 Interferences

6.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

6.2 For the Walkley Black method, organic carbon that is not easily oxidizable results in a low bias. As described in the 1933 Walkley Black paper, this incomplete oxidation results in a recovery of organic carbon in the range of 60 to 86 percent. A correction factor was determined from their studies to be a multiplier of 1.32. This is solely based on the 20 soils in their study. Additional papers have demonstrated recoveries to be in the range of 59 to 94%. As a result, by applying the 1.32 multiplier the TOC result may be biased low or high. This procedure follows the modification to the Walkley Black method and heats the sample in the presence of the dichromate and H2SO4 at 150°C for two hours. This modification results in the oxidation of difficult to oxidize organic carbon and provides a more accurate result.

6.3 Chloride results in a positive interference. Chlorides are oxidized to free chlorine by chromic acid. Where consumption of dichromate is used to determine organic carbon, the presence of Cl can result in erroneously high organic carbon. Mercuric sulfate in the digestion tubes complexes the chlorides and minimizes the interference from chlorides.

6.4 The presence of ferrous iron (Fe^{2+}) consumes the dichromate and results in a positive interference. Drying of the sample during preparation for analysis oxidizes Fe^{2+} to Fe^{3+}, minimizing the amount of Fe^{2+}.

7 Sample Collection, Preservation, Shipment and Storage

7.1 The lab provides appropriate bottle ware, including preservative, for requested testing. Where applicable, the bottle ware is demonstrated to be free of target analytes. When bottle ware not originating from the lab is used, the data may be qualified with either one or both of the following data qualifiers:

7.1.1 Sample field preservation does not meet EPA or method recommendations for this analysis.

7.1.2 Sample container did not meet EPA or method requirements.

7.2 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Matrix	Method	Container(s)	Preservation	Hold	Shipment Conditions	Lab Storage Conditions
Solid	Walkley Black	Clean 4oz glass containers	Thermal to ≤6°C	28 days	On ice ≤6° Celsius	≤6°C

8 Definitions

8.1 Total Carbon (TC): TC includes both the Inorganic and Organic constituents of a sample.

8.2 Total Organic Carbon (TOC): All carbon atoms covalently bonded in organic molecules. TOC is the carbon stored in the soil organic matter.

8.3 Total Inorganic Carbon (TIC): TIC is the sum of the inorganic carbon species in a sample. These can include bicarbonate, carbonate, and other carbonate minerals.

8.4 Soil Organic Matter (SOM): Organic matter is the organic component of soil. It consists of varying proportions of plant and other organisms, both living and decomposing as well as stable organic matter known as humus. SOM is estimated to be 58% soil organic carbon (OM = TOC x 1.72). This assumption can vary with the type of organic matter, soil type, and soil depth.

8.5 Fractional Organic Carbon ($f_{oc,}$ FOC): The FOC of soil is the fraction of the organic matter that is carbon. It can be simply defined as the TOC content and can be expressed as a decimal fraction (i.e. 2.5% TOC = 0.025 FOC).

8.6 Additional definitions can be found in Definitions Section of the Pace Analytical Services Quality Manual.

9 Equipment and Supplies

9.1 Equipment

Equipment	Manufacturer	Model(s) /Catalog Number
Top loading Balance	Mettler Toledo	AE160
Spectrophotometer	Hach	DR2000
COD reactor block	Hach	45600
Adjustable pipettor (0.5-5.0mL)	Eppendorf / Fisher	3123000071 / 13-690-033
Drying Oven (74-76°C)	Curtin Matheson	213-454
Mortar and Pestle	Coors	60316, 60317
Vented Hood	Hamilton or Equivalent	

Or equivalent

9.2 Supplies

Supplies	Manufacturer	Catalog #
COD digestion vials	Columbia Analytical Instruments / Fisher	COD15000
Thermal gloves	Fisher	19-013-541
5000 µL Pipette tips	Unifit National Scientific / MG Scientific	NUN05ME-BP
Mortar	CoorsTek / Fisher	60316 / 12-961A
Pestle	CoorsTek / Fisher	60317 12-961-5A
10 mL volumetric flasks	Kimble / Fisher	92812G10 / 10-310-235
50 mL volumetric flasks	Pyrex / Fisher	564050 / S14290
100 mL volumetric flasks	Pyrex / Fisher	5660100 / S14291
1000 mL volumetric flask	Pyrex / Fisher	56401L / S14295
Wire racks	Fisher	Cat# 14802

Or Equivalent

10 Reagents and Standards

10.1 Stock Standards and Reagents

Standard/Reagent	Alias	Purchased From	Catalog Number	Concentration / Purity	Expiration	Storage
Nanopure® Water	Water	In House	NA	≥18 Mega ohm	6 months	Room Temp
Primary Source	5310C-STK	SCP Science	250-250-051	1,000 mg/L	Manufacturer's	Room Temp
Secondary Source	TOC-SPK1000	Ultra Scientific	IQC-106-5	1,000 mg/L	Manufacturer's	Room Temp

10.2 Working Standards and Reagents

Standard / Reagent	Alias	Stock or Intermediate	Amount Used	Final Volume (W/Diluent)	Diluent	Final Concentration	Expiration	Storage
Calibration Standard 0	CAL0, MB, ICB, CCB	Water	2.0 mL	50 mL	Water	0 mg/L	made daily	NA
Calibration Standard 3	CAL3	5310C-STK	2.5 mL	50 mL	Water	50 mg/L	made daily	NA
Calibration Standard 1	CAL1, CRDL	CAL3	2.0 mL	10 mL	Water	10 mg/L	3 Months	≤6°C
Calibration Standard 2	CAL2	CAL3	4.0 mL	10 mL	Water	20 mg/L	made daily	NA
Calibration Standard 4	CAL4	5310C-STK	2.0 mL	10 mL	Water	200 mg/L	made daily	NA
Calibration Standard 5	CAL5, CCV	5310C-STK	20 mL	50 mL	Water	400 mg/L	3 Months	≤6°C
Calibration Standard 6	CAL6	5310C-STK	8.0 mL	10 mL	Water	800 mg/L	made daily	NA
Initial Calibration Verification	ICV	TOC-SPK1000	4.0 mL	10 mL	Water	400 mg/L	made daily	NA
Secondary Spike	5310C-SPK	TOC-SPK1000	32 mL	40 mL	Water	800 mg/L	3 Months	≤6°C
Laboratory Control Spike/Laboratory Control Spike Duplicate	LCS/ LCSD	5310C-SPK	1 mL	2 mL	Water	400 mg/L	made daily	NA
Matrix Spike/Matrix Spike Duplicate	MS/ MSD	5310C-SPK	1 mL	2 mL	Water	400 mg/L	made daily	NA

11 Calibration and Standardization

11.1 A new calibration curve should be prepared yearly at minimum or whenever the continuing calibration standard does not pass control criteria.

11.2 Prepare a calibration curve using calibration standards and a blank. Add 2.0 mL of each curve standard to a digestion tube and digest per Section 12.

11.3 Turn on Spectrophotometer and set to 620 nm and allow it to warm up for at least 20 minutes.

11.4 Wipe the outside of each vial with tissue paper to remove any fingerprints. Place Level 0 calibration standard into the spectrophotometer and zero the instrument.

11.5 Place the standard cells into the spectrophotometer and record standard absorbance at 620 nm into the electronic prep log. Calculate and lock curve to set as the new curve.

11.6 The data is saved within the program and can be referenced when needed. Note: Record the HBN# associated to the curve as it needs to be entered for each new preplog worksheet created.

11.7 In order for the calibration to be acceptable a linear regression analysis of the absorbance values against the corresponding concentrations must yield a correlation coefficient of >0.995. The y-intercept must be less than the Pace Reporting Limit and the analysis of an ICV, ICB, and CRDL must be within acceptance criteria prior to analyzing samples.

11.8 If the calibration does not pass acceptance criteria, correct the problem and recalibrate.

12 Procedure

12.1 Soil samples that are collected in regulated domestic areas or that are of foreign origin must be handled in accordance with the Pace SOP: S-GB-S-001, *Regulated Soil Handling* (most current revision or replacement).

12.2 Sample Preparation.

12.2.1 Oven-dry overnight and grind to a size that the sample will pass through a 0.5-mm sieve.

12.2.2 Mix the sample thoroughly before selecting a portion for analysis.

12.2.3 Discard any foreign objects such as sticks, leaves, and rocks.

NOTE: Document all sample sizes, standards and reagents used in the digestion in the electronic Preplog.

12.3 Analytical

12.3.1 Samples are batched in Horizon/Epic Pro (the LIMS) with the appropriate batch QC.

12.3.2 The electronic Prep Log is used to document the Prep and Analytical steps and to post the data to the LIMS. Use the TOC Walkley Black Prep and WB TOC Walkley Black Analytical templates.

12.3.3 Label COD digestion vials

12.3.4 Unseal the vials and carefully weigh out 0.05 g using a calibrated balance. Place the 0.05 g of soil into a vial. Pipette 1.95 mL of water into a vial such that it forms a layer on top of the reagents contained in the vial.

12.3.5 Prepare CCV, CCB, MB, LCS, MS, and MSD.

12.3.6 Carefully seal the vial. During digestion, the reagents and sample are raised to a point just below boiling. Improperly sealed vials may leak or break.

12.3.7 Thoroughly mix the contents of the sealed vial by shaking. CAUTION: The vial will get very hot during mixing. It is recommended that vials be mixed

either in racks or with the use of insulated gloves. Eye protection MUST be worn.

12.3.8 Place the digestion vials in the reactor block at 150°C ± 5°C for 2 hours.

12.3.9 Cool about ½ hours. Then invert vials to mix.

12.3.10 Cool to room temperature. Allow any suspended precipitate to settle.

12.3.11 Turn on Spectrophotometer and set to 620 nm. Let warm up at least 20 minutes.

12.4 Wipe the outside of each vial with tissue paper to remove any fingerprints. Place ICB/CCB into the spectrophotometer and zero the instrument. Then, place the sample vials into the spectrophotometer and record sample and QC absorbance at 620 nm into the prep log.

12.4.1 The prep log will calculate the sample results in mg/L TOC, upload from the prep log, and report the final results in LIMS.

13 Quality Control:

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 **Initial Calibration Verification (ICV):**

13.2.1 The ICV must be analyzed immediately after calibration, prior to samples.

13.2.2 The recovery must be within ±10% of the true value.

13.2.3 When measurements are outside the control limits, reanalyze once. If the measurement is still outside of the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified.

13.2.4 The source of the purchased 1,000mg/L TOC standard used to make the ICV must be different from that of the calibration curve standards and CCV.

13.3 **Continuing Calibration Verification (CCV):**

13.3.1 The CCV is analyzed after every 10 samples.

13.3.2 Concentration must be within ±10% of the true value.

13.3.3 When measurements are outside the control limits, reanalyze once. If the measurement is still outside of the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified. If the reset CCV fails recalibrate and reanalyze all samples back to the last acceptable CCV.

13.4 **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB):**

13.4.1 The ICB must be analyzed after the ICV and before samples. The CCB must be analyzed after each CCV and before samples.

13.4.2 The absolute value must be <LOQ.

13.4.3 When the absolute value of the measurements, are greater than the LOQ, the blank may be immediately reanalyzed. If the reanalyzed blank passes, continue with analysis. If the reanalyzed blank fails, recalibrate and reanalyze all samples back to the last acceptable instrument blank.

13.4.4 No samples may be reported if bracketed by an ICB or CCB that is outside of the control limits, with the following exceptions:

13.4.4.1 If the sample concentration is greater than ten times the absolute measurement in the ICB or CCB, the samples do not need to be reanalyzed and can be reported without qualification.

13.4.4.2 For positive blank failures, with a sample that is a non-detect, the sample does not need to be reanalyzed and can be reported without qualification.

13.5 **Reporting Limit Verification Standard (CRDL) –** A standard prepared at the concentration of the Pace Reporting Limit.

13.5.1 It is analyzed after the calibration and also before each new batch,

13.5.2 Concentration must be within ±40% of the true value.

13.5.3 If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.6 **Method Blank (MB)**

13.6.1 The MB is laboratory grade water analyzed exactly like a sample. The MB is used to verify that interferences caused by contaminants in the solvents, reagents, glassware, etc. are known and minimized.

13.6.2 A MB must be analyzed with each batch of samples or every 20 samples, whichever is more frequent.

13.6.3 Acceptance Criteria: The MB is evaluated for both positive and negative bias and must have an absolute value less than the LOQ. For samples reporting down to the LOD, the MB measurements are evaluated to the LOD. In these cases qualify applicable samples for MB measurements from >LOD to <LOQ.

13.6.4 If the MB is greater than the LOQ, perform the following:

13.6.4.1 Check for errors in calculations. If an error or problem is found and can be corrected by amending the calculations and the result falls within the limits, accept the data and report without a qualifier flag.

13.6.4.2 If there is sufficient sample available and hold time remaining, re-prepare the MB and all associated. If the MB is less than the LOQ in this analysis, accept the second set of data. If the MB is still outside the RL after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant MB.

13.6.4.3 If sufficient sample volume is not available, report the sample data with a qualifier flag on each of the samples associated with the non-compliant MB. Contact the project manager regarding the occurrence.

13.6.5 MB data qualifying

13.6.5.1 In the absence of project specific requirements, samples with concentrations greater than 10 times the absolute blank measurement may be reported unqualified.

13.6.5.2 In the absence of project specific requirements, samples that are non-detect may be reported unqualified if the blank measurement demonstrates a positive bias.

13.6.5.3 In the absence of project specific requirements, samples that are non-detect must be qualified if the blank measurement demonstrates a negative bias between and including the LOD and LOQ. Non-detect samples may not be reported with a blank negative bias greater than the LOQ.

13.6.5.4 For samples that need qualification resulting from MB measurements that are positive, apply a B data qualifier to the analyte. B = “Analyte was detected in the associated method blank.”

13.6.5.5 For samples that need qualification resulting from MB measurements that are negative, apply a hand entered qualifier with the measurement and the units. "Analyte was measured in the associated method blank at a concentration of -#.# units."

13.7 **Laboratory Control Sample (LCS):**

13.7.1 The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples. A Laboratory Control Spike Duplicate (LCSD) must be analyzed if there is insufficient sample volume to perform a matrix spike/matrix spike duplicate or if the client requests one.

13.7.2 The recovered concentration must be within default limits of ± 20%.

13.7.3 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a data qualifier.

13.7.4 If no errors are found reanalyze once. If the measurement is still outside of the control limits and sufficient sample is available, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still outside the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.7.5 If sufficient sample volume is not available, report the sample data with appropriate data qualifier on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.7.6 The precision between the LCS and LCSD must be ≤ 20% RPD.

13.7.6.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.6.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.7.7 The source of the 1,000mg/L TOC standard used to make the CCV is not the same for the LCS.

13.8 **Matrix Spike (MS) and Matrix Spike Duplicate (MSD):**

13.8.1 MS/MSD pairs are analyzed in each batch at a 10% frequency or one pair per 10 environmental samples. Both the MS and MSD are evaluated for accuracy and precision.

13.8.2 The recovered concentration must be within default limits of ±20%.

13.8.2.1 If four times the concentration of the spike is less than the analyte concentration of the parent, accuracy need not be calculated.

13.8.2.2 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a data qualifier.

13.8.2.3 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.8.3 The precision between the MS and MSD must be ≤ 20% RPD.

13.8.3.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.3.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.8.4 The sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. Avoid the use of field, filter, trip or equipment blanks for MS/MSD parent samples.

13.8.5 The source of the 1,000mg/L TOC standard used to make the CCV is not the same for the MS and MSD.

13.9 Duplicate sample (D) –A duplicate aliquot of a sample to be analyzed along with the original sample. Duplicate analysis indicates the precision associated with the sample collection, preservation and storage, as well as, laboratory procedures. An MSD is normally performed instead of a Duplicate Sample.

13.9.1 Duplicate sample analysis will be performed if requested by the client.

13.9.2 Acceptance limits: The RPD must be within 0-20% between the original sample and the duplicate.

13.9.3 If the RPD is exceeded, then:

13.9.3.1 Check for errors in calculations and sample preparation. If an error or problem is found and can be corrected by amending the calculations and the result falls within the limits, accept the data and report without a qualifier flag.

13.9.3.2 If no errors are found in calculations report the parent sample with the appropriate data qualifier.

13.10 **Hold:** When preparation of a sample exceeds 28 days past the time of collection, notify the project manager before proceeding. If a sample is run past 28 days after collection, flag the result with appropriate data qualifier.

13.11 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with appropriate data qualifier.

14 Data Analysis and Calculations

14.1 $$\frac{\text{(mg/L TOC from the curve) X (Final Volume (mL))}}{\text{(Weight in (g))}} = \text{TOC (mg/Kg)}$$

14.2 TOC (%) = (concentration in mg/kg / 10,000)

14.3 Accuracy:

A laboratory control spike / laboratory control spike duplicate is analyzed for each analytical batch of 20 or fewer samples.

$$\text{LCS, \% Recovery} = \frac{\text{TOC, mg/kg}}{\text{True Value mg/Kg}} \times 100$$

14.4 Precision:

The precision is calculated based on the recovery of the sample / sample duplicate result. A sample duplicate is performed at a frequency of 10% or one per batch whichever is more frequent and must meet laboratory specific limits for precision.

Relative percent difference (RPD) calculation:

$$\% \text{ RPD} = \frac{|S-SD|}{(S+SD)/2} \times 100$$

S = Sample Value
SD = Sample Duplicate Value

15 Data Assessment and Acceptance Criteria for Quality Control Measures

Table A. QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**Walkley Black**	**Acceptance Criteria**
Method Blank (MB)	One per batch of samples, up to 20 environmental samples.	<LOQ. Detections ≥ LOD but < LOQ must be evaluated for data qualification.
Laboratory Control Spike and Duplicate (LCS/LCSD)	One per batch of samples, up to 20 environmental samples. A LCSD is required if MS/MSD is not performed.	Recovery must be within ± 20% of the true value. If LCSD is run RPD <20%
Matrix Spike / Matrix Spike Duplicate (MS/MSD)	One pair per batch of samples, up to 10 environmental samples.	Recovery must be within ± 20% of the true value. RPD <20%
Sample Duplicate (DUP)	Upon client request	Project Specific or RPD <20%
Initial Calibration	Minimum of 5 standards plus blank. Performed once a year at a minimum.	Correlation coefficient ≥0.995
CRDL	After the calibration and following the initial CCV/CCB in each batch.	Recovery must be within ± 40% of the true value.
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. CCV – analyzed after every 10 samples.	Recovery must be within ± 10% of the true value.
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. CCB – analyzed after every CCV pair.	Project Specific or <LOQ

16 Corrective Actions for Out-of-Control Data

Table B. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Walkley Black If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Sample Duplicate	• 4
Initial Calibration	• 5
CRDL standard	• 6
Initial / Continuing Calibration Verification	• 7
Initial / Continuing Calibration Blank	• 8

1. If not <LOQ, verify by second analysis. If second analysis confirms contamination for target analyte at or greater than the LOQ, re-digest sample batch and batch QC provided sufficient sample volume remains. If insufficient sample volume remains, consult with project manager and client on how to proceed. For MB detections greater than or equal to the LOD, but less than the LOQ; qualify applicable sample results. For negative measurements more negative than the LOD, applicable data is given the following data qualifier: "Analyte was measured in the associated method blank at a concentration of -#.# units."
   * For positive MB failures, samples that are non-detection need not be qualified. In addition, samples that are greater than 10 times the MB detection need not be qualified.
   * For negative MB failures samples that are greater than 10 times the MB detection need not be qualified.

2. If the parent, MS, or MSD is greater than the reportable linear dynamic range, dilute and reanalyze the parent, MS, and MSD. If the concentration of the spike is less than 25% of the concentration of the parent the MS and MSD recoveries are not evaluated. Any failures resulting from this are qualified appropriately. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately qualify the parent sample if either the MS and/or MSD fail accuracy. If the MS and MSD fail precision control limits flag the parent with the appropriate precision data qualifier.

3. Verify failure by second analysis. If second analysis confirms LCS (LCSD) failure, re-digest sample batch and batch QC provided sufficient sample volume remains. If insufficient sample volume remains, consult with project manager and client on how to proceed.

4. If no errors are found in calculations report the parent sample with the appropriate data qualifier.

5. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

6. It is analyzed after the calibration and following the initial CCV/CCB in each batch, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

7. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed.

8. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed.

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 See Section 16.

18 Method Performance

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

18.2 The analyst must read and understand this procedure with written documentation maintained in his/her training file. Additionally, staff must read and understand the Pace SOP: S-GB-S-001 *Regulated Soil Handling* (most current revision) in addition to receiving Regulated Soil Training upon hire and annually thereafter.

18.3 An initial demonstration of capability (IDOC) must be performed per the most recent version of S-ALL-Q-020, *Orientation and Training Procedure*s (most current revision or replacement). A continuing demonstration of capability (CDOC) must be performed annually. A record of the DOCs will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

18.4 At a minimum, the 40CFR part 136 appendix b study must be performed every year, per the most recent version of S-GB-Q-020, *Determination of the LOD and LOQ* (most current revision or replacement). Additional studies may be performed to achieve a realistic LOD and LOQ. This is to be done for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

18.5 Periodic performance evaluation (PE) samples are analyzed per the most recent version of S-GB-Q-021 *PE/PT Program* (most current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office. These are performed twice a year per matrix.

18.6 A linear dynamic range study must be conducted at least once. The study is conducted for each analyte by analyzing increasing concentrations (at least 3 levels) until the results generated exceed ±10% difference from the true value. The highest concentration within the 10% criteria is the maximum of the linear range for that analyte. Once the linear dynamic range study determination is performed, keep the data, and then quarterly at a minimum verify with a single high point. Pace Analytical Services, LLC – Green Bay Laboratory will not use any data over the highest calibration standard used. All samples will be diluted and reanalyzed that are over the calibration range.

19 Method Modifications

19.1 The Walkley Black (WB) method uses an acidic dichromate solution to react with a soil sample to oxidize the organic matter. It is then titrated with ferrous sulfate to a color change endpoint from a green to reddish brown color. The endpoint can be difficult to determine in the stirred up sample during titration. This procedure utilizes COD vials with the same acidic dichromate solution to oxidize the organic matter. This reduces the hazardous waste volume produced by the test and allows for a quicker, more accurate spectrophotometric analysis of the sample.

19.2 If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the "M5" data qualifier.

19.3 The 0.68 correction factor also does not need to be used, as the COD vials are heated with the sample in them at 150±5°C for 2 hours. This completes the oxidation process and eliminates the need for the correction factor.

19.4 The Walkley Black method describes samples as "Finely divided soil, passing a 100-mesh sieve, taken in amounts between 10 and 25 mgm of carbon". This procedure does not pass the sample through a 100 mesh sieve, but does pulverize the samples by mortar and pestle after drying and prior to subsampling for analysis.

20 Instrument/Equipment Maintenance

20.1 See the Hach DR200 instrument Maintenance and Operator's Manual.

21 Troubleshooting

21.1 See the Hach DR200 instrument Maintenance and Operator's Manual.

22 Safety

22.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

22.2 A reference file of Safety Data Sheets (SDS) is made available to all personnel involved in the chemical analysis, and is located at the following link: **https://msdsmanagement.msdsonline.com/c0ce0b0a-17d3-4f3c-afc6-25352729b299/ebinder/?nas=True**. A formal safety plan has been prepared and is distributed to all personnel with documented training.

22.3 Regulated soil samples are to be handled in accordance with Pace SOP: S-GB-S-001, *Regulated Soil Handling* (most current revision or replacement).

23 Waste Management

23.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

23.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management*.

24 Pollution Prevention

24.1 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25 References

25.1 Pace Quality Assurance Manual (most current revision or replacement).

25.2 The NEALC Institute (TNI): Volume 1, Module 2, “Quality Systems” (most current revision or replacement).

25.3 Walkley, A.; Black, I.A. 1934. An examination of the Degtjareff method for determining soil organic matter, and a proposed modification of the chromic acid titration method. Soil Science 37:29-38.

25.4 Recommended Soil Testing Procedures for the Northeastern United States, 2nd Edition, Northeastern Regional Publication No. 493, Revised December 15, 1995.

25.5 Methods of Soil Analysis, 1982 Second Edition, Method 29-3.5.2.1 Walkley-Black Procedure.

25.6 Nelson D W, Sommers L E. A Rapid and Accurate Method for Proceedings of the. Indiana Academy of Science, 1975, 84: 456-462.

25.7 EPA Manual 600 4-79-020, March 1983, Method 410.4, 40CFR Part 136

26 Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

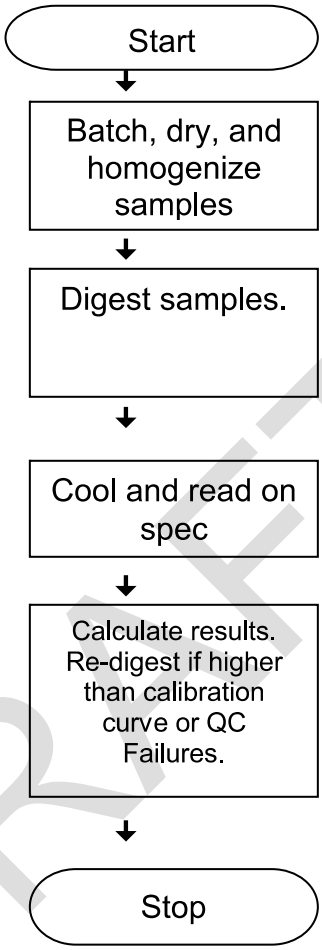
26.1 Attachment I: Flowchart

27 Revisions

Revision Number	Reason for Change	Date
S-GB-I-037-Rev.04	Updated SOP references throughout document. Section 2—Added reference to the LOD and LOQ Throughout document: Incorporated electronic preplog. Section 8, 10 and 11: Updated to pre-made standards.	15Aug2012
S-GB-I-037-Rev.04	Throughout document: Removed Method References for SW846 7196. Updated formatting following current revision of SOP: S-GB-Q-017 *Preparation of SOPs*. Section 7.2: Changed temperature to ≤6°C. Moved Attachments I & II into sections 15 &16	19Nov2014
S-GB-I-037-Rev.05	Signature Page: Updated from Inc to LLC, updated QM name. General: made administrative edits that do not affect the policies or procedures within the document. Throughout Document: Added information on Regulated Soils. Section 6: Addressed additional interferences. Section 8: Added definitions for TC, TIC, SOM and FOC. Section(s) 9 and 10: Updated to Table Format. Section 13: Added QC not previously listed. Section 19: Added 19.4. Section 22.2: Added SDS link. Section 25: Added Pace and TNI references. Section 27: Removed previous revision information that can be found in prior version of SOP	12Jan2018

Attachment I:

FLOWCHART



ATTACHMENT C-32

GAMMA SPECTROSCOPY ANALYSIS-PREP 901.1
PACE PITTSBURGH

Document Information

Document Number: ENV-SOP-GBUR-0088 **Revision:** 00

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Department(s): Rad Chem

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All Dates and Times are listed in: Central Time Zone

Signature Manifest

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Review

Name/Signature	Title	Date	Meaning/Reason
Raelyn Sylvester (005634)	Qa Analyst I	21 Feb 2019, 11:29:49 AM	Reviewed
Nasreen Derubeis (009976)	Quality Manager II	21 Feb 2019, 04:23:04 PM	Reviewed

STANDARD OPERATING PROCEDURE

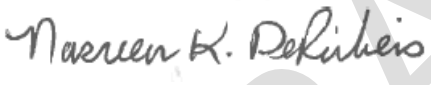
Gamma Spectroscopy Analysis - Sample Preparation

Method: 901.1

SOP NUMBER:	S-PGH-R-040-rev.07
REVIEW:	R. Kinney
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	PGH-R-040-6

APPROVALS

Department Manager/Supervisor — 03/01/18 Date



Senior Quality Manager — 03/01/18 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date

TABLE OF CONTENTS

1. Purpose

1.1 This SOP documents the analytical process of preparing and analyzing a variety of matrices for gamma emitters using the HPGe gamma spectrometry detector.

2. Scope and Application

2.1 This method describes the use of HPGe gamma spectroscopy detector to measure the gamma photons emitted from multiple radionuclides in a single sample. This technique makes it possible to determine the concentration of specific gamma emitters in homogenous aqueous and solid samples and is considered a non-destructive test for most matrices.

2.2 In the case of man made nuclides in drinking water, limits set forth in PL 93-523, 40 FR 34324 set the limiting concentration that will produce an annual dose equivalent to 4 mrem/year. This calculation is based on the consumption of 2 liters of drinking water per day and utilizes the 168 hour data listed in NBS Handbook 69. If multiple radionuclides are present, the sum of their annual doses must not exceed 4 mrem/year.

2.3 This procedure will be applicable only to the use of the HPGe detectors, but may be adapted to include the use of NaI(Tl) detectors, which are more efficient at detecting photons, but have poorer energy resolution. Due to the energy resolution advantage and the availability of the large active volume, HPGe detectors are recommended for measuring gamma emitting radionuclides.

2.4 This method is applicable for analyzing samples with gamma photon energy ranges from 60 to 2000 keV. The National Interim Drinking Water Regulations, Section 141.25, lists the required sensitivities of measurement for more hazardous gamma emitters. For compliance, the detection limits for photon emitters must be 1/10 of any applicable limits.

2.5 As written, this SOP is compliant with the EPA Method 901.1, Gamma Emitting Radionuclides in Drinking Water.

2.6 Solid samples analyzed using this SOP are prepared according to Pace SOP PGH-R-024, current revision (Radchem Sample Prep).

3. Summary of Method

3.1 Preserved samples are transferred to a standard geometry for counting purposes. Counting efficiencies must be determined for each standard geometry using a standard (known) radionuclide activity.

3.2 Samples are counted long enough to meet the required detection limit for each assessed radionuclide. Drinking water method detection limits are specified by the NIPDWR. Non-drinking water matrix method detection limits may be assigned by the client depending on their needs.

3.3 The gamma spectrum is processed by the gamma spectroscopy analysis software, and stored in the gamma system computer. The final analytical data are printed and results are entered into the LIMS database for reporting.

4. Interferences

4.1 Sample preservation ensures sample homogeneity in water and aqueous samples, by preventing the disposition of the nuclides on the container walls. Samples must be preserved in accordance with Section 8 of this SOP.

4.2 Solid samples must be dried and ground to a consistent mesh size. Settling must be minimized during the sample geometry preparation process by tapping and shaking the container to remove air and achieve level height/volume of sample in the can or jar.

4.3 Significant interferences occur when counting the sample on a NaI (Tl) detector and the sample radionuclides emit nearly identical gamma energies. This is minimized by using HPGe detectors.

4.4 Several radionuclides emit multiple gamma photons with multiple energies each having different abundances. The gamma software must be capable of calculating the final concentration of any one nuclide from another.

5. Safety

5.1 Procedures must be carried out in a manner that protects the health and safety of all personnel. Since this analysis is for a radioactive constituent, the sample must be treated as radioactive. Analysts must be trained as radiation workers and personal dosimeter worn.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves will be cleaned immediately.

5.3 When mixing or diluting acids, always add the acid slowly to water and swirl. Dilution of acids must always be done in a hood. Appropriate eye-protection, gloves, and lab coat must be worn.

5.4 Exposure to radioactivity and chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous and/or non-radioactive, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 In order to minimize the potential for cross contamination of high and low levels of radioactive samples, good housekeeping and good laboratory practices are essential and must be strictly adhered to.

5.6 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory.

6. Definitions

6.1 Refer to the Glossary Section of the most recent revision of the Pace Analytical Services, LLC. Quality Manual for the definitions of commonly used laboratory terms used throughout this SOP.

6.2 Batch: For gamma spectroscopy analysis, a batch consists of 20 samples of similar matrix in uniform geometry counted with a single set of QC samples. An analysis batch may be counted over several days by multiple persons, and is distinctly different from a preparation batch.

6.3 Geometry: For gamma spectroscopy analysis, geometry refers to a container with specified dimensions and physical composition utilized in sample counting to provide uniformity in reporting results from one sample to another. A calibration source is required for each type of geometry utilized in gamma spectroscopy counting.

6.4 Throughout this procedure, approximate weights and measures will be designated by the use of whole numbers when referring to masses exceeding one (1) gram or volumes in excess of one (1) milliliter. Measurements of mass and volumes so designated can be made with top loading balances, graduated cylinders, etc. For approximate measures below one gram or one milliliter, the word "approximately" must be used prior to the described weight or volume.

6.5 Throughout this procedure, exact or critical mass and volumes will be designated by the use of one or more decimal places. Measurements of masses and volumes so designated should be made with accurate analytical instruments such as analytical balances, calibrated pipettes, etc. The method utilized for obtaining the sample aliquot, whether on a balance, in a graduated cylinder, or by pipette, must be clearly annotated in the preparation logbook.

6.6 When measuring samples on a balance, the observed mass on the balance must be recorded in preparation logbooks to the lowest weight indicated on the balance. Sample aliquot masses must not be targeted. Once sample is removed from the sample container and transferred to a beaker, it must not be removed from the beaker.

6.7 Samples which are diluted to a specific geometry for analysis by gamma spectroscopy must be transferred to a new labeled container large enough to contain the entire diluted sample volume and clearly labeled as having been diluted. The label must include the dilution factor, date, analyst initials, and how the dilution was prepared, either by volume or by mass.

7. Responsibilities and Distribution

7.1 General Manager/Assistant General Manager (GM/AGM)

7.1.1 The GM/AGM has the overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the laboratory involving the collection and reporting of analytical data.

7.1.2 The GM/AGM and Senior Quality Manager/Quality Manager have final review and approval authority for all SOPs prepared within the laboratory.

7.2 Senior Quality Manager/Quality Manager (SQM/QM)

7.2.1 The SQM/QM will maintain a master file of all SOPs applicable to the operations departments.

7.2.2 The SQM/QM will assign a unique number to each SOP prepared prior to approval and distribution.

7.2.3 The SQM/QM will distribute SOPs to applicable personnel and maintain an accurate accounting of such distribution to ensure that the SOPs, in the hands of the users, are current and complete.

7.3 Department Manager/Supervisor

7.3.1 The Department Manager/Supervisor is responsible for ensuring all staff members read, follow, and are adequately trained in the use of the SOPs

7.3.2 The Department Manager/Supervisor coordinates the preparation and revision of all SOPs within the department whenever a procedure changes.

7.3.3 The Department Manager/Supervisor provides initial approval of all SOPs within the department.

7.3.4 The Department Manager/Supervisor makes recommendations for SOP revision to the SQM/QM via written memo.

7.4 Individual Staff

7.4.1 Individual staff members are responsible for adherence to the specific policies and procedures contained in the applicable SOPs.

7.4.2 Individual staff members will only use a signed, controlled copy of the SOP. Each person may make recommendations to the Department Manager/Supervisor for revising SOPs as the need arises.

7.4.3 Personnel are responsible for ensuring that any deviations from this SOP are reported to the Department Manager/Supervisor.

8. Sample Collection, Preservation, and Handling

8.1 Aqueous Samples

8.1.1 Containers used for sample collection must never be reused. Either plastic or glass containers may be used for sample collection.

8.1.2 Aqueous samples not requiring I-131 analysis must be preserved at the time of collection by adding enough concentrated (16N) HNO_3 to the sample to make the sample pH <2. Typically, two mL 16N HNO_3 per liter of sample is sufficient to obtain the desired pH.

8.1.3 Samples must be preserved within five days of collection. If samples are collected without preservation, they must be received by the laboratory and preserved within five days of collection. Following preservation with acid, samples must be held in the original container for a minimum of 16 hours. After a minimum of 16 hours has elapsed, the sample pH must be re-checked and verified to be less than the required pH of 2. The date and time, as well as the analyst initials, and results of the pH check must be

recorded in the pH Verification Logbook, along with any comments or further adjustments. The pH must be verified to be less than 2 for a minimum of 16 hours before analysis or transfer of sample.

8.1.3.1 For dissolved analysis, samples must be filtered through a 0.45μm membrane filter and then preserved to a pH <2.

8.1.3.2 For total analysis, the sample is not filtered, but is preserved to a pH<2.

8.1.4 Samples requiring I-131 analysis must not be preserved upon collection. The addition of acid to samples may cause the complete loss of iodine in the sample due to co-precipitation of iodide with chlorides found to be present in the sample.

8.1.5 Refrigeration is not required for aqueous or solid samples, but is recommended for all biological samples, especially urine.

8.1.6 Urine: It is recommended that the sample be preserved before a sample aliquot is taken to ensure that the requested analytes do not adhere to the sample container.

8.1.6.1 This is especially true if the sample has visible solids present.

8.2 Solid Samples: No preservation or refrigeration is required. Samples must be dried at 105°C and ground to a fine mesh according to Pace SOP PGH-R-024, current revision.

8.3 Analysts should consult with the Radiochemistry Department Supervisor for direction on samples which do not meet the above criteria. If samples are received non-compliant (not within the criteria of preservation) as dictated in section 8.1, then the Project Manager and the client must be notified. Sample analysis can only proceed after the client has given permission to do so.

9. Equipment and Supplies

9.1 HPGe detectors, >50cm3, Ortec, Canberra, or equivalent.

9.2 Gamma Spectrometry Analyzer with a minimum 2048 channels for HPGe or 512 for NaI(Tl).

9.3 Associated analysis software for each detector type, computer, and printer.

9.4 Standard geometry sample counting containers: 3.0L Marinelli beakers, 500 gram plastic wide mouth jar, 8 oz salmon can, 2 oz can, centrifuge tube, etc.

9.4.1 Marinelli beakers are the only analysis containers that may be re-used for sample analyses. Following analysis of samples in Marinelli beakers, transfer the sample to the original sample container or into a new appropriately labeled container.

9.4.2 Rinse the Marinelli beaker using ASTM Type II DI water into the sink/drain designated for sample disposal. To the Marinelli beaker

and lid, add an appropriate quantity of Contrad solution and add enough ASTM Type II DI water to create an adequate volume for washing. Scrub the interior surfaces of the Marinelli beaker and lid using a soft bristle brush or sponge pad. Once washing is complete, rinse the beaker and lid using ASTM Type II DI water until soap is removed. Transfer a minimum of 2.0 liters of 2 M nitric acid to the beakers and soak for a minimum of 30 minutes to dissolve trace levels of solids from the beakers. Rinse the beakers using ASTM Type II DI water and drain into the sink/drain designated for sample disposal. Hand-dry the beakers and lids using paper towels.

9.5 Additional geometries may be prepared as needed, however, for each geometry, a calibration source must be prepared and used for instrument calibration for the specific geometry prior to sample counting. A second source different from the calibration source must be prepared in each counting geometry for the performance of LCS analysis.

9.6 Top loader balance capable of weighing 1.00g to 3000.00g.

9.7 Graduated Cylinder, 1.0 Liter, Class A.

10. Reagents and Standards

10.1 ASTM Type II (DI) water for reagent and standard preparation and sample dilution. ASTM Type II DI water generated as specified in Pace SOP PGH-C-027, current revision.

10.2 Nitric acid, 16N: HNO3 (conc.), sp. gr. 1.42, 70.4%, high purity grade.

10.3 Nitric acid, 2N: Mix 124mL of concentrated nitric acid with ASTM Type II DI water and dilute to a final volume of 1.0L with ASTM Type II DI water.

10.4 Calibration and Control sample solutions must contain a minimum of 7 radionuclides to include low, mid, and high energy gamma photon emitters within the range of 60-2000 keV. All standard solutions must be NIST certified.

10.5 Sodium Chloride, NaCl, reagent grade.

11. Calibration

11.1 Specific Details regarding instrument calibration are documented in SOP-PGH-R-023, current revision. The following are general comments regarding the calibration process:

11.2 Prepare a stock solution containing a minimum of the following nuclides: Am-241 (or Pb-210), Cd-109, Co-57, Ce-139, Cs-137, and Co-60 (both energy lines).

11.3 Prepare as many geometries as desired for future analysis and count the sample to obtain a minimum of 10000 net peaks for each nuclide present.

11.4 Upon counting, follow the steps outlined in the operations manual to adjust the amplifier “gain” and analog to digital converter “zero offset” to locate the peak in the appropriate channel. For HPGe detectors, a 0.25 keV per channel calibration is recommended.

11.5 Counting efficiencies for the various energies are determined by comparing the activity counts to the known values. An energy versus channel number calibration is performed first followed by an instrument counting efficiency versus gamma energy calibration. The calibration process is completed for each container geometry and for each detector that is to be used for sample analysis.

11.6 Efficiency calibration equations are generated within the applicable software program. The efficiency curve is acceptable if the difference between the calculated efficiency and the applied efficiency is less than 10% for all radionuclides measured in the calibration source. This is automatically calculated during the calibration process but the analyst must be aware of this requirement and work with the Department Supervisor to ensure that each data points used complies with this requirement.

12. Procedure

12.1 Aqueous Samples:

12.1.1 The preferred geometry for aqueous samples is a 2.0 liter volume in a 3 Liter Marinelli beaker. Measure sample volumes by transferring to a 1.0 liter Class A graduated cylinder. For samples with 2.0 liters available for analysis, measure two 1.0 liter volumes. Transfer sample volumes into the labeled Marinelli Beaker. Record the sample volume for analysis in the sample preparation logbook. For samples that do not have 2.0 liters available for analysis, transfer the available volume into the graduated cylinder and record the volume in the sample preparation logbook. Add ASTM Type II DI water to dilute the total volume to 2.0 liters. Transfer sample volume into the labeled Marinelli beaker. In all instances, record the sample volume on the beaker lid in addition to the sample ID. Some clients prefer analysis of aqueous samples by mass. For these clients, measure the sample volume using the Class A graduated cylinder but transfer the sample into a tared Marinelli beaker. Record the sample mass in the sample preparation logbook. Never remove sample after dilutions are performed. Fortify diluted samples with additional nitric acid to ensure a starting pH of <2.

12.1.2 Prepare a batch method blank by weighing the same amount of ASTM Type II DI water into the same size clean labeled standard geometry container.

12.1.3 Perform gamma spectroscopy instrument daily checks in accordance with the current revision of the SOP PGH-R-023, Gamma Spectroscopy Instrument Operations.

12.1.4 Count the method blank, samples, and one duplicate sample for the desired amount of time to achieve the required MDC for all analytes desired to be reported to the client.

12.1.5 Count a laboratory control sample of the same standard geometry for a duration determined to generate a minimum ratio of 5:1 of analyte concentration versus analyte Minimal Detectable

Concentration for all quality control analytes. Ordinarily for aqueous samples, sufficient sample volume is not available for sample duplicate analysis. In these cases, perform a duplicate count of the LCS for precision assessment.

12.1.6 Process the data and enter the results into LIMS. Submit the data for review.

12.2 Solid Samples:

12.2.1 Dry and homogenize the samples in accordance with the current revision of the SOP PGH-R-024, Sample Preparation.

12.2.2 Weigh an appropriate mass of sample into the largest standard geometry available for which there is enough sample. Do not tamp the cans, as the process of tamping causes striation of the varied particle sizes of the soil. The final volume of the sample must be equivalent to the volume used during calibration. Using a clean, flat press, press the soil and add additional processed soil until the can is full. Record the mass of sample and the canning/sealing date in the gamma spectroscopy preparation logbook.

12.2.3 If Ra-226 analysis is desired, seal the container completely to allow for ingrowth and capture of the Ra-226 decay daughters. Ingrowth is considered ideal after 21 days, but with both the clients' and Supervisor's permission, a shorter ingrowth period may be used.

12.2.4 Prepare a method blank using a radionuclide-free reagent grade sodium chloride (NaCl).

12.2.5 Count the method blank, samples, and one duplicate sample for the desired amount of time to achieve the required MDC for all analytes desired to be reported to the client.

12.2.6 Count a laboratory control sample of the same standard geometry for a duration determined to generate a minimum ratio of 5:1 of analyte concentration versus analyte Minimal Detectable Concentration for all quality control analytes.

12.2.7 Process the data and enter the results into LIMS. Submit the data for review.

12.3 Miscellaneous Samples

12.3.1 In instances where the sample cannot be configured into an existing geometry for which there is a calibration, it may be necessary to prepare a calibration.

12.3.2 If a new calibration cannot be prepared for the new counting geometry, the sample must be counted using the closest geometry calibration comparable to the sample counting geometry.

12.3.3 For example, if a composite filter is received for analysis, and a calibration does not exist for this material, and preparing a

calibration for this material is not practical, the best geometry to use would be a solid geometry in the same size to which the filter composite can be tightly packed.

13. Calculations

13.1 Refer to the operation manuals for each of the operating systems for all gamma spectroscopy calculations and algorithms.

14. Quality Control

14.1 Daily instrument Quality Control checks for Gamma Spectroscopy Counting Systems must be completed following the instructions detailed in Pace SOP PGH-R-023 current revision, the SOP for Gamma Spectroscopy Counter Operations.

14.2 The LCS solutions or Well characterized material (WCM) must consist of at least 3 of the same nuclides as those used for calibration, one in each of the low, medium and high energy regions. The LCS solutions or WCM must come from a source other than that used for the calibration.

14.3 See Appendix I for performance indicator evaluation calculations and criteria.

14.4 Method Blank (MB)

14.4.1 One MB must be prepared for each analytical batch. The purpose of the MB is to monitor for cross contamination during the analytical process. When available, the MB should be prepared from a similar matrix as samples contained in the analytical batch. If appropriate blank matrix material is not available, ASTM Type II DI water (Reagent Blank) must be carried through the procedure for analysis of aqueous samples. For solid sample analyses NaCl must be used for MB analysis. A reagent blank may be used for sample correction purposes following approval of the Department Manager or a Manager-specified designeerand affected clients.

14.4.2 The results of the method blank must be less than the Contract Required Detection Limit (CRDL).

14.4.2.1 If the method blank is out of control, individual sample results may still be reportable if results are less than the CRDL (contract required detection limit) or greater than 10 times the blank result. Relative sizes of the sample and blank aliquots must be factored when making this determination (raw counts).

14.4.2.2 Additionally, the Z-score for the MB (Zblank) as discussed in Attachment I should be used to determine if the MB result indicates a positive detect or if the result could be a statistical aberration.

14.5 Laboratory Control Sample (LCS)

14.5.1 One LCS must be analyzed for each analytical batch. The LCS is usually a “static” source that is prepared once but used repeatedly for batch analysis.

14.5.2 Spike solution activities of the radionuclides analyzed in the LCS must be greater than 2 times their respective detection limit.

14.5.3 A reference material containing a known concentration of at least 3 of the radionuclides analyzed in the calibration and in the same matrix as the batch is analyzed with the batch.

14.5.3.1 If this material is not available, a well-characterized material (WCM) may be used.

14.5.3.2 If neither of these is available, ASTM Type II DI water may be spiked with the appropriate standard(s).

14.5.4 Percent Recovery Calculation

$$\%REC = \frac{(LCSConc)}{TrueValue} * 100$$

Where:

LCSConc = Analytical result of the LCS
TrueValue= Known concentration of the LCS

14.5.5 LCS %REC acceptance limits are 75-125% for each measured analyte.

14.5.6 Additionally, the Z-score for the LCS (Z_{LCS}) as discussed in Attachment I should be used to determine if the analysis variables for the LCS count (count duration, etc.) were sufficient to document acceptable analysis or if the result could be a statistical aberration.

14.6 Laboratory Control Sample Duplicate (LCSD)

14.6.1 A LCSD is not required for gamma analyses; however analysis of an LCSD must be utilized to measure batch precision whenever adequate sample volume is not available for sample DUP analysis. The LCSD must be analyzed in an identical fashion as the LCS and processed identically as for other samples.

14.6.2 The LCSD must pass the acceptance criteria for the LCS and the criteria established for duplicate precision.

14.6.3 Additionally, the Z-score for the LCS (Z_{LCS}) as discussed in Attachment I should be used to determine if the analysis variables for the LCS count (count duration, etc.) were sufficient to document acceptable analysis or if the result could be a statistical aberration. Likewise, the Z_{DUP} should be calculated.

14.7 Sample Duplicate (DUP)

14.7.1 One Duplicate Sample (DUP) may be randomly assigned within each batch. Analysis batch must include either analysis of a sample duplicate or a LCSD. The purpose of the sample DUP is to measure precision of the analytical process. Laboratory duplicates are not intended to assess precision related to the sample collection process. Sample collection precision can only be assessed through collection of duplicate samples at the time of

sample collection. The sample DUP is a duplicate volume of sample processed identically as other samples in the analytical batch.

14.7.2 Relative Percent Difference Calculation

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

Where:

R1 = Result Sample 1
R2 = Result Sample 2

14.7.3 Duplicate sample RPD acceptance limits are <25% for all gamma radionuclides reported.

14.7.4 Additionally, the Z-score for the Duplicate (Z_{DUP}) as discussed in Attachment I should be used to determine if the observed precision is acceptable. For low-level analysis results, it is highly likely that precision will not be within the percent recovery limits as recovery limits are generated using results with higher concentrations such as observed for LCSs.

14.8 Summary of QC related Activities:

Method Blank	One per Batch
Duplicate Sample	One per Batch
Matrix Spike	N/A
Matrix Spike Duplicate	N/A
Laboratory Control Sample	One per Batch
Laboratory Control Sample Dup	One per Batch for samples in absence of Duplicate sample.

14.9 Corrective Actions for Out-Of-Control Data

14.9.1 Method Blank (Reagent Blank) (MB/RB) – Individual samples that do not meet the acceptance criteria must be reanalyzed. If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

14.9.2 Duplicate (DUP) – DUP analysis that fails the replicate test must be reanalyzed to determine if analytical failure or sample heterogeneity was the cause of the problem.

14.9.3 Matrix Spike Recovery (MS) – Sample Matrix Spikes are not performed for gamma spectroscopy since the test is a non-destructive method for which chemical separations are not employed.

14.9.4 Matrix Spike Duplicate (MSD) - See comments for the Matrix Spike Analysis documented in section 14.9.3.

14.9.5 Laboratory Control Sample (LCS) – If an LCS analysis does not meet the acceptance criteria, the entire analytical batch must be re-prepped and reanalyzed.

14.9.5.1 The results of the batch may be reported, with qualification in the final report, if the LCS recoveries are high and the sample results within the batch are less than the reporting limit.

14.9.6 Laboratory Control Sample Duplicate (LCSD) – If an LCSD does not meet the recovery acceptance criteria, the entire analytical batch must be reanalyzed.

14.9.6.1 The results of the batch may be reported, with qualification, if the LCS recoveries are high and the sample results within the batch are less than their the reporting limit, and duplicate precision meets the acceptance criteria.

14.9.7 If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

14.10 Contingencies for Handling Out-of-Control or Unacceptable Data

14.10.1 Method Blank (Reagent Blank): If the sample is exhausted, evaluate the usefulness of the data in the final report.

14.10.2 Laboratory Control Sample: If sample is still available, recount the entire batch, otherwise evaluate the usefulness of the data in the final report.

14.10.3 Duplicates: If the sample is exhausted, evaluate the usefulness of the data in the final report.

15. Method Evaluation

15.1 Laboratory control samples are analyzed with each batch, the results are charted to monitor control limits and trending.

15.2 Each analyst must read and understand this procedure with written documentation maintained in their training file on the Learning Management System (LMS).

15.3 An initial demonstration of capability (IDOC) study must be performed. A record of the IDOC will be maintained on file in each analysts training file in the LMS.

15.4 On an annual basis, each analyst will complete a continuing demonstration of capability (CDOC).

16. Pollution Prevention and Waste Management

16.1 Place radioactive waste into appropriate receptacles.

16.2 Discard acidified samples and unusable standards into proper waste drains.

16.3 Dispose waste materials in accordance to type: Non-hazardous, hazardous, non-radioactive, radioactive or mixed.

17. References

17.1 Krieger, H. L. and Whittaker, E. L., Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, "Gamma Emitting Radionuclides in Drinking Water," Method 901.1, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH, August, 1980.

17.2 ASTM E181-93, Standard Test Methods for Detector Calibration and Analysis of Radionuclides, ASTM Standards, Vol. 12.02.

17.3 Table of Radioactive Isotopes, Brown and Firestone, Shirley editor, John Wiley & Sons, 1986.

17.4 Currie, L., Limits for Quantitative Detection and Quantitative Determination, Analytical Chemistry, Vol. 40. No. 3, Pg 586-593, 1968.

17.5 Currie, L., Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements, NUREG/CR - 4007, USNRC, 1984.

17.6 "Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)", July 2004.

17.7 Nuclide Identification Algorithms and Software Verification and Validation Manual, 07-0464-02, Canberra Industries, November 1993.

17.8 Peak Search Program Algorithm Manual, 07-0064, Canberra Industries, March 1985.

17.9 Gamma-Vision 3.0 Program Manual, Ortec, 2000.

17.10 "American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories", ANSI N42.23-1996.

17.11 Department of Defense Quality System Manual for Environmental Laboratories (DoD QSM), current version.

17.12 EML Procedures Manual, HASL-300, 27th Edition, Volume 1, 1990, Method 4.5.2.3 – Gamma.

17.13 Pace SOP PGH-R-023, current revision (Gamma Spectroscopy Instrument Operation).

17.14 Pace SOP PGH-R-024, current revision (Rad Sample Preparation).

17.15 Pace SOP PGH-C-027, current revision (Deionized Water Quality and Suitability).

17.16 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (most recently approved revision).

17.17 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.

17.18 Pace Analytical Services, LLC. - Pittsburgh Laboratory Quality Assurance Manual, current version.

18. Tables, Diagrams, Flowcharts, Appendices, etc.

18.1 Appendix I, Numerical Performance Indicators

19. Method Modifications

19.1 None

20. Revisions

Document Number	Reason for Change	Date
PGH-R-040-0	1. Table of Contents added (Updateable Version). 2. All references to Ge(Li) detectors replaced with HPGe. 3. Section 8 modified to specify non-preservation of samples requiring I-131 analysis and require specific authorization to proceed when non-compliant sample collection requirements are observed. 4. Section 9.5 updated to expand on requirement to use calibration source for detector calibration. 5. Section 10 modified to require use of ASTM Type II water and use of high-purity concentrated nitric acid. 6. Section 11 modified to include reference to the instrument operation SOP for calibration instructions. Order of calibrations energy followed by efficiency added. 7. Section 12.1.2 added instructions for performing and recording aqueous sample dilutions. 8. Method Evaluations Section added as Section 15. 9. Rotated previous Sections 15-18 as new Sections 16-19. 10. Updated references at 17.11 and 17.12 to include ANSI N42, 23 and reference to the TNI standard. 11. Renamed Section 19, “Deviations for Promulgated Methods” as “Method Modifications”	29May2012

Document Number	Reason for Change	Date
PGH-R-040-2	1. Annual review and update. 2. Added specifications for DI water as ASTM Type II DI water and included reference to SOP PGH-C-027, the SOP that documents the DI water production and testing process. 3. Updated references to require Department Manager or Manager-specified designee approval of technical modifications removing approval by a senior analyst. 4. Section 9, added procedure to be used for washing re-usable Marinelli beakers. 5. Section 12 modified to require recording of sample weights for volume analyses, dis-allowing the targeting of weights and removal of sample transferred for analysis. 6. Modified Section 14 to clarify the allowed use of empty geometry containers for solid sample MB analysis. 7. Modified Section 14 to clarify use of LCSDs for precision analysis in lieu of sample duplicates, if desired. 8. Modified Section 14.5 to allow use of “static” sources for batch LCS/LCSD analyses. 9. At 17.14 Added reference to *EML Procedures Manual*, HASL-300, 27th Edition, Volume 1, 1990, Method 4.5.2.3 – Gamma.	26Jun2013
PGH-R-040-3	1. Annual review and update 2. Section 6 – Updated for not targeting weights, recording measuring apparatus, not returning sample to containers once removed. 3. Section 8 – Updated for pH verification check and recording. 4. Added references to Pace SOP for Sample Preparation, ASTM Type II DI water preparation. 5. Reformatted document.	17Jul2014
PGH-R-040-4	1. Moved sections 8.1.4.1 & 8.1.4.2 under section 8.1.3.	03Dec2015
PGH-R-040-5	1. Section 12.2.5 modified to require use of sodium chloride for solid sample method blank analysis. 2. Section 10 modified to list sodium chloride as a chemical.	01Mar2016
PGH-R-040-6	1. Section 12.2 modified to eliminate the use of tamping to fill gamma analysis containers. 2. Section 12.2.4 added to define process for preparing solid PT samples on an “as-received” basis.	14Feb2017

Document Number	Reason for Change	Date
S-PGH-R-040-rev.07	3. Section 9.4 modified to remove reference to 1.0 Liter Marinelli Beakers. 4. Section 9.4.2 modified to document process used for acid-soaking Marinelli Beakers. 5. Section 9 modified to include Class A graduated cylinder and a top-loading balance as necessary equipment. 6. Section 10.3 modified to remove 1 N nitric acid as a reagent and add in 2 N nitric acid as a reagent. 7. Section 12.1.1 modified to document revised process for preparation of aqueous samples. 8. Sections 12.1.5 and 12.2.6 modified to remove a specific count duration for LCS samples. 9. Sections 14.9.3 and 14.9.4 modified to document that sample matrix spikes and MS duplicates are not analyzed for gamma-spectroscopy.	01Mar2018

Appendix I

(Numerical Performance Indicators)

1. **Method Blank (MB)**

1.1 The numerical performance indicator for the method blank is calculated by:

$$Z_{Blank} = \frac{x}{u(x)}$$

Where:
x = measured blank activity
u(x) = standard uncertainty in the blank measurement

1.2 MB performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. MB performance indicator values should be recorded on a control chart.

2. **Laboratory Control Sample (LCS)**

2.1 The numerical performance indicator for a laboratory control sample is calculated by:

$$Z_{LCS} = \frac{x - c}{\sqrt{u^2(x) + u^2(c)}}$$

Where:

x = Analytical result of the LCS
c = Known concentration of the LCS
$u^2(x)$ = combined standard uncertainty of the result squared.
$u^2(c)$ = combined standard uncertainty of the LCS value squared.

2.2 LCS performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. Performance indicator values should be recorded on a control chart.

3. Duplicates (DUP)

3.1 These criteria are applicable for the evaluation of the Duplicate, Matrix Spike Duplicate and Laboratory Control Sample Duplicates.

3.2 The numerical performance indicator for laboratory duplicates is calculated by:

$$Z_{Dup} = \frac{x_1 - x_2}{\sqrt{u^2(x_1) + u^2(x_2)}}$$

Where:
x_1, x_2 = two measured activity concentrations
$u^2(x_1)$, $u^2(x_2)$ = the combined standard uncertainty of each measurement squared.

3.3 Duplicate sample performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been

established as –2 to 2. DUP performance indicator values should be recorded on a control chart for each QC sample type (Dup, MSD, LCSD)

4. **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

4.1 The numerical performance indicator for a matrix spike sample is calculated by:

$$Z_{MS} = \frac{x - x_0 - c}{\sqrt{u^2(x) + u^2(x_0) + u^2(c)}}$$

Where:
x = measured concentration of the spiked sample
x_0 = measured concentration of the unspiked sample
c = spike concentration added
$u^2(x)$, $u^2(x_0)$, $u^2(c)$ = the squares of the respective standard uncertainties of these values.

4.2 MS performance for all matrices is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. MS performance indicator values should be recorded on a control chart.

ATTACHMENT C-33

GAMMA SPEC INSTRUMENT OPERATIONS – 901.1
PACE PITTSBURGH

Document Information

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ENV-SOP-GBUR-0078

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Nasreen Derubeis (009976)	Quality Manager II	22 Feb 2019, 11:52:15 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Nasreen Derubeis (009976)	Quality Manager II	22 Feb 2019, 11:01:17 AM	Approved
Richard Kinney (005816)	Manager - Lab Operations	22 Feb 2019, 11:40:31 AM	Approved

1. Purpose

This procedure describes the general operation and maintenance of the gamma spectroscopy instrumentation utilized by the Pace Analytical Services, LLC. For additional operational details not discussed in this SOP, the user should consult the instrument and software operations manuals referenced in Section 17. These documents are located in the gamma instrument room.

2. Scope and Application

2.1 Pace SOP ENV-SOP-GBUR-0044 current revision (Laboratory Equipment) addresses general requirements regarding the purchase, installation, operation, maintenance, and disposition of laboratory equipment.

2.2 This procedure addresses the process for analyzing sample sources generated as the product of current revision analytical SOPs: ENV-SOP-GBUR-0080 current revision (Analysis of Neutron Dosimeter Wires by Gamma Spectroscopy), ENV-SOP-GBUR-0081 current revision (Analysis of Neutron Dosimeter Capsules for Cesium-137), and ENV-SOP-GBUR-0082 current revision (Iodine-129 Analysis).

2.3 Samples for non-destructive gamma spectroscopy analysis are prepared using the current revision of Pace SOP ENV-SOP-GBUR-0079 current revision.

2.4 This procedure applies to the measurement of gamma particles emitted from a variety of sample types. Several detection systems are utilized, but all analysis, data processing, storage, and reporting can be performed using different software commands within one flexible user interface for each system type. Each system type has an independent user platform consisting of a computer and detector-specific software that controls instrument operation, spectral analysis, and calculations.

2.5 Sample specific preparation is not discussed in this SOP. The final sample geometry must conform to one of the existing calibration geometries at the time of counting. Deviations in sample geometry from the calibration geometry require a new calibration.

2.6 This procedure describes the performance of the gamma detector efficiency, energy, and FWHM calibration; monthly background determination; and daily or prior to use (PTU) continuing calibration verification checks. Also discussed are the general systems operating requirements and maintenance.

2.7 This procedure is applicable to gamma spectroscopy detectors of HPGe “p” type, HPGe “n” type, or LeGe (LEPS) type, utilizing multiple software components.

2.8 Gamma spectral analysis software can decay correct data for all nuclides based on the individual half-lives listed in the nuclide library. Decay correction for individual nuclides is applied from the client-specified collection date and time to the instrument analysis start date and time.

3. Summary of Method

3.1 The system is calibrated for efficiency, energy, and resolution full width-half max (FWHM) for all new counting geometries. Sources may be commercially available or laboratory prepared with NIST traceable materials

and equipment. Long backgrounds (minimum of 1000 minute duration) are performed within 30 days prior to all sample analyses or more frequently as necessary. Daily (or PTU) checks of the efficiency, energy, FWHM, and background are performed to ensure detector function prior to use.

3.2 Samples are counted upon completion of a daily check, utilizing the system software available, on a detector capable of detecting the nuclides of interest in the sample. Once counting is complete on Canberra system, the system software automatically processes the data, and the primary count analyst must review spectra and enter the data into LIMS for client delivery. For the Ortec system, the user must manually process data.

4. Interferences

4.1 Sample counting geometries must be of uniform consistency for accurate results. Phase separations in liquid samples or grading in solid samples may bias sample results.

4.2 In order to prevent possible detector contamination, aqueous samples should be contained in a plastic bag or other suitable device to ensure secondary containment, with the first container being the durable sample container such as a Marinelli beaker or plastic “jar.” In the event of a spill, the detector must be cleaned immediately, and backgrounds must be measured.

4.3 High activity samples may cause excessive dead time in detectors and should be analyzed utilizing a geometry that increases the sample to detector distance, thereby decreasing the observed count rate. Optionally, a portion of sample is diluted using inert sand and re-prepared in the desired geometry.

4.4 Detector electronics must be kept cool while counting is in progress. Liquid nitrogen is stored in a dewar at the base of the instrument for this purpose. The level must be maintained by regularly filling the dewar with liquid nitrogen. System performance is greatly affected by temperature fluctuations, and damage to detectors can result from operating without proper cooling.

4.5 Many radioisotopes emit gamma rays that are indistinguishable (by energy) from other gamma-emitting radioisotopes. One such example, Ra-226, emits a predominant gamma ray very near that of a gamma ray emitted by U-235. Common gamma spectroscopy systems cannot easily identify the source of the gamma ray. For this reason, interfering gamma rays should be excluded from use for quantitation. Alternatively, if the interfering gamma rays are used for quantitation, the results must be qualified as potentially biased due to conflicts with other radionuclides.

5. Safety

5.1 Procedures must be carried out in a manner that protects the health and safety of all personnel.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves will be cleaned immediately.

5.3 When mixing or diluting acids, always add the acid slowly to water and swirl. Dilution of acids must always be done in a hood. Appropriate eye protection, gloves, and lab coat must be worn.

5.4 Exposure to radioactivity and chemicals must be maintained as low as reasonably achievable therefore, unless they are known to be non-hazardous and/or non-radioactive, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 In order to minimize the potential for cross contamination of high and low levels of radioactive samples, good housekeeping and good laboratory practices are essential and must be strictly adhered to.

5.6 Organic samples of unknown content must be handled with extreme caution and under the direct instruction of a senior analyst. Direct treatment of organic matrices with strong oxidizing chemicals such as nitric acid and/or hydrogen peroxide is strictly prohibited.

5.7 Hydrofluoric acid is particularly hazardous because a serious skin exposure may cause no immediate sensation of pain. The acid penetrates the skin and spreads internally, causing tissue damage deep under the skin. The resulting burn is painful, difficult to treat, and easily infected. Gloves must be checked for pinhole leaks before use. They must be rinsed before they are removed and must be discarded after use. HF burn gel shall be put on suspected HF burns after flushing (except the eyes) until medical help can be obtained. Medical attention shall be sought even if suspicions arise after working hours. Contact the group leader immediately for further information if an HF burn is suspected.

5.8 In addition, HF vapors are also hazardous. Exposure can cause permanent damage. Breathing HF vapors even for a short time and at a low temperature can be injurious to the respiratory system, and even fatal. All such direct contact must be avoided.

5.9 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the SDS files maintained in the laboratory.

5.10 Refer to the Pace Analytical Services, LLC. – Pittsburgh Chemical Hygiene Plan/Safety Manual for the specific safety requirements to be followed when working in the laboratory.

5.11 The toxicity and carcinogenicity of each reagent used in this procedure has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. At a minimum, personal protective equipment must include a lab coat, gloves, and safety glasses.

5.12 Analysts must be familiar with the Safety Data Sheets (SDS) for all chemicals and reagents used in this procedure, and the location of the SDS within the laboratory.

6. Definitions

6.1 Hardware equipment and software terms are defined in the applicable operating manuals by the equipment and/or software manufacturer.

6.2 Refer to the current Quality Manual for definitions.

7. Responsibilities and Distribution

7.1 General Manager/Assistant General Manager (GM/AGM)

7.1.1 The GM/AGM has the overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the laboratory involving the collection and reporting of analytical data.

7.1.2 The GM/AGM and Senior Quality Manager/Quality Manager have final review and approval authority for all SOPs prepared within the laboratory.

7.2 Senior Quality Manager/Quality Manager (SQM/QM)

7.2.1 The SQM/QM will maintain a master file of all SOPs applicable to the operations departments.

7.2.2 The SQM/QM will assign a unique number to each SOP prepared prior to approval and distribution.

7.2.3 The SQM/QM will distribute SOPs to applicable personnel and maintain an accurate accounting of such distribution to ensure that the SOPs, in the hands of the users, are current and complete.

7.3 Department Manager/Supervisor

7.3.1 The Department Manager/Supervisor is responsible for ensuring all staff members read, follow, and are adequately trained in the use of the SOPs

7.3.2 The Department Manager/Supervisor coordinates the preparation and revision of all SOPs within the department whenever a procedure changes.

7.3.3 The Department Manager/Supervisor provides initial approval of all SOPs within the department.

7.3.4 The Department Manager/Supervisor makes recommendations for SOP revision to the SQM/QM via written memo.

7.4 Individual Staff

7.4.1 Individual staff members are responsible for adherence to the specific policies and procedures contained in the applicable SOPs.

7.4.2 Individual staff members will only use a signed, controlled copy of the SOP. Each person may make recommendations to the Department Manager/Supervisor for revising SOPs as the need arises.

7.4.3 Personnel are responsible for ensuring that any deviations from this SOP are reported to the Department Manager/Supervisor.

8. Sample Collection, Preservation, and Handling

8.1 Refer to the applicable SOP and certified test method for sample collection and preservation requirements. The list of applicable SOPs and reference methods is located in Sections 2.2 and 2.3.

8.2 Aqueous samples should be bagged to prevent leaking that may contaminate detectors.

8.3 Solid samples should be sealed in order to prevent detector contamination in the event that the sample is dropped.

8.4 The maximum hold time for most analytes measured by this SOP is 180 days from sample collection to sample analysis. For I-131, the hold time is 8 days from samples collection to sample analysis.

9. Equipment and Supplies

9.1 High Purity Germanium (HPGe) “p” type, “n” type and Low-Energy Germanium (LeGe) or Low-Energy Photon Spectrometer (LEPS) type detectors.

9.2 Computer Data Analysis system run on a Canberra instrument-compatible computer. Canberra utilizes multiple software applications joined together to form the Genie 2000/Procount software package. For issues/processes not addressed within this SOP, refer to the instrument manual current revision.

9.3 Ortec GammaVision® software. Ortec GammaVision®-compatible computer. For issues/processes not addressed within this SOP, refer to the instrument manual current revision.

9.4 Front End Electronic components:

9.4.1 High Voltage Power Supply: supplies power to detectors.

9.4.2 Analog to Digital converter: converts analog voltage from crystal to digital readout.

9.4.3 Multi Channel Analyzer (MCA): Detector acquisition and control is performed through an MCA. The MCA is supported through a combination of hardware and software and functions as the analyzer. It is the link between spectra and the computer system.

9.5 Kim wipes® or lint free cloths.

9.6 Plastic bags.

10. Reagents and Standards

10.1 Reagents should be prepared from reagent grade chemicals, unless otherwise specified below. Reagent (DI) water must be at least ASTM Type II quality or better. NOTE: Consult Safety Data Sheets for the properties of these reagents, and how to work with them.

10.2 Distilled or deionized water, ASTM Type II generated as specified in Pace SOP ENV-SOP-GBUR-0008, current revision.

10.3 Liquid nitrogen, ACS grade, for detector cooling.

11. Calibration and Quality Control

Note: Refer to the instrument operations manual for instructions regarding specific system setup options.

11.1 Daily Checks

11.1.1 Daily or prior to use, a continuing calibration verification (CCV) check source is counted for five minutes to determine detector function, assessing the energy, efficiency (activity), and resolution (FWHM) calibrations.

11.1.2 High Purity Germanium Detectors with associated electronics are sensitive to temperature variations within the count room environment. The probable effect of variations in temperature is the slight drifting of gamma peaks in the high-energy range (>1MeV) of the analytical spectrum. Marginal shifts in the ambient temperature do not cause spectral shifts that would negatively impact analytical results. In order to optimize gamma spectral analysis, the system fine amplifier gain may be adjusted to correct for minor peak drifting. This process applies exclusively to Ortec gamma detectors.

11.1.3 Gain Adjustment

11.1.3.1 Place the check source used for instrument performance checks on the endcap of the applicable detector. Perform a manual count of the check source while observing the spectral location of the 1332.5 keV peak of Co-60. Adjust the system fine gain incrementally in order to position the peak at channel 5330. Record the fine gain adjustment value in the detector Maintenance Logbook.

11.1.3.2 Clear the analysis spectrum and re-start the qualitative count of the check source. Confirm the location of the 1332.5 keV peak to be centered at channel 5330. Clear the analysis spectrum and perform performance checks as documented below.

11.1.4 Each energy, efficiency (activity), and resolution (FWHM) is assessed using defined tolerances for each parameter. For the efficiency (activity) assessment, the defined tolerance is used as a total allowable deviation for the count source. For control purposes, the tolerance is divided by 3.0 to generate a standard tolerance deviation. Warning limits are set at +/- 2.0 standard tolerance deviations and control limits are set at +/- 3.0 standard tolerance deviations. The assessments of peak energy and resolution (FWHM) are not statistical measurements. For these variables, tolerances have been set based on manufacturer's recommendations. Limits have been set to establish a warning and control boundaries.

11.1.5 Min/Max control limits are established at +/- 3% for efficiency. This tolerance was chosen based upon the requirements cited in the DOD QSM Table B-17 (MARLAP 18.5.6.2)

11.1.6 The low warning limit for energy has been established as -0.25 keV difference from the known peak energy. The low control limit has been established as -0.50 keV from the known peak energy. The high warning limit and high control limit for energy have been established as 0.25 keV and 0.50 keV, respectively.

11.1.7 The control assessment for resolution is measured as a ratio of the source check peak resolution versus the calibration-defined resolution. The low warning limit for resolution (FWHM) has been established at 0.9 with a low control limit at 0.8. The high warning limit and high control limit for resolution (FWHM) have been established as 1.2 and 1.5 respectively.

11.1.8 A minimum five minute daily background of the empty shield is conducted and compared to the running median background counts to determine if the detector shield has been contaminated.

11.1.8.1 The control limit for the daily background count has been established statistically using a representative population of background counts. Warning and control limits have been established for each detector system with warning limits established at +/- 2.0 standard deviations of the system total background count rate in units of counts per second and control limits set at +/- 3.0 standard deviations of the system total background count rate in units of counts per second.

11.1.9 To perform the daily check, place the designated check source in the sample holder onto the shield, and close the cover. For the Canberra system: Select QC, then CALIBRATION CHECK. Choose the appropriate detector from the dropdown list. Select QCC-A (or the appropriate detector-specific file), then Control Det A (or the appropriate detector-specific control program), followed by OK to initiate the five-minute count. For the Ortec system: Double-click appropriate desktop icon (Det “#” QA Daily Check). Click “OK” at the following prompt.

11.1.10 Upon completion, the QC report will automatically be generated. QC reports are stored electronically and may be retrieved for performance troubleshooting. Data from each QC assessment are automatically loaded into the QA database for automated assessment and charting.

11.1.11 The analyst must verify that the detector meets the control criteria. To determine system acceptance for use, review the instrument check printouts for the assessment criteria OR review the control chart for each performance parameter monitored. QA assessment is software controlled within each counting system type. Control limits are statistical for activity assessments and are based on generally recognized tolerances for resolution and peak location.

11.1.12 Any daily check not meeting the control criteria must be recounted. Check the positioning of the source within the detector and verify the correct placement of the check source prior to initiating a recount. Two consecutive acceptable recounts are required for the detector to be in service. If the recount is unsuccessful, the detector must be locked out until a successful daily check is performed. If a detector fails several consecutive daily checks, the cause must be investigated. Consult with a senior analyst on how to proceed.

11.2 Monthly Background Measurement

11.2.1 Every month, at a minimum, (or within thirty days of sample analysis, at a minimum) an extended background count is completed for use in sample analysis. Additionally, backgrounds must be re-acquired following calibration, detector cleaning due to confirmed contamination, or maintenance. Typically, extended background counts are performed weekly. For all sample analyses, the prior (but most recent) background must be used for sample analysis.

11.2.2 Clean the detector shields prior to counting backgrounds with a lint free cloth dampened with ASTM Type II DI water.

11.2.3 Ensure daily checks have been performed and detectors have passed control criteria prior to performing background counts.

11.2.4 Canberra System:

11.2.4.1 For the Canberra system: Start counts by selecting COUNT and Start A LONG BACKGROUND COUNT. Choose the appropriate detector when prompted, followed by LONG BKG and NO SPECIFIC GEOMETRY. Select OK to begin the acquisition. Select OK in the following window, since all of the criteria has been pre-selected. The detector will begin counting. The shield cover must remain closed during the count.

11.2.4.2 When counting is complete, a background report will automatically be generated.

11.2.5 Ortec System:

11.2.5.1 For the Ortec system, ensure that the detector shield is empty. Perform step 12.1.6 as indicated below, acquiring an empty chamber count using a count time of 1000 minutes (60,000 seconds). The convention for naming the background count is LB (Long-background) Detector Number (DX), date of counting (MMDDYY). A background count performed for detector 2 on June 1, 2015 would be named, “LBD2060115.”

11.2.5.2 The Ortec Background Spectrum must be analyzed using each combination of detector/geometry calibration/library to be used for sample analysis.

11.2.5.3 Upon completion of the background count, recall the background spectrum into the processing “buffer.” To apply the appropriate calibration file: from GV toolbar (Fig 1. below), click CALIBRATION and select RECALL CALIBRATION. From the list of archived calibration files, select the file for the appropriate geometry for which to create the background analysis file. Next, select the appropriate nuclide library. From the GV toolbar, click LIBRARY and click SELECT.

11.2.5.4 Click ANALYZE, place cursor over SETTINGS and select SAMPLE TYPE. A new window will appear into which

sample data will be entered. From the SAMPLE tab (Fig 2.), click BROWSE, near the file entry blank, and select the appropriate .sdf file. Also from the SAMPLE tab, select the appropriate library and calibration files.

11.2.5.5 For the background spectral analysis, the sample size and decay correction date are not necessary. The "Peak-Background Correction" (PBC) file created for the background analysis only requires the energy calibration file which relates peak channel to energy and the library file which relates peak energy to detected analyte. At the bottom of the window select CLOSE and click YES, when prompted to save.

11.2.5.6 Lastly, from the GV toolbar, click ANALYZE and ANALYZE ENTIRE SPECTRUM IN MEMORY. If the printer output has been selected, the analyzed report will be printed.

11.2.5.7 To create the individual "Peak-Background-Correction" (PBC) files, click "ANALYZE" on the GV menu bar. Select "SETTINGS". Select "Peak Background Corrections", then "Select PBC" Choose the file for a recently-completed PBC file and select "OPEN."

11.2.5.8 Using the cursor, depress the "Cut" button repeatedly until all analytes have been removed from the active PBC file. Once all analytes have been removed, select the window in the very upper left-hand corner of the PBC box. Select the "Show Background Analysis" option.

11.2.5.9 The system will show all of the available un-formatted output "UFO" files for each sample spectrum previously analyzed. Find and choose the .UFO file for the analyzed background file you wish to create the PBC file from. The .UFO file will have the name of the background spectrum with a .UFO extension. For the example cited in section 11.2.5.1 above the .UFO file will correspond as "LBD2060115.UFO."

11.2.5.10 Once the .UFO file has been high-lighted, select the window in the very upper left-hand corner of the PBC window. Select, "Save PBC table as"…." Type in the name for the new detector/geometry/library PBC combination and select, "Save."

11.2.6 For each detector platform, the analyst must check the report and compare it to previous long backgrounds to determine if contamination is present.

11.2.7 Background changes must be tracked to determine if detectors need more vigorous cleaning. All detectors which do not pass the control criteria must be locked out to prevent accidental use during sample analysis.

11.2.8 The background count for detectors failing control criteria may be re-acquired following maintenance to improve background performance, including reassessment of potential contamination sources and re-cleaning of the detector shields.

11.3 Detector Calibration

11.3.1 Efficiency, energy, and FWHM calibrations for specific counting geometries must be completed initially or following major system maintenance, such as detector replacement. Calibration for specific counting geometries must be performed following major analytical SOP changes that would change the final geometric configuration of the final counting source. For example, an analytical change resulting in counting an aqueous sample in a 1.0L Marinelli beaker to counting an aqueous sample in a 0.5L Marinelli beaker requires calibration for the additional geometry.

11.3.2 Calibrations must be performed using the same protocols as used in sample analysis, with adjustment to sample count time being the only factor to change. Calibration instructions for each analysis type are included in the current revision of each applicable SOP. The list of applicable SOPs and reference methods is located in Sections 2.2 and 2.3 of this SOP.

11.3.3 Although the gamma analysis software contains features which would allow manual integration of regions of interest, this feature has been disabled. Manual integrations of peaks is not allowed.

11.3.4 Gamma Spectroscopy Instrument Calibration Requirements

11.3.4.1 Energy calibrations are performed prior to geometry-specific efficiency calibrations. The energy calibration documents the peak channel number to the peak energy. Geometry efficiency calibrations relate the peak energy to the detector response as percent of emissions observed versus emitted.

11.3.4.2 A minimum of 10000 net counts for each nuclide of interest utilized in the calibration source must be obtained. Nuclides of interest should be chosen in order to encompass various energy ranges consistent with those encountered in routine sample counting. Typically, a minimum of ten nuclides, are utilized in a calibration source. For each geometry-specific efficiency calibration performed, a known standard reference material (SRM) in an identical configuration is counted and assessed using the statistical control limits used for Laboratory Control Samples (LCS) in the related Gamma analysis SOP. Sources used for the initial calibration verifications of a newly-established calibration must incorporate the use of a SRM that is not related to the calibration source parent solution. Subsequent annual calibration verifications are performed as specified in sections 11.3.4.4 and 11.3.4.5.

11.3.4.3 Data are analyzed by the Genie 2000/Procount® or Ortec GammaVision® software package.

11.3.4.4 Calibrations must be verified annually for all aqueous geometries, including those used for drinking water analysis. For drinking water analyses, the same source utilized for the initial calibration may be used for verifying the calibration. A newly-prepared verification source may be used for DW calibration verification if the original calibration source is no longer available. For other aqueous geometries, annual calibration verification is performed using a known standard reference material (SRM) un-related to the initial calibration source. The verification source is prepared in an identical configuration as the initial calibration source and is counted and assessed using the statistical control limits used for Laboratory Control Samples (LCS) in the related Gamma analysis SOP. In all instances, a minimum of three of the nuclides used in the initial calibration must be used to verify the calibration. The three nuclides should span the energy range (low, mid, high) of the initial calibration, for example Am-241(Pb-210), Cs-137, and Co-60. The verification source result for all three nuclides, individually, must be within 10% of the known nuclide concentration for the verification to be acceptable.

11.3.4.5 For all other calibration geometries and matrices, calibrations must be verified annually. Calibration verifications are performed using sources un-related to the initial SRM used for calibration. Calibration standard reference materials (SRMs) such as spiked soils are purchased in limited quantities that do not allow simultaneous creation of all solid calibration sources used by the lab. For this reason, SRM material is often recycled to perform all of the necessary geometry calibration verifications. A minimum of three of the nuclides representative of the initial calibration energy range must be used to verify the calibration. The three nuclides should span the energy range (low, mid, high) of the initial calibration, for example Am-241(Pb-210), Cs-137, and Co-60. The verification source result for all three nuclides must be within 10% of the known nuclide concentration for the verification to be acceptable.

12. Operating Procedures

12.1 Sample Counting and Processing

12.1.1 Before beginning any counts, the detector run log information must be completed. This information includes: the date, analyst, and passing detectors.

12.1.2 Prior to counting, ensure that daily checks have been completed satisfactorily and the detector is free of check sources and contamination.

12.1.3 Insert the sample to be counted and record the detector in which it will be counted in the gamma instrument run log. Record all sample

information in the run log. Place sample into the detector and close the shield lid.

12.1.4 For the Canberra system: select COUNT and START AND COUNT from the Procount-ESP Screen. Select the detector, then choose the ANALYTICAL SEQUENCE FILE (ASF), which will determine the criteria by which the sample will be analyzed. Choose the geometry, then select OK to start the acquisition. Complete the sample information form, and select OK. A report will be generated automatically upon count completion.

12.1.5 For analysis of nuclear dosimetry chain samples, it is imperative that peak-fitting algorithms are optimized to ensure accuracy and precision. The primary peak for Co-58 analysis is located at 810.6 keV. An interfering peak of unknown origin is located near 821.4 keV. Lower peak sensitivity settings may cause inclusion of the 821.4 keV peak for the Co-58 peak at 810.6 keV, causing a high-biased result. This issue is controlled through proper assignment of the peak sensitivity setting. The default peak sensitivity setting has been established as 8.0. The sample spectra for nuclear dosimetry chain samples must be reviewed manually, with the 821.4 keV peak reviewed on-screen to ensure optimal peak fitting. Visual inspection of peaks without an optimal fit will have a noted inclusion of the peak located at 821.4 keV. Samples with this unacceptable characteristic must be re-analyzed manually including a peak-sensitivity adjustment until visual inspection confirms an acceptable peak-fit. Confirmation of the peak sensitivity assessment is documented on the respective gamma analysis report by adding a check-mark to the recorded peak sensitivity setting, with the assessors initials and date.

12.1.6 For the Ortec system: Double-click the appropriate desktop icon (Det "#" Sample). When prompted, enter the requested sample information. After entering the time, in seconds, select CLOSE. Finally, click OK on the following popup. Upon completion of sample counting, the user will be required to manually process data

Fig 1. (GV toolbar)

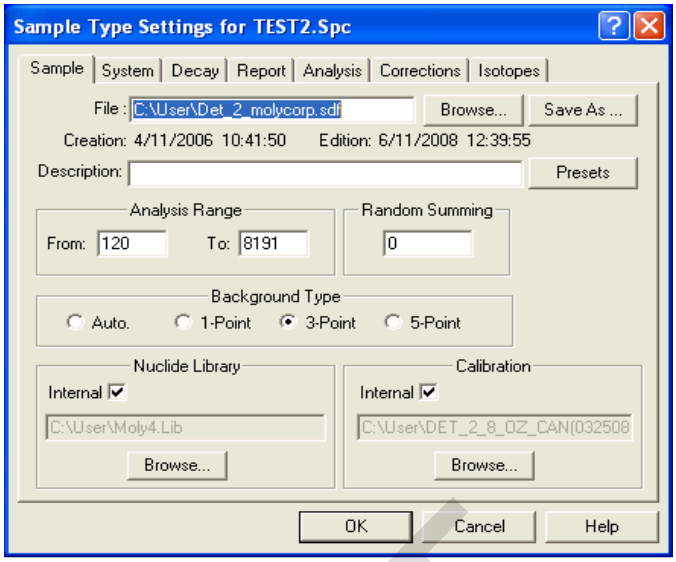

Fig 2. (Sample Tab)

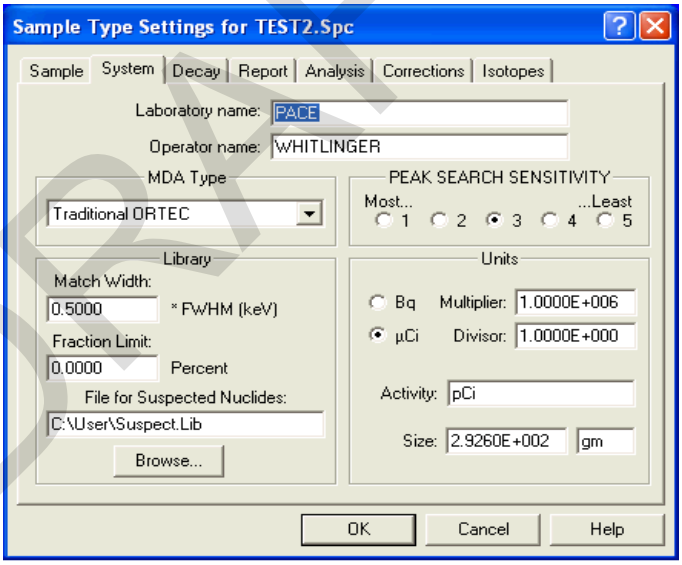

Fig 3. (System Tab)

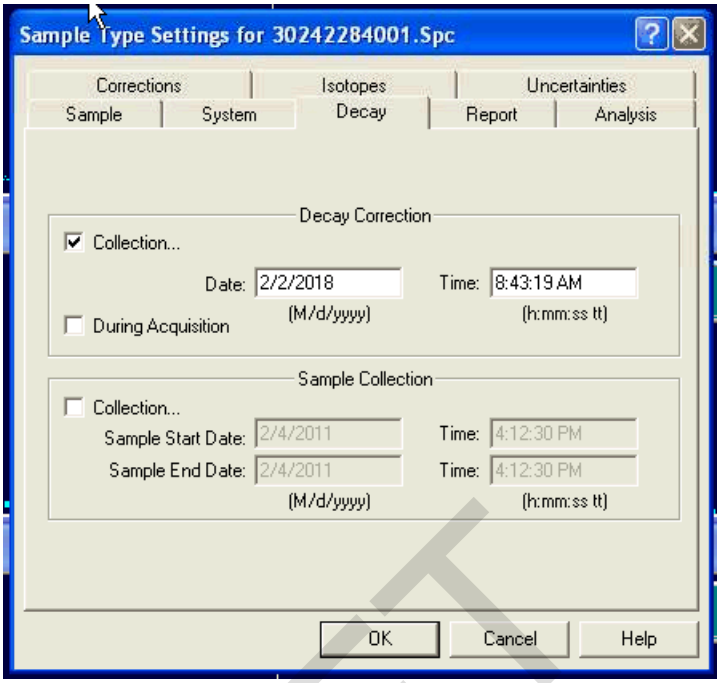

Fig 4. (Decay Tab)

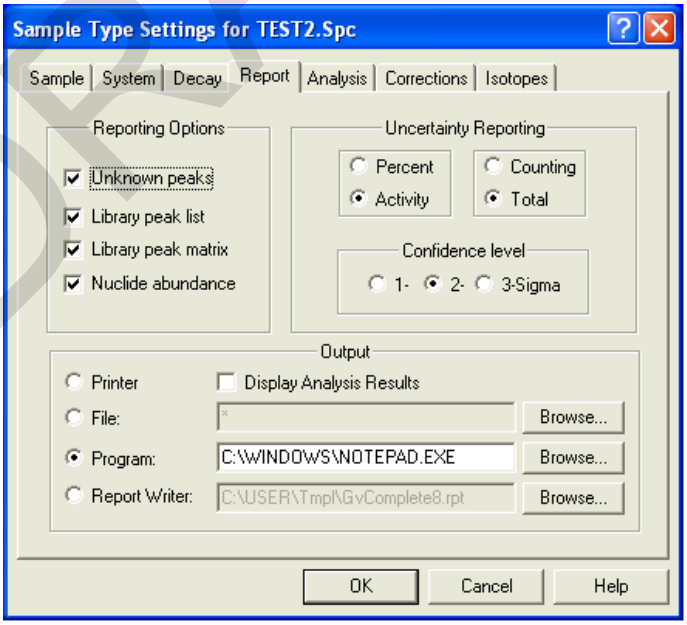

Fig 5. (Report Tab)

12.1.7 To process data from the Ortec system, the analyst must first recall the desired spectrum, using the GammaVision(GV)® program. To apply the appropriate calibration file: from GV toolbar (Fig 1.), click CALIBRATION and select RECAL INFORMATION. From the list of

archived calibration files, select the most recent file for the appropriate geometry. Next, select the appropriate nuclide library. From the GV toolbar, click LIBRARY and click SELECT.

12.1.8 Click ANALYZE, place cursor over SETTINGS and select SAMPLE TYPE. A new window will appear into which sample data will be entered. From the SAMPLE tab (Fig 2.), click BROWSE, near the file entry blank, and select the appropriate .sdf file. Also from the SAMPLE tab, select the appropriate library and calibration files.

12.1.9 From the "System" tab (Fig 3.), enter the sample size, in grams/Liters. From the "Decay" tab (Fig 4.), enter the sample collection date and time. From the "Report" tab (Fig. 5), select the output option, printer or file. At the bottom of the window select CLOSE and click YES, when prompted to save.

12.1.10 Finally, from the GV toolbar, click ANALYZE and ANALYZE ENTIRE SPECTRUM IN MEMORY. If the printer output has been selected, the analyzed report will be printed.

12.1.11 The gamma report must be reviewed by the analyst, and the spectrum must be viewed and compared to the report. Sign the report and enter the calculated activities into LIMS for the final report.

12.1.12 Manual integrations of peaks is not allowed and the feature for this has been disabled.

12.2 Maintenance

12.2.1 Detector

12.2.1.1 Detectors can be cleaned and maintained with ASTM Type II DI water and lint free cloths, or KImwipes®.

12.2.1.2 Removal and replacement of any detector requires a new calibration be established and must be recorded in the equipment maintenance logbook.

12.2.1.3 Detector temperature must be maintained with liquid nitrogen. Maintaining the level of liquid nitrogen in the dewar under each detector is crucial for system operation.

12.2.2 Major Instrument Maintenance

12.2.2.1 Consult with a senior analyst prior to performing troubleshooting steps to identify the source of system problems. Refer to the instrument manual for guidance on diagnosing system problems.

12.2.2.2 Consult with the manufacturer for troubleshooting major system problems.

12.3 All maintenance functions involving power disruptions to the instrument must be documented in the appropriate instrument maintenance logbook at the time of occurrence.

13. Calculations

13.1 Refer to the Canberra "Spectroscopy Applications Algorithms and Software Verification and Validations Manual 07-0368, September 1991" for analysis calculation documentation.

14. Quality Control

14.1 See Section 11.0 of this SOP for system calibration and for system Quality Control requirements.

14.2 Analytical batch Quality Control is documented for each analyte in the applicable SOP. The list of applicable SOPs and reference methods is located in Section 2.2 and 2.3.

14.3 Corrective Actions for Out-Of-Control Data

14.3.1 Method Blank (Reagent Blank) (MB/RB) – Individual samples that do not meet the acceptance criteria must be reanalyzed. If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

14.3.2 Duplicate (DUP) – The duplicate sample is typically a duplicate count of the Laboratory Control Sample (LCS) DUP analysis that fails the replicate test must be reanalyzed to determine if analytical failure or sample heterogeneity was the cause of the problem.

14.3.3 Matrix Spike Recovery (MS) – MS samples are not typically analyzed for gamma spectral content due to inability of the lab to homogenize spike material with sample media. If performed, MS recoveries that do not meet the acceptance criteria must have that sample reanalyzed. The batch may be reportable if the acceptance criteria for the LCS is met. If a Matrix Spike Duplicate is also analyzed and the recovery is comparable to the MS, the results are reported and noted in the final report. Calculations of MS activity include source decay correction to account for decay between the spike solution source certificate reference date/time and the MS sample collection date/time.

14.3.3.1 The analyst must evaluate the MS results to attempt to determine the cause of the failure and the appropriate action to take based on that evaluation. All decisions made must be documented.

14.3.4 Matrix Spike Duplicate (MSD) - If an MSD is analyzed and the recovery is comparable to the MS, the results are reported with qualification in the final report.

14.3.5 Laboratory Control Sample (LCS) – The LCS used for gamma-spectral analysis is a static source of relative consistent content of project samples, matching the geometric configuration of prepared project samples. If an LCS analysis does not meet the acceptance criteria, the entire analytical batch must be re-prepped and reanalyzed. Calculations of LCS activity include source decay correction to account for decay between the LCS spike solution certificate reference date/time and the LCS sample analysis date/time.

14.3.5.1 The results of the batch may be reported, with qualification in the final report, if the LCS recoveries are high and the

sample results within the batch are less than the reporting limit.

14.3.6 Laboratory Control Sample Duplicate (LCSD) – The LCSD is typically a replicate count of the static LCS source described in section 14.3.5. If a LCSD does not meet the recovery acceptance criteria, the entire analytical batch must be reanalyzed.

14.3.6.1 The results of the batch may be reported, with qualification, if the LCS recoveries are high and the sample results within the batch are less than the reporting limit, and duplicate precision meets the acceptance criteria.

14.3.7 If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

15. Method Performance

15.1 Laboratory control samples (LCS) are analyzed with each batch.

15.2 Each analyst must read and understand this procedure with written documentation maintained electronically.

15.3 An initial demonstration of capability (IDOC) study must be performed. A record of the IDOC will be maintained for each analysts.

15.4 On an annual basis, each analyst will complete a continuing demonstration of capability (CDOC).

16. Pollution Prevention and Waste Management

16.1 Place radioactive waste into appropriate receptacles.

16.2 Discard acidified samples and unusable standards into proper waste drains.

16.3 Dispose of waste materials in accordance to type: Non-hazardous, hazardous, non-radioactive, radioactive or mixed.

17. References

17.1 ASTM E181-93, Standard Test Methods for Detector Calibration and Analysis of Radionuclides, ASTM Standards, Vol. 12.02.

17.2 User's Manual, Model 480726 Genie-ESP System, September, 2000.

17.3 Advanced Concept's Manual, Model 480198 Genie-VMS Spectroscopy System, September 2000.

17.4 Command Descriptions Manual, Model 480198 Genie-V<MS Spectroscopy System, September, 2000.

17.5 Crystal Reports, Version 8.5, Seagate, 2001.

17.6 User's Manual, Model 480206 Genie-VMS Quality Assurance Software, September, 2000.

17.7 User's manual, Model 480720 PROcount-ESP, September, 2000.

17.8 User's Manual Maestro-32 MCA Emulator, Ortec, Version 6.05, 2002.

17.9 Programmer's Guide, Model 480198 Genie-VMS Spectroscopy System, September, 2000.

17.10 Nuclide Identification Algorithms and Software Verification and Validation Manual, 07-0464-02, Canberra Industries, November 1993.

17.11 Peak Search Program Algorithm Manual, 07-0064, Canberra Industries, March 1985.

17.12 Spectroscopy Applications Algorithms and Software Verification and Validation Manual, 07-0368, September 1991.

17.13 Table of Radioactive Isotopes, Brown and Firestone, Shirley editor, John Wiley & Sons, 1986.

17.14 Krieger, H. L. and Whittaker, E. L., Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, "Gamma Emitting Radionuclides in Drinking Water," Method 901.1, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH, August, 1980.

17.15 "Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)", July 2004, Final.

17.16 Department of Defense Quality System Manual for Environmental Laboratories (DoD QSM), current version.

17.17 *EML Procedures Manual*, HASL-300, 27th Edition, Volume 1, 1990, Method 4.5.2.3 – Gamma.

17.18 Pace SOP ENV-SOP-GBUR-0044 (Laboratory Equipment), current revision.

17.19 Pace SOP ENV-SOP-GBUR-0079 (Gamma Spec Prep), current revision.

17.20 Pace SOP ENV-SOP-GBUR-0080 (Neutron Dosimetry), current revision.

17.21 Pace SOP ENV-SOP-GBUR-0081 (Cs-137 Dosimeter), current revision.

17.22 Pace SOP ENV-SOP-GBUR-0082 (I-129), current revision.

17.23 Pace SOP ENV-SOP-GBUR-0008, current revision (Deionized Water Quality and Suitability).

17.24 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (most recently approved revision).

17.25 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.

17.26 Pace Analytical Services, LLC. - Pittsburgh Laboratory Quality Assurance Manual, current version.

18. Tables, Diagrams, Flowcharts, Appendices, etc.

18.1 See the operations manual supplied by the manufacturer for diagrams and tables associated with the operation or maintenance of gamma spectroscopy detectors.

19. Method Modifications

19.1 Not Applicable.

20. 20. Revisions

Document Number	Reason for Change	Date
PGH-R-023-5	1. Table of contents added. 2. Section 1 modified to specify location of referenced documents. 3. Section 2 modified to specify hardware and software as required by DOD QSM. 4. Section 3 modified to incorporate "resolution" term in conjunction with FWHM calibration. Long Background count time corrected to minimum of 1000 minutes. Long Background frequency defined to reflect specific requirements. 5. Section 4.2 modified to clarify secondary containment of aqueous count geometries for prevention of cross-contamination. 6. Section 6 Revised to discuss the location of definitions for terms related to the equipment hardware and system software. 7. Section 8 modified to remove extraneous analysis sample analysis protocols. Specification that geometries containing solid sample should be sealed. 8. Section 9 modified to describe computer requirements for the two analysis platforms used currently 9. Section 11 modified to cite requirement for daily performance check assessment by analyst. Also, order of calibrations is documented to require energy calibration prior to geometry-specific efficiency calibrations. 10. Section 15, 19 and 20 added. 11. Section 15 renamed as Method Performance. Method performance requirements added. 12. Section 17: Added Method 901.1 reference 13. Section 19 renamed as Method Modifications	18May2012
PGH-R-023-6	1. Annual Review and Update (2013). 2. Added specifications for DI water as ASTM Type II DI water and included reference to SOP PGH-C-027, the SOP that documents the DI water production and testing process. 3. Section 11.1.2 Specified the requirement of a minimum five minute daily background check for gamma instruments. 4. Section 11.2.1 specified that extended background counts used for sample analyses are performed monthly at a minimum but specified that backgrounds are typically run weekly. 5. Section 11.3.3.3 added Ortec GammaVision software to the list of software used for calibration calculations. 6. Section 17.19 Added reference to *EML Procedures Manual*, HASL-300, 27th Edition, Volume 1, 1990, Method 4.5.2.3 – Gamma.	25Jun2013
PGH-R-023-7	1. Annual SOP Review (2014) 2. Section 2.2 and 2.3 - Included reference to Pace SOPs and methods for analyses performed by gamma counting. 3. Section 11 – Updated for verification procedure and acceptance criteria. 4. Section 11 – Updated to include aqueous and Drinking	13Jul2014

Document Number	Reason for Change	Date
	Water calibration and other geometry calibration verification requirements. 5. Document updated to include reference to all applicable Pace SOP and method references for analyses where a gamma detector/system is utilized. 6. Section 17 – Updated to include Pace SOP and method references related to gamma spectroscopy counting. 7. Reformatted document.	
PGH-R-023-8	1. Section 11 (11.1.2) updated to include process for amplifier gain adjustments for Ortec gamma detectors.	20Feb2015
PGH-R-023-9	1. Corrected formatting in Section 8. 2. Section 9.1, defined Gamma Detector Types. 3. Section 9.2 and 9.3, added suggestion to consult the respective instrument manuals for issues not covered by this SOP. 4. Section 11.1.5, revised SOP to indicate that hard-copy performance check data are being stored electronically. Printouts are no longer stored. 5. Section 11.2.5, added procedure for performing long background counts on the Ortec system. 6. Section 13.3, added clarifying information regarding the LCS and duplicate counts.	03Dec2015
PGH-R-023-10	1. Section 2.8 – Added for decay correction of results per nuclide based on the nuclide library. 2. Section 8.4 – Hold time for gamma nuclides and I-131. 3. Section 11.3.3 and 12.1.11– Manual integration feature disabled and manual integration of peaks is prohibited. 4. Section 12.- Re-labeled as section 13 Calculations 5. Section 14.3.3 and 14.3.5 – Updated to discuss decay correction of spiking solutions. 6. Sections 13 through 19, re-labeled section 14 through 20.	21Dec2016
PGH-R-023-11	1. 1.Section 12.1.5 added to document the peak analysis process utilized for nuclear dosimetry samples.	13Feb2017
S-PGH-R-023-rev.12	1) Sections 11.3.4.4 and 11.3.4.5 revised to specify calibration verification requirements for Drinking Waters following the guidance from the Manual for the Certification of Drinking Water Laboratories. Calibration verifications for all other matrices revised to follow guidance from the current TNI standard. 2) Figure 4 updated to be correct figure. The previous figure was a duplicate of Figure 3. 3) Section 14.3.6 updated to correct reference to section 14.3.5, not 13.3.5.	02Feb2018
ENV-SOP-GBUR-0078 Rev 01	1. Section 11.1.5 modified to require a daily efficiency check tolerance of 3% as required by the DOD QSM. 2. Periodic review as required for compliance. 3. Sections 2,10, 11, and 17 modified to update current SOP IDs. 4. Updated section 14.3.3.1. 5. Updated section 15, removed references to LMS.	22Feb2019

ATTACHMENT C-34

ANALYSIS OF SAMPLES FOR ALPHA EMITTING ACTINIDES AND PU-241
PACE PITTSBURGH

Document Information

Document Number: ENV-SOP-GBUR-0068 **Revision:** 00

Document Title: Analysis of Samples for Alpha Emitting Actinides and Pu-241

Department(s): Rad Chem

Previous Document Number: S-PGH-R-008-rev.13

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Effective Date: 08 Feb 2018

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Review

Name/Signature	Title	Date	Meaning/Reason
Nasreen Derubeis (009976)	Quality Manager II	20 Feb 2019, 01:49:46 PM	Reviewed
Jaclyn O'Donnell (003227)	Qa Analyst I	21 Feb 2019, 07:23:35 AM	Reviewed
Raelyn Sylvester (005634)	Qa Analyst I	21 Feb 2019, 08:46:43 AM	Reviewed

Pace Analytical™

STANDARD OPERATING PROCEDURE

Analysis of Samples for Alpha Emitting Actinides and Plutonium-241

Methods: ASTM Method D-3972-90 and HASL 300 Method U-02

SOP NUMBER:	S-PGH-R-008-rev.13
REVIEW:	R. Kinney
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	PGH-R-008-12
REVIEW DATE:	Upon Procedural Change

APPROVALS

Department Manager/Supervisor — 02/08/18 Date

Senior Quality Manager — 02/08/18 Date

PERIODIC REVIEW
SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

This is **COPY#**_____ distributed on _____ by _____and is ___**CONTROLLED or** __**UNCONTROLLED.**

TABLE OF CONTENTS

1. Purpose

1.1 This SOP describes the procedure to be used for the determination of micro-quantities of Americium, Curium, Thorium, Plutonium (including Pu-241), Neptunium and Uranium in various sample matrices. It also addresses routinely difficult matrices such as filters, solids in quantities over two grams, and aqueous samples where precipitation as described in the procedure may not be successful.

1.2 Samples not meeting the criteria outlined in this procedure should be brought to the attention of the Department Manager/Supervisor or designee for further direction.

1.3 This procedure is applicable for determining compliance for isotopic uranium in drinking water and is substantially compliant with ASTM Method D-3972-90 and HASL 300 Method U-02. Deviations from these methods are addressed in Section 19 of this procedure.

1.4 This procedure is designed for the determination of the following radio-nuclides:

Isotope	Half-life	Alpha Energy MeV (Abnd.)
Americium 241	*458 yrs*	*5.49 (86%) 5.44 (13%)*
Americium 243	*7370 yrs*	*5.28 (88%) 5.23 (11%)*
Curium 242	*162.79 days*	*6.069 (25%) 6.112 (74%)*
Curium 244	*18 yrs*	*5.80 (76%) 5.76 (23%)*
Plutonium 238	*87.8 yrs*	*5.50 (72%) 5.46 (28%)*
Plutonium 239	*24131 yrs*	*5.16 (73%) 5.14 (15%)*
Plutonium 240	*6569 yrs*	*5.17 (74%) 5.12 (26%)*
Plutonium 241	*14.35 yrs*	*Beta 20.81Kev (5.23 Ave)*
Plutonium 242	*375850 yrs*	*4.90 (78%) 4.86 (22%)*
Neptunium 237	*2140000 yrs*	*4.79 (47%) 4.77 (25%)*
Thorium 228	*1.9132 yrs*	*5.42 (73%) 5.34 (27%)*
Thorium 230	*77000 yrs*	*4.69 (76%) 4.62 (23%)*
Thorium 232	*14050000000 yrs*	*4.01 (77%) 3.95 (23%)*
Thorium 229	*7340 yrs*	*4.85 (56%) 4.90 (10%)*
Thorium 227	*18.718 days*	*6.03 (24%), 5.98 (23%), 5.76 (20%)*
Uranium 232	*72 yrs*	*5.32 (69%) 5.26 (31%)*
Uranium 238	*4468000000 yrs*	*4.19 (77%) 4.15 (23%)*
Uranium 235	*703800000 yrs*	*4.39 (55%) 4.36 (11%)*
Uranium 236	*23415000 yrs*	*4.49 (74%) 4.45 (26%)*
Uranium 234	*244500 yrs*	*4.78 (72%) 4.72 (27%)*
Uranium 233	*159200 yrs*	*4.82 (84%) 4.78 (13%)*

2. Scope and Application

2.1 This procedure covers the measurement of various isotopic alpha emitters in various matrices including drinking water. Since all plutonium isotopes behave the same, this procedure also addresses the isolation of beta emitting Pu-241. With the exception of drinking water sources, most other matrices, especially soils, contain elements, which may complex some actinides, making it necessary to aggressively treat such samples.

2.2 This method is a laboratory promulgated method based on multiple accredited methods and the behavior of individual alpha emitters.

2.3 Sample results are decay corrected to the client supplied collection date and time for all analytes reported using this SOP.

2.4 Alpha count sources generated as the product of this SOP are analyzed as documented in the current revision of Pace SOP PGH-R-020, "Alpha Spectroscopy Instrument Operations."

2.5 Plutonium-241 count sources generated as the product of this SOP are analyzed as documented in the current revision of Pace SOP PGH-R-022, "Liquid Scintillation Counter Operations."

2.6 Beta-emitting tracer sources produced from application of this SOP are analyzed for yield determination as documented in the current revision of Pace SOP PGH-R-002, "Gas Flow Proportional Counter Operation."

3. Summary of Method

3.1 The nuclides listed in Section 1 are first separated from the interfering substances by iron hydroxide precipitation, and appropriately dissolved in either a hydrochloric acid or nitric acid solution. Uranium and Plutonium separation can be accomplished using an anion exchange column. Subsequent separation and purification of Americium and Curium are accomplished using Tru-Resin™ columns. For Thorium and Neptunium, separation can be accomplished using anion exchange columns. Additionally, Americium/Curium spectral resolution can be improved by using TEVA-Resin™ columns.

3.2 Following separation, the individual isotopes are micro-precipitated onto a filter and the corresponding alpha activity is determined using an alpha spectrometer.

3.3 Counting efficiency is determined by micro-precipitating a solution composed of three different energy range alpha standards (such as Cm-244, Pu-239, and Th-230) and counting the sources in each detector of an alpha spectrometry system or using a commercially prepared source with at least 3 different energy range nuclides.

4. Interferences

4.1 Any nuclide with an alpha emission similar in energy to the isotopes in question that cannot be separated from the target nuclide will interfere with this analysis.

4.1.1 It is not always necessary to re-prep samples where both sufficient tracer counts and an interfering nuclide are present.

4.1.2 A clean-up may be performed on the micro-precipitated source to remove the interfering nuclides.

4.1.3 Analysts should consult with the Department Manager/Supervisor or the specified designee on how to proceed.

4.2 Samples that contain isotopes employed as working tracers will lead to an overestimate of yield and low bias in measured results. If the sample activity is known, or can be determined, and the tracer activity corrected to reflect the true concentration of the tracer nuclide present in the sample, accurate results can be obtained.

4.3 Daughters of Th-229 will interfere with Cm-244 analysis. Thorium and americium should be run separately in order to exclude such interferences.

4.3.1 If sample volume limits running these analyses separately, Method 4 (Purification of Americium Using Teva-Resin™) must be performed, subsequent to Method 3 (Purification of Americium Using Tru-Resin™), to remove any thorium decay daughters from the americium/curium fraction.

4.4 Unless isolated separately, Neptunium-237 will interfere with the Pu-242 yield calculation, therefore Pu-236 should be used as a tracer when it is desirable to extract and count plutonium with neptunium on the same counting source.

4.5 Plutonium-236 decays to U-232, which in turn decays to Th-228, therefore it is important to know when the last U-232 separation was performed on the Pu-236 working tracer.

4.5.1 Analysts should be mindful of the potential for a bias in the uranium tracer recovery depending on the length of time since the separation of U-232 from the working tracer.

4.5.2 Plutonium and thorium can be run sequentially if Th-228 is not desired or thorium decay daughters were previously separated from the Pu-236 working tracer.

4.6 Excess fluoride during sample digestions may produce insoluble actinide complexes leading to low yields. Addition of saturated boric acid solution must be performed at the completion of sample digestion to avoid this.

4.7 Silica in solution may cause problems with uranium and thorium separations and analyte retention on columns.

4.8 Excess carbonate in solution will prevent the iron hydroxide precipitation of the uranium isotopes. To prevent this, it is extremely important to thoroughly boil water samples during the initial precipitation steps of uranium analysis.

4.9 The analysis volume for samples known to contain elevated concentrations of uranium should be controlled so as to limit the quantity of uranium carried through to the final source preparation. Excessive mass

contained in the final micro-precipitated count source may negatively affect peak resolution (FWHM) for which there are control limits required by this SOP.

5. Safety

5.1 Procedures must be carried out in a manner that protects the health and safety of all personnel. Since this analysis is for a radioactive constituent, the sample must be treated as radioactive.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately.

5.3 When mixing or diluting acids, always add the acid slowly to water and swirl. Dilution of acids must always be done in a hood. Appropriate eye-protection, gloves, and a lab coat must be worn.

5.4 Exposure to radioactivity and chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous and/or non-radioactive, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 In order to minimize the potential for cross contamination of high and low levels of radioactive samples, good housekeeping and good laboratory practices are essential and must be strictly adhered to.

5.6 Organic samples of unknown content must be handled with extreme caution and under the direct instruction of the Department Manager or Manager-specified designee. Direct treatment of organic matrices with strong oxidizing chemicals such as nitric acid and/or hydrogen peroxide is strictly prohibited.

5.7 Hydrofluoric acid is particularly hazardous because a serious skin exposure may cause no immediate sensation of pain. The acid penetrates the skin and spreads internally, causing tissue damage deep under the skin. The resulting burn is painful, difficult to treat, and easily infected. Gloves must be checked for pinhole leaks before use. They must be rinsed before they are removed and must be discarded after use. HF burn gel shall be put on suspected HF burns after flushing (except the eyes) until medical help can be obtained. Medical attention shall be sought even if suspicions arise after working hours. Contact your group leader immediately for further information if a HF burn is suspected.

5.8 In addition, HF vapors are also hazardous. Exposure can cause permanent damage. Breathing HF vapors even for a short time and at a low temperature can be injurious to the respiratory system and even fatal. All such direct contact must be avoided.

5.9 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety

information can be obtained from the MSDS files maintained in the laboratory.

6. Definitions

6.1 Batch: For all analysis, an analytical batch contains 20 or fewer samples of a similar matrix, prepared at the same time, by the same analyst, using the same reagents.

6.2 SRM: Standard Reference Material.

6.3 Throughout this procedure, approximate weights and measures will be designated by the use of whole numbers when referring to masses exceeding 1g or volumes in excess of 1mL. Measurements of masses and volumes so designated can be made with top loading balances, graduated cylinders, etc. For approximate measures below 1g or 1mL, the word "approximately" must be used prior to the described mass or volume.

6.4 Throughout this procedure, exact or critical masses and volumes will be designated by the use of one or more decimal places. Measurements of weights and volumes so designated should be made with accurate analytical instruments such as analytical balances, calibrated pipettes, etc.

6.5 When aliquotting samples on a balance, the observed mass on the balance must be recorded in preparation logbooks to the lowest weight indicated on the balance. Sample aliquot masses must not be targeted. Once sample is removed from the sample container and transferred to a beaker, it must not be removed from the beaker.

6.6 The method utilized for obtaining the sample aliquot, whether on a balance, in a graduated cylinder, or by pipette, must be clearly annotated in the preparation logbook.

7. Responsibilities and Distribution

7.1 General Manager/Assistant General Manager (GM/AGM)

7.1.1 The GM/AGM has the overall responsibility for ensuring that SOPs are prepared and implemented for all activities appropriate to the laboratory involving the collection and reporting of analytical data.

7.1.2 The GM/AGM and Senior Quality Manager/Quality Manager have final review and approval authority for all SOPs prepared within the laboratory.

7.2 Senior Quality Manager/Quality Manager (SQM/QM)

7.2.1 The SQM/QM will maintain a master file of all SOPs applicable to the operations departments.

7.2.2 The SQM/QM will assign a unique number to each SOP prepared prior to approval and distribution.

7.2.3 The SQM/QM will distribute SOPs to applicable personnel and maintain an accurate accounting of such distribution to ensure that the SOPs, in the hands of the users, are current and complete.

7.3 Department Manager/Supervisor

7.3.1 The Department Manager/Supervisor is responsible for ensuring all staff members read, follow, and are adequately trained in the use of the SOPs

7.3.2 The Department Manager/Supervisor coordinates the preparation and revision of all SOPs within the department whenever a procedure changes.

7.3.3 The Department Manager/Supervisor provides initial approval of all SOPs within the department.

7.3.4 The Department Manager/Supervisor makes recommendations for SOP revision to the SQM/QM via written memo.

7.4 Individual Staff

7.4.1 Individual staff members are responsible for adherence to the specific policies and procedures contained in the applicable SOPs.

7.4.2 Individual staff members will only use a signed, controlled copy of the SOP. Each person may make recommendations to the Department Manager/Supervisor for revising SOPs as the need arises.

7.4.3 Personnel are responsible for ensuring that any deviations from this SOP are reported to the Department Manager/Supervisor.

8. Sample Collection, Preservation, and Handling

8.1 Aqueous Samples

8.1.1 Containers used for sample collection must never be re-used. Either plastic or glass containers may be used for sample collection.

8.1.1.1 Aqueous samples must be preserved at the time of collection by adding enough concentrated (16N) HNO_3 to the sample to make the sample pH <2. Typically, 2mL of 16N HNO_3 per liter of sample is sufficient to obtain the desired pH. Samples must be preserved within five days of collection. If samples are collected without preservation, they must be received by the laboratory and preserved within five days of collection. Following preservation with acid, samples must be held in the original container for a minimum of 24 hours before analysis or transfer of sample. For samples preserved at the time of receipt, the pH must be re-checked by laboratory personnel prior to removing sample for analysis. The pH re-check date and time, the initials of the analyst verifying the pH, as well as any adjustments or notes regarding the preservation must be recorded in the pH Verification Logbook.

8.1.1.2 For dissolved analysis, samples must be filtered through a 0.45μm membrane filter and then preserved to a pH <2.

8.1.1.3 For total analysis, the sample is not filtered, but is preserved to a pH<2.

8.1.2 Refrigeration is not required for aqueous or solid samples, but is recommended for all biological samples.

8.1.4 The maximum hold time for samples analyzed by this procedure is 180 days between sample collection and sample analysis.

9. Equipment and Supplies

9.1 Multi-channel Analyzer:

9.2 Alpha Spectrometer: Refer to SOP PGH-R-020, current revision "Alpha Spectroscopy Instrument Operation" for instructions on alpha spectroscopy system operation.

9.3 Electric hot plate or griddle.

9.4 Ion exchange columns, 2mL, disposable from Eichrom or equivalent.

9.5 TRU and TEVA cartridges from Eichrom.

9.6 Vacuum box apparatus and applicable parts, disposable yellow and white tips, 10-50mL syringe barrels, etc from Eichrom or equivalent.

9.7 Polypropylene filters, 25mm, 0.1μm pore size from Environmental Express, or equivalent.

9.8 PTFE FEP Beakers, 100mL size and other assorted sizes, or equivalent.

9.9 Multi-port vacuum filtering apparatus for 25mm filters (referred to as the filter rig).

9.10 Miscellaneous glassware: beakers, watch glass covers, and stir rods.

9.11 Membrane Filters, 5.5cm diameter, 0.45μm pore size.

9.12 Analytical Balance: Sensitivity to 0.1mg, capacity 0 - 160g.

9.13 Top loader balance, sensitivity to 0.1g, capacity 0-2000g.

9.14 Vacuum filtration apparatus for 5.5cm diameter membrane filters.

9.15 General-purpose centrifuge and disposable centrifuge tubes, 50mL made of high-density polyethylene or equivalent.

9.16 Vortex mixer.

9.17 Liquid scintillation vials, glass.

9.18 Muffle oven capable of 105ºC to 550ºC, with or without ramping capabilities.

9.19 Software supplied with the instrument to control instrument operation. Refer to SOP PGH-R-020, current revision "Alpha Spectroscopy Instrument Operation" for applicable software details.

9.20 Computer capable of running the Alpha spectrometer Counter System software, monitor, mouse, keyboard, and printer. Refer to SOP PGH-R-020, current revision "Alpha Spectroscopy Instrument Operation" for computer hardware specifications.

10. Reagents and Standards

10.1 Reagents must be prepared from reagent grade chemicals, unless specified otherwise. Distilled or deionized (DI) water. ASTM Type II as produced using the specifications documented in SOP PGH-C-027, current revision. Consult the Safety Data Sheets for the properties of these reagents, and how to work with them.

10.2 Anion exchange resin, Bio-Rad AG 1x8 (100 - 200 mesh, Cl^- form) or equivalent. Slurry the resin in a squirt bottle with ASTM Type II DI water.

10.3 Ammonium Hydroxide, 15N, concentrated, sp. gr. 0.90, 56.6%.

10.4 Ammonium Thiocyanate, 6M: Dissolve 476g ammonium thiocyanate in 300mL of ASTM Type II DI water and dilute to 1.0L with ASTM Type II DI water.

10.4.1 CAUTION! The reaction is extremely exothermic and the bottle may become very slippery.

10.5 Ammonium Thiocyanate, 3M/ 0.1N Formic acid: Dilute 250mL 6M ammonium thiocyanate and 50mL 1.0N formic acid to 500mL with ASTM Type II DI water. Prepare fresh daily.

10.6 Ammonium Thiocyanate, 1M/ 0.1N Formic acid: Dilute 100mL 6M ammonium thiocyanate and 60mL 1.0N formic acid to 600mL with ASTM Type II DI water. Prepare fresh daily.

10.7 Ascorbic Acid, 1.0M: Dissolve 17.6g ascorbic acid into 50mL ASTM Type II DI water and dilute to 100mL with ASTM Type II DI water. Prepare weekly.

10.8 Boric Acid, 5% Saturated Solution: Add 50g granulated boric acid to 500mL ASTM Type II DI water and dilute to 1L with ASTM Type II DI water. The boric acid should not completely dissolve.

10.9 Distilled or deionized DI water. Resistance value between 0.5 and 2.0 Mmhos (2.0 to 0.5µohms/cm specific conductivity) at 25˚C.

10.10 Deionized Water adjusted to a pH of 10.0 by the addition of 6-8 drops of ammonium hydroxide to each 500mL of DI.

10.10.1 The pH should be checked prior to each addition of ammonium hydroxide, since it is not always necessary to add the full 6-8 drops each time pH 10 DI is made up.

10.11 Ethanol, 80%. 800mL of reagent grade alcohol mixed with 200mL of ASTM Type II DI water.

10.12 Formic acid, concentrated, 88%.

10.13 Formic acid, 1.0N: Dilute 42.5mL concentrated formic acid to 1.0L with ASTM Type II DI water.

10.14 Hydrochloric acid, 12N, concentrated, sp. gr. 1.19, 37%.

10.15 Hydrochloric acid, 9N / 0.10% Hydrogen Peroxide: For every 100mL of 9N HCl, add 0.1mL of 30% H_2O_2. Shake well and allow to sit for 5 minutes prior to use. Prepare daily.

10.16 Hydrochloric acid, 9N: Dilute 750mL concentrated 12N HCl to 1L with ASTM Type II DI water.

10.17 Hydrochloric acid, 6N: Dilute 500mL concentrated 12N HCl to 1L with ASTM Type II DI water.

10.18 Hydrochloric acid, 4N: Dilute 332mL concentrated 12N HCl to 1L with ASTM Type II DI water.

10.19 Hydrochloric acid, 0.1N: Dilute 8.3mL concentrated 12N HCl to 1L with ASTM Type II DI water.

10.20 Hydrochloric acid, 6N / 0.52N Hydrofluoric acid Solution: Dilute 500mL of concentrated 12N HCl and 18mL of concentrated HF to 1L with ASTM Type II DI water.

10.21 Hydrochloric acid, 9N / 0.05M Ammonium Iodide Solution: For every 100mL of 9N HCl, add 0.724g of NH_4I solid. Shake well and let stand for 5 minutes prior to use. Prepare daily.

10.22 Hydrazine Dihydrochloride, 25% Solution: 25g hydrazine dihydrochloride dissolved in 75mL of ASTM Type II DI water.

10.23 Hydrogen Peroxide (30%) H_2O_2.

10.24 Hydrofluoric acid, 29N, concentrated, sp. gr. 1.18, 49%. Must be stored in a plastic container.

10.25 0.1% Hydrofluoric acid: Dilute 1.0mL concentrated HF to 1L with DI.

10.26 Iron Carrier: 10.0mg Fe/ml: Dissolve 72.4 g $[Fe(NO_3)_3]\bullet 9\ H_2O$ in 500mL of ASTM Type II DI water and dilute to 1L with ASTM Type II DI water.

10.27 Neodymium carrier (0.5mg/mL): Dilute 5.0mL of 10mg/mL of neodymium carrier to 100mL of ASTM Type II DI water.

10.28 Nitric acid, 16N, concentrated, sp. gr. 1.42, 70%.

10.29 Nitric acid, 2N: Dilute 125mL of conc. 16N HNO_3 to 1L with DI water.

10.30 Nitric acid, 8N: Dilute 500mL of conc. 16N HNO_3 to 1L with DI water.

10.31 Nitric acid, 2N / 0.5M $Al(NO_3)_3$: Dissolve 188g aluminum nitrate nonahydrate in 500mL of ASTM Type II DI water. Add 125mL conc. 16N HNO_3 to the solution and then dilute to 1L with ASTM Type II DI water.

10.32 Nitromethane, ACS reagent.

10.33 Potassium Thiocyanate Solution, 0.1N: purchased ready made from Fisher Scientific, or equivalent.

10.34 Tru-Resin™: Eichrom Inc., Mix 100g (s-grade) of Tru-Resin with 428mL ASTM Type II DI water and 2mL of conc. 16N HNO_3.

10.35 TEVA-Resin™: Eichrom Inc., Mix 100g (s-grade) of TEVA-Resin with 428mL of ASTM Type II DI water and 2mL conc. 16N HNO_3.

10.36 Titanium (III) Chloride: 10% wt. Solution.

10.37 Standards

10.37.1 Tracer: A solution containing Am-243, Np-239 (prepared from Am-243), Pu-242, Pu-236, Th-229, Th-234 (prepared from U-238), or U-232 prepared from a NIST-traceable and certified solution, or equivalent. Tracer spike aliquots shall have DPM values consistent with the activity of the samples. Check the certificate for the expiration date.

10.37.2 Laboratory Control Sample (LCS)

10.37.2.1 Liquid: A solution of Am-241, Cm-244, Pu-239, Pu-241, Np-237, Th-230, or U-238 prepared from a NIST-traceable and certified source, or equivalent. Check the calibration certificate for the expiration date.

10.37.2.2 Solid: A soil containing Am-241, Cm-244, Pu-239, Pu-241, Np-237, Th-230, or U-238 prepared from a NIST-traceable and certified source, or equivalent. Check the certificate for the expiration date.

Note: Equivalent is defined as being traceable to any international source that provides a certificate of calibration. As the project requires, a solution may be used where the isotopic activity has been confirmed by multiple laboratory analyses.

11. Calibration

11.1 Plated sources can be purchased from a NIST supplier, but they must have the same size effective area and they must be positionable so the effective area is the same distance from the detector as those sources prepared for sample counting.

11.2 If plated sources cannot be purchased, sources can be prepared using NIST traceable standards as follows.

11.3 Sources for alpha spectroscopy system calibration must be prepared in a fashion that will ensure >99% recovery of each calibration source analyte.

11.4 Concentrated standards or SRMs that limit the volume of the standard to less than 0.5mL per radioisotope must be used in order to ensure that the acid concentration is kept as dilute as practical. Quantitative precipitation of many actinides from acid solutions with concentrations greater than 0.5N is impossible and will lead to erroneous results.

11.5 Transfer an appropriate volume or mass of analytical standard to a labeled, disposable centrifuge tube. Record the standard numbers utilized and mass or volume information in the appropriate standard dilution logbook. Dilute the combination of standards to 20mL with 0.1N HCl.

11.6 Add the following to the solution in the centrifuge tube:

11.6.1 0.1mL of neodymnium carrier solution (0.5mg/mL)

11.6.2 Four drops of the iron carrier solution.

11.6.3 Seven drops of the 1M ascorbic acid solution

11.6.3.1 This reduces the iron to the +2 state.

11.6.3.2 Proper reduction of iron will result in a clear, colorless solution.

11.7 Cap each tube and swirl the solution to mix the contents. Place tubes in a warm water bath to ensure proper equilibration between the carrier and the radioisotopes.

11.8 After five minutes, add 3mL concentrated HF acid to each source and swirl the source to mix the contents. Let the sources sit in the warm water bath for 15 minutes, then remove them, and allow them to cool for 15 minutes.

11.9 Prepare a filter apparatus by placing a filter funnel into an HDPE vacuum flask. Turn on the vacuum and rinse the apparatus with several mLs of ASTM Type II DI water.

11.10 Carefully wash and rinse a filter funnel that is dedicated to, and set aside for, the preparation of calibration sources.

11.11 Place a polypropylene filter onto the filtering apparatus and ensure it is centered.

11.12 Place the clean filter funnel on the rig and secure it, being careful not to rip the filter in the process.

11.13 Rinse the inside surface of each funnel with the 80% ethanol solution then rinse the inside surface of each funnel with approximately 10mL of ASTM Type II DI water.

11.14 Transfer the calibration solution to the filter funnel and allow it to pass through the filter. After all of the calibration solution has passed through the filter, rinse the centrifuge tube with approximately 5mL of the 80% ethanol solution and transfer it to the filter funnel as a rinse.

11.15 Rinse the inside of the funnel with an additional 2-3mL of the 80% ethanol solution.

11.16 Label the metal backside of a one inch counting disk with the appropriate standard number as determined from the standard preparation logbook, and remove the paper backing from the pre-taped side of the disk.

11.17 Remove the filter funnel and carefully remove the calibration filter from the filtration rig and affix it to the disk.

11.18 Allow the disk to air dry for a minimum of 15 minutes.

11.19 When the calibration source has dried, place it in a labeled petri dish and submit it to the count room for counting.

11.20 Perform calibration of alpha spectrometry detectors as specified in the current version of Pace SOP PGH-R-020, “Alpha Spectroscopy Instrument Operations”. Count sources long enough to obtain a minimum of 10000 net counts in each of the three nuclides regions of interest.

11.21 Transfer the contents of the vacuum flask to a 600mL PTFE beaker.

11.21.1 Rinse the flask three times with 50mL of ASTM Type II DI water and add the rinses to the beaker.

11.22 Evaporate the solution to dryness on a hotplate on a low to medium heat setting.

11.23 Carefully add 10mL of concentrated nitric acid and 1mL of 30% hydrogen peroxide solution to the beaker and evaporate the solution to dryness.

11.24 Carefully add 10mL of concentrated nitric acid and 1mL of 5% boric acid solution to the beaker and evaporate the solution to dryness.

11.25 Dissolve the residue into 20mL of 8N nitric acid solution by heating the solution.

11.26 Allow the solution to cool, then transfer it to a 100mL volumetric flask.

11.26.1 Rinse the beaker three times with 10mL of ASTM Type II DI water and add the rinses to the volumetric flask.

11.26.2 Dilute the solution to the mark with ASTM Type II DI water.

11.26.3 Transfer the diluted calibration effluent to a labeled HDPE bottle.

11.27 Analyze 10mL of each calibration effluent sample for gross alpha content using the most recent revision of SOP PGH-R-001.

11.27.1 Calculate the total alpha content for the entire source effluent.

11.27.2 Calculate the total alpha activity of each calibration source.

11.27.3 Compare the total effluent activity to the theoretical activity to ensure that the effluent contains less than 1% of the total activity. Calibration sources must contain >99% of each analyte in order to be accepted for calibration purposes.

11.27.4 Consult with the Department Manager/Supervisor or specified designee if the sources are not acceptable.

11.28 The Plutonium-241 calibration is performed separately on a liquid scintillation counter. Add 1000 to 2000 dpm of Pu-241 by mass to ten, labeled, glass scintillation vials. Add 1.0mL of concentrated HCl to each vial and heat the sources to dryness on a hotplate.

11.29 Add 2.0mL of 0.1N HCl to each vial to dissolve the residue. Then add 15mL of Ultima Gold AB liquid scintillation cocktail to each vial. Cap each vial and shake it vigorously to mix the contents.

11.30 Establish variations in quench by carefully opening the vials and adding nitro methane to each vial in increasing increments. Nitro methane is not added to one of the vials and not to the background vial. For example, add 10 µL to one vial, 20 µL to another, 40 µL, 70 µL, 100 µL and so on, documenting the amount of nitro methane added to each vial for future reference.

11.31 Count calibration sources according to the Pace SOP, PGH-R-022, “Liquid Scintillation Counter Operations”. Count the samples long enough to obtain 10,000 net counts in the Pu-241 window. Count times will vary from source to source based on the quench factor. Adjust the amount of nitro

methane in each vial and recount the set as necessary to achieve the most representative range of values for the quench curve.

11.32 Calculate the Pu-241 efficiency based on the quench values using Microsoft Excel or equivalent curve plotting software.

11.33 Calibrations must be verified initially prior to counting samples and annually thereafter by counting a separate source in the detector and processing the source as a sample. The resulting activity of the source must be within 10% of the known target activity of the source for the calibration to be deemed acceptable. Additionally, all criteria with regards to FWHM and spectral resolution must be satisfied. Verifications which do not meet these criteria require a new calibration to be performed. In some instances this may involve replacing the actual detector prior to attempting a re-calibration. A new calibration is required whenever a detector is replaced or when calibration verification does not meet the defined acceptance criteria. Calibrations related to the analysis of Drinking Waters must be verified initially using a source that is un-related to the initial calibration source. Subsequent annual calibration verifications may be performed using the initial calibration source or a source that is un-related to the initial calibration source.

12. Procedure

Unless specified otherwise, the documented analysis process must be followed, as written, including the order of analytical process and the addition of chemicals.

12.1 Waters and Liquids, Including Drinking Water

12.1.1 Shake each sample and weigh 300g of aqueous sample into an appropriately sized beaker. Record the observed measured mass of sample to the lowest decimal on the balance. Do not remove sample from the beaker once it has been added. The actual quantity of sample used may be less than 300g if matrix interferences are expected or there is limited sample quantity available to the laboratory. If less than 300g of sample is used, dilute the sample with ASTM Type II DI water to the 300mL mark on the beaker. Fortify the pH of diluted samples by adding 2mL of HNO_3.

12.1.2 Prepare appropriate batch Quality Control samples including a Method Blank, Laboratory Control Sample (LCS), and LCS Duplicate (LCSD) by weighing an appropriate quantity of ASTM type II DI water to an appropriate size beaker. To each QC sample add 5mL of concentrated 16N HNO_3.

12.1.3 Add the applicable spikes to the appropriate QC samples in the amounts specified in Section 14 of this SOP. Add a pre-selected amount of the applicable working tracers to each of the samples and QC samples.

12.1.4 Add 1mL of iron carrier to each sample.

12.1.5 If there is a significant amount of residue present in the sample and total analysis is requested,

12.1.5.1 Filter the sample through a 0.45µm Metricel™ filter.

12.1.5.1.1 Transfer filtered sample to a labeled glass beaker

12.1.5.1.2 Transfer the Metricel™ filter and the residue to a labeled PTFE beaker.

12.1.5.2 To digest the filter and solids, add 10mL conc. 16N HNO_3, 10mL conc. 12N HCl, and 10mL conc. HF to the PTFE beaker. Cover the PTFE beaker with a cover and reflux on a hotplate for one hour.

12.1.5.3 Remove the cover and continue to heat the sample to dryness.

12.1.5.3.1 Repeat Step 12.1.5.2 as necessary based on the amount of residue remaining.

12.1.5.4 Dissolve the residue in 10mL conc. 12N HCl and 1mL saturated boric acid. Place the sample back on the hot plate and heat it to dryness again.

12.1.5.5 Dissolve the digested filter in 10mL of conc. 16N HNO_3. Place the sample back on the hotplate and heat to dryness.

12.1.5.6 Dissolve the digested filter in 10mL conc. 16N HNO_3. Use 8N HNO_3 to transfer the digested filter into the corresponding glass beaker (12.1.5.1.1) that contains the filtered aqueous sample fraction.

12.1.6 Place a watch glass on the glass beaker to cover the sample and heat it to boiling on a hotplate for a minimum of one hour.

12.1.7 Carefully add several mLs of conc. 15N NH_4OH to the sample while stirring until the pH of the sample is adjusted to 10 and a visible precipitate forms within the sample.

12.1.8 Heat the sample to boiling for another thirty minutes or longer, until an iron hydroxide precipitate breaks up into small particles.

12.1.9 Remove the sample from the hot plate and allow it to cool and the precipitate to settle.

12.1.10 Decant the excess supernate from the sample and discard it in the appropriate waste stream. Transfer the precipitate to a labeled disposable centrifuge tube with pH 10 ASTM Type II DI water.

12.1.11 Centrifuge the sample, and discard the supernate in the appropriate waste stream.

12.1.12 Rinse the iron precipitate with at least double its volume in pH 10 ASTM Type II DI water.

12.1.12.1 Vortex and centrifuge the sample. Discard the rinse in the appropriate waste stream. This rinse will eliminate excess ammonium hydroxide.

12.1.13 Proceed to the appropriate separation method beginning in Section 12.5. For Uranium in drinking water analysis, perform the uranium-specific method detailed in section 12.5.

12.2 Filter Samples

12.2.1 If the filter is cellulose:

12.2.1.1 Place a representative portion depending on the client requirements or radioactivity levels of the sample (usually one half or one quarter) of the filter into a clean ceramic crucible and pipette a selected amount of the working tracers onto it.

12.2.1.2 Cover the sample with aluminum foil and heat sample in a muffle furnace using the RAMP feature to ash the filter.

12.2.1.2.1 Use the temperature ramping guidelines in the current revision of the sample preparation SOP.

12.2.1.2.2 NOTE: Mark the PACE sample ID on each crucible with a high temperature wax pencil.

12.2.2 If the filter is glass fiber or otherwise non-organic:

12.2.2.1 Place a suitable portion of the filter into a PTFE beaker and pipette a selected amount of the working tracers onto the sample.

12.2.3 Proceed to step 12.3.3 for the sample digestion.

12.3 Soil and Vegetation Samples

12.3.1 Weigh a suitable amount of the dried sample into a ceramic crucible and pipette a selected amount of the working tracers onto the sample. Prepare a crucible designated as the LCS and LCSD if applicable and spike with the appropriate spike solutions. Prepare a crucible designated as the MB, and add 0.5mL iron carrier to the samples and all QC samples.

12.3.2 Cover the crucibles with a ceramic lid and place the crucible into a cold muffle furnace. Set the final temperature at 550ºC. Once this temperature has been reached, maintain for at least 4 hours or longer depending on the amount of material in the crucible. (For samples which are obviously highly organic, use the muffle oven's RAMP feature to prevent flashover as the sample organic components burn off.)

12.3.2.1 At the end of 4 hours, turn off the oven, allow the samples to cool to room temperature, and remove the samples from the furnace.

12.3.3 Remove the crucibles from the oven and re-label the crucibles. Add 10mL of conc. HNO_3 and 10mL of conc. HCl to the sample and heat it to dislodge the solids.

12.3.4 If the soil/solid sample does not require uranium or thorium then proceed to 12.4. Otherwise transfer the sample to a clean, labeled PTFE beaker.

12.3.4.1 Use a PTFE scraper to remove any residue that is stuck to the bottom of the crucible and rinse it into the PTFE beaker with 9N HCl.

12.3.5 Add 10mL of conc. HF to each sample in the PTFE beaker and then cover with a PTFE watch glass.

12.3.6 Reflux on an electric griddle or hotplate set at 250ºC for a minimum of thirty minutes. After 30 minutes, remove the watch glass cover and allow the sample to evaporate to dryness.

12.3.7 Add 10mLconc. HCl, 10mL conc. HNO_3, and 10mL conc. HF to the sample. Return the sample to the griddle, and allow it to evaporate dryness. This process may be repeated as often as necessary to ensure complete dissolution of the sample. In some matrices, high temperature oxides have been formed which severely limit analyte recovery. These highly insoluble oxides must be treated with repeated and prolonged contact with mineral acid. Acids must cover the samples for multiple days at low heat in order to properly dissolve the sample.

12.3.8 Add 10mL conc. HNO_3 and 10mL conc. HCl to the sample. Return the sample to the griddle and allow it to evaporate to dryness.

12.3.9 Add 10mL 12N HCl or 16N HNO_3 depending on the acid used for the load solution in order to minimize complexants. (For example, if the load solution for the column work will be 15mL 8N HNO_3, add 10mL 16N HNO_3 to the sample for the final cookdown to remove as much chloride as possible.) Add 1mL saturated boric acid to the sample and return it to the griddle and allow it to evaporate to dryness.

12.3.10 Dissolve the sample in the appropriate load solution depending on the column method to be utilized, for example 8N HNO_3 for uranium and thorium isolation using nitric conditioned anion columns, or 9N HCl/H_2O_2 for americium and plutonium isolation using chloride conditioned anion columns.

12.3.11 Heat to aid in dissolution of the sample solids. Scrape the bottom of the beaker to dislodge any solids. Centrifuge the sample as necessary prior to loading the sample onto columns.

12.3.12 Proceed to the appropriate separation method beginning in section 12.5.

12.4 Soil leach for samples not requiring Uranium or Thorium

12.4.1 Transfer the muffled and traced sample from the crucible to a labeled 100mL glass beaker. A larger beaker may be used if necessary.

12.4.2 Add 15mL HNO_3 and 5mL of HCl to each sample and cover with a watch glass. More acid may be necessary for larger aliquots but the ration of 3:1 should remain the same.

12.4.3 Reflux on a hotplate or griddle for a minimum of thirty minutes at medium to low heat. Samples must be kept from overheating, boiling or bumping.

12.4.4 Remove the samples from the hotplate and allow them to cool. Slurry the samples in the beaker and transfer the leachate and solids to a clean, labeled centrifuge tube with the aid of ASTM Type II DI water.

12.4.5 Centrifuge the samples for 15 minutes at maximum speed.

12.4.6 Decant the leachate to a 250mL labeled glass beaker. Place this beaker on a hotplate at medium and evaporate to dryness.

12.4.7 To the solids in the centrifuge tube repeat the steps from 12.4.2 to 12.4.6 using the same labeled glass beaker until the solids appear light gray or tan.

12.4.8 After evaporating to dryness, solids must be converted to the appropriate acid type by treatment with a quantity of the appropriate concentrated acid. If the chemistry load solution is to be a nitrate column, add 10mL of concentrated nitric acid and evaporate to dryness. Likewise, if the chemical load solution is chloride-based, add 10mL of concentrated HCl and evaporate to dryness. This step is not necessary for the isotopic plutonium/neptunium analysis.

12.4.9 Proceed to the appropriate separation method beginning in Section 12.5.

12.5 Method 1: Separation of Actinides on Anion Resin Using Hydrochloric Acid Solution

12.5.1 This method uses an anion exchange resin and the parameters dissolved in a hydrochloric acid solution to separate and purify any of the following groups of actinides.

- Americium, Plutonium (Neptunium), and Uranium
- Thorium, Plutonium (Neptunium), and Uranium
- Plutonium, Neptunium, and Uranium
- Uranium in drinking water samples.

12.5.1.1 This is the default method for waters and soils (less than 2 grams) where little or no interferences are expected to be present.

12.5.1.2 It is not easy to separate americium/curium from thorium using this method, so if sequential analysis requires both analyses be performed on a single aliquot, start with Method 2 to isolate the thorium fraction first from all of the other nuclides.

12.5.2 Dissolve the sample precipitate in 15mL of 9N HCl / 0.10% H_2O_2. The hydrogen peroxide need only be used if plutonium will be isolated.

12.5.3 Prepare a 2mL resin column using Bio-Rad AG 1x8.

12.5.3.1 Slurry the resin with ASTM Type II DI water to give a resin bed of about 3cm. The final resin volume must be consistent for all columns.

12.5.3.2 Once the ASTM Type II DI water has drained, condition the column with 10mL of 9N HCl.

12.5.4 If heated, allow the sample to cool, then transfer it to the prepared resin column.

12.5.5 Collect the effluent from the column in a clean labeled centrifuge tube if either americium/curium or thorium analysis is desired.

12.5.5.1 Rinse the sample beaker or centrifuge tube with an additional 10mL 9N HCl / 0.10% H2O2 and collect.

12.5.5.2 Rinse the column with 20mL of 9N HCl and collect.

12.5.5.3 Transfer the contents of the centrifuge tube to a clean labeled glass beaker.

12.5.5.4 If the contents are to be analyzed for americium/curium, proceed to Method 3: Americium/Curium Purification on TRU-Resin™ for americium/curium analysis.

12.5.6 If it is desirable or necessary to isolate isotopic Plutonium from Neptunium, elute the plutonium from the column into a clean, labeled glass beaker with 20mL 9N HCl / 0.05N NH_4I.

12.5.6.1 If analysis for plutonium is not required, do not perform this step and proceed to step 12.5.7.

12.5.6.2 Add 10mL conc. HNO3 and 3-4 drops of the iron carrier to the plutonium fraction.

12.5.6.3 Allow enough time for the nitric acid and the hydrochloric acid to react.

12.5.6.4 With a disposable pipette, and in a drop wise fashion, slowly add approximately 1mL of 30% H_2O_2 to the solution.

12.5.6.5 Place the beaker on a hotplate and allow the plutonium fraction to evaporate to dryness.

12.5.6.6 Add 5mL concentrated HCl to each beaker and evaporate the contents to dryness on a hotplate.

12.5.6.7 Dissolve the residue in the beaker with 10mL 9N HCl.

12.5.6.8 Transfer the plutonium fraction to a labeled centrifuge tube with ASTM Type II DI water and dilute to 20mL with ASTM Type II DI water.

12.5.6.9 Proceed to Step 12.9.2, Micro-Precipitation of Plutonium.

12.5.7 Rinse the column with 15mL of 6N HCl / 0.52N HF, which will elute any neptunium from the sample (Use 20 mL if step 12.5.6 was not utilized and Plutonium is being eluted with Neptunium.

12.5.7.1 Proceed to Step 12.9.5 for the Micro-Precipitation of Neptunium. (Step 12.9.2 if analyzing for Plutonium and Neptunium simultaneously.)

12.5.7.2 If analysis for neptunium is not required, discard the effluent.

12.5.8 Rinse the column with 10mL of 9N HCl and discard the effluent.

12.5.9 Elute the uranium fraction from the column into a clean, labeled centrifuge tube with 20mL of 0.1N HCl.

12.5.9.1 Proceed to Step 12.9.3 for the Micro-Precipitation of Uranium.

12.6 **Method 2: Separation of Actinides on Anion Resin using Nitric Acid Solution**

12.6.1 This method uses an anion exchange resin and the parameters dissolved in a nitric acid solution to separate and purify the following group of actinides:

- Americium, Plutonium and Uranium from Thorium

12.6.1.1 This method is best used for solids (any amount), waters, or filters (organic based, cellulose), and when it is desirable to reuse the resin column after isolating Thorium.

12.6.2 Dissolve the sample into 15mL of 8N HNO_3

12.6.2.1 Heat the solution if necessary to aid in the dissolution of the precipitate.

12.6.3 Prepare a 2mL resin column with Bio-Rad AG 1x8.

12.6.3.1 Slurry the resin with ASTM Type II DI water to give a resin bed of about 3cm.

12.6.3.2 When the ASTM Type II DI water has drained, condition the column with 20mL 8N HNO_3.

12.6.4 Cool the sample and transfer it to the prepared resin column.

12.6.4.1 Collect the effluent from the column in a clean glass beaker if uranium/plutonium analysis is required. This fraction will also contain any Americium/Curium or Neptunium if present in the sample.

12.6.4.2 Rinse the column with an additional 15mL 8N HNO_3 and collect. The rinse will contain any residual americium, plutonium, and uranium from the sample.

12.6.4.3 Repeat step 12.6.4.2. Collect this rinse.

12.6.4.4 Place the americium, plutonium, uranium fraction on a hotplate and allow it to evaporate to dryness.

12.6.4.5 Continue with the separation of analytes in the solution in step 12.6.4.4 by proceeding to Step 12.5.2 after adding 15mL conc. HCl, and evaporating the samples to dryness.

12.6.5 Elute the thorium from the column with 25mL of 9N HCl into a labeled centrifuge tube.

12.6.5.1 Transfer the solution containing the thorium from the centrifuge tube to a clean, labeled plastic cup and add 0.8mL of 10mg/mL iron carrier.

12.6.5.2 Dilute the samples to approximately 220mL with ASTM Type II DI water.

12.6.5.3 Precipitate thorium with iron as a hydroxide by adding 22-25mL of concentrated ammonium hydroxide to each sample dilution. Stir the solution vigorously with a stir rod for several seconds until a distinct iron hydroxide precipitate forms.

12.6.5.4 Allow the samples to settle for about 15 minutes, and stir vigorously once more to further break up the iron hydroxide precipitate.

12.6.5.5 Remove the stir rod and allow the samples to completely settle for a minimum of one hour.

12.6.5.6 Decant the excess supernate and transfer the iron hydroxide precipitate to the original labeled centrifuge tubes from Step 12.6.5.1.

12.6.5.7 Centrifuge the samples and discard the supernate. Dissolve the precipitate in 3mL 9N HCl. Dilute the samples to 25mL with ASTM Type II DI water.

12.6.5.8 Proceed to Step 12.9.4 for the Micro-Precipitation of Thorium.

12.6.5.9 If the column is to be reused for the uranium purification, rinse the column with 25mL of ASTM Type II DI water.

12.6.6 Perform all of steps listed in Method 1 to purify the americium, plutonium, and uranium as required.

12.7 **Method 3: Purification of Americium/Curium Using Tru-Resin™**

12.7.1 This method may be used as the initial separation step for liquids or waters only if americium/curium is requested. It must be used subsequent to Method 1 in all other instances to purify americium/curium and separate them from thorium.

12.7.2 Add 3-4 drops of the iron carrier to the americium fraction collected in Step 12.5.5.4. Place the sample on a hotplate and evaporate the sample to dryness.

12.7.3 Add 5mL of concentrated nitric acid to each sample and evaporate to dryness to remove residual chlorides.

12.7.4 Dissolve the sample residue into 10mL of 2N HNO_3 /0.5M $Al(NO_3)_3$. Additional 2N HNO_3/0.5M $Al(NO_3)_3$ may be used to dissolve the sample fraction, but the total load volume must not exceed 15mL.

12.7.4.1 Heat the sample gently as necessary to aid in the dissolution.

12.7.5 Add one drop of 0.5M potassium thiocyanate to the sample and swirl it, then add 6-8 drops of 1.0M ascorbic acid to the sample and swirl it.

12.7.5.1 The color of the sample should go from clear to red and then back to clear again, unless there is no significant iron present in the sample.

12.7.6 The sample should be centrifuged prior to loading it on the column if there are any solids present.

12.7.7 Prepare a resin column with 5 drops of pre-resin filter followed by 3.5mL (2 grams) of Tru-Resin™.

12.7.7.1 Allow any excess water to drain.

12.7.7.2 Condition the columns by passing 5mL of 2N HNO_3 through them and discarding the effluent.

12.7.8 Load the sample onto the column.

12.7.8.1 A change in resin color should be observed, usually pink, but it may be multi-colored.

12.7.8.2 Discard the effluent.

12.7.9 Rinse the column with three 5mL aliquots of 2N HNO_3 allowing each to pass through the column. Discard the effluents.

12.7.10 Place a clean, labeled centrifuge tube under the columns and elute the americium/curium from the resin with 2mL of 9N HCl.

12.7.11 Complete the elution of the americium/curium from the column by adding 10mL 4N HCl. Combine the effluent from the column

in the centrifuge tube. If interferences are expected to cause spectral resolution issues, proceed directly to step 12.8.3.

12.7.12 Proceed to Step 12.9.1 for the Micro-Precipitation of Americium.

12.8 **Method 4: Purification of Americium/Curium Using TEVA-Resin**

12.8.1 After Method 3 has been preformed, the following steps are available to further purify the americium/curium.

12.8.2 Transfer the americium/curium eluate from step 12.7.11 to a small glass beaker. Place the sample on a hotplate and allow it to evaporate to dryness

12.8.2.1 Do not allow the samples to bake.

12.8.3 Dissolve the residue in the beaker in 10mL conc. HCl and place it back on the hotplate allowing it to evaporate to dryness.

12.8.3.1 Do not allow the sample residue to bake.

12.8.4 Add 1mL conc. formic acid to each sample and allow them to evaporate to dryness.

12.8.5 Dissolve the sample in 15mL 3M ammonium thiocyanate/ 0.1N formic acid.

12.8.5.1 Heat the sample as necessary to aid in the dissolution.

12.8.5.2 The sample should take on a light pink color.

12.8.6 Prepare a 2cm column with 3.5mL prepared (2 grams) TEVA resin™.

12.8.6.1 Allow any excess water to drain through.

12.8.6.2 Condition the column with 5mL of the 3M ammonium thiocyanate/ 0.1N formic acid.

12.8.7 Ensure that the sample has cooled and load it onto the conditioned column.

12.8.7.1 Rinse the sample beaker with 2mL 1M ammonium thiocyanate/ 0.1N formic acid.

12.8.7.2 Pour the rinse onto the column when the sample has completely passed through.

12.8.8 Rinse the column first with 3mL and then with 5mL of the 1M ammonium thiocyanate/ 0.1N formic acid.

12.8.8.1 Discard all rinses to waste. (Note: Ammonium thiocyanate should be neutralized with dilute nitric acid in an open container prior to disposal to avoid violent reactions.)

12.8.9 Elute the americium/curium from the column with 15mL of 2N HCl into a clean, labeled centrifuge tube.

12.8.10 Proceed with Step 12.9.1 for Micro-Precipitation of Americium.

12.9 Micro-precipitation of the Actinides

12.9.1 Americium/Curium

12.9.1.1 Add 0.1mL of the 0.5 mg/mL neodymnium carrier to the sample. Swirl the sample to mix the contents.

12.9.1.2 Add 3mL conc. HF to the sample. Cap the centrifuge tube and shake the sample vigorously to mix the contents.

12.9.1.3 Allow the sample to sit for thirty minutes, then proceed with Step 12.9.6 for filtration.

12.9.2 Plutonium

12.9.2.1 Add 0.1mL 0.5mg/mL neodymnium carrier to the sample and swirl to mix.

12.9.2.2 Add 12 drops of 25% dihydrazine dihydrachloride solution to each sample.

12.9.2.3 Swirl the solution to mix the contents, then allow the sample to sit for 5 minutes.

12.9.2.4 Add 3mL of conc. HF to the sample. Cap the sample and shake it vigorously to mix the contents.

12.9.2.5 Allow the sample to sit for thirty minutes.

12.9.2.6 Proceed to Step 12.9.6 for filtration.

12.9.3 Uranium

12.9.3.1 Add 0.1mL 0.5mg/mL neodymnium carrier to the sample and swirl to mix.

12.9.3.2 Add enough of the titanium chloride solution to the sample to maintain a light purple color when swirled (approximately 1-2mL).

12.9.3.3 Swirl the solution to mix the contents, then allow the sample to sit for a few minutes.

12.9.3.4 Add 3.0mL conc. HF to the sample. Cap the sample and shake it vigorously to mix the contents.

12.9.3.5 Allow the sample to sit for thirty minutes.

12.9.3.6 Proceed with Step 12.9.6 for filtration.

12.9.4 Thorium

12.9.4.1 Add 0.1mL 0.5mg/mL neodymnium carrier to each sample. Swirl the contents to mix.

12.9.4.2 Add 3.0mL conc. HF to the sample. Cap the sample and shake it vigorously to mix the contents.

12.9.4.3 Allow the sample to sit for thirty minutes.

12.9.4.4 Proceed with Step 12.9.6 for filtration.

12.9.5 Neptunium

12.9.5.1 Add 0.1mL 0.5 mg/mL neodymnium carrier to each sample. Swirl the contents to mix.

12.9.5.2 Add 3.0mL conc. HF to the sample. Cap the sample and shake it vigorously to mix the contents.

12.9.5.3 Allow the sample to sit for thirty minutes.

12.9.5.4 Proceed with Step 12.9.6 for filtration.

12.9.6 Filtration

12.9.6.1 Prepare the filtration unit by turning on the vacuum and rinsing each apparatus with severalmLs of ASTM Type II DI water.

12.9.6.2 Carefully wash and rinse each of the filter funnels and set them aside.

12.9.6.3 Place a polypropylene filter onto each unit and ensure that it is centered.

12.9.6.4 (Carefully) Place the funnels on the filtration unit.

12.9.6.4.1 Do not rip the filters.

12.9.6.4.2 Rinse the sides of the funnels with 80% reagent grade alcohol.

12.9.6.5 When the sample has completely passed through the filter, rinse the sides of the funnel with a few mLs of the 0.1% HF solution.

12.9.6.6 Rinse the funnel and filter with a few mLs of 80% reagent grade alcohol.

12.9.6.7 When the rinses have completely passed through the filter, remove the filter funnels, turn off vacuum, and carefully remove the filter paper with tweezers.

12.9.6.8 Place the filter paper onto a labeled pre-taped metal disc for counting.

12.9.6.8.1 Allow the filter to completely dry prior to counting by placing it in a labeled petri dish, and storing it in the count room until ready to count.

12.9.6.9 Count the samples in an alpha spectrometry detector as instructed in the current revision of the instrument SOP, PGH-R-020.

12.9.6.10 Obtain instrument printouts and perform calculations as detailed in Attachment 1 of this SOP.

12.9.6.11 If plutonium-241 analysis is desired, the Pu counting source must be removed from the taped disk after alpha spectroscopy counting using a minimal amount of acetone.

12.9.6.12 Place the filter in a glass liquid scintillation vial. Cover the vial with aluminum foil and muffle it in an oven at 550°C for a minimum of 2 hours or until the filter has completely ashed away and there is no black residue left in the vial.

12.9.6.13 Remove the vial from the oven and discard the foil. Add 6 drops of saturated boric acid and 1mL of concentrated HCl to each sample and heat to dryness.

12.9.6.14 Add 2.0mL 0.1N HCl to each sample to dissolve the residue and 15mL of Ultima Gold AB liquid scintillation cocktail. Cap the vial and shake it vigorously.

12.9.6.14.1 The samples should be free of any color.

12.9.6.15 Clean the outside of each vial with acetone followed by ASTM Type II DI water to remove any finger prints or residue.

12.9.6.16 Dark-adapt the samples for one hour prior to counting in a calibrated, liquid scintillation counter in accordance with the liquid scintillation instrument operating SOP, PGH-R-022, current revision.

13. Calculations

13.1 Refer to Attachment I of this SOP for all actinide analysis associated calculations.

13.2 Any verified result for drinking water that exceeds the maximum contaminant level (MCL) established for Uranium must be reported to the appropriate personnel and agencies according to the specific requirements of the state where the water was sampled. The directions for reporting and results that exceed the MCL limits are documented in the State Drinking Water Emergency Reporting Requirements Binder and Pace SOP PGH-C-025, current revision.

13.2.1 Uranium MCL >= 20 pCi/L total uranium (U-238 + U-235 + U-234)

14. Quality Control

14.1 General guidelines for drinking water samples with results that exceed the Maximum Contaminant Level include the following: (All steps are to be conducted as soon as the exceedence has been identified.)

14.1.1 Verify the result(s) to ensure that there were no transcription or calculation errors and that all QC results are within the acceptable limits. Correct any problems and determine the new result. If there were no errors or the result still exceeds the MCL, continue with the reporting process.

14.1.2 Immediately notify the Department Manager/Supervisor, and QA Department that a reportable result has been identified. Use telephone notifications to inform the contact people if the variance is identified after hours along with an e-mail follow up to document the event.

14.1.3 Refer to the State Drinking Water Emergency Reporting Requirements Binder for the state specific information regarding the proper course of action to take. Time is of the essence during this process with some of the state reporting requirements as short as 1 hour from the verification of an exceedence

14.2 Each analyst who performs this test must satisfactorily complete a Demonstration of Capability Study as documented in Section 3.4 of the most recent revision of the Quality Assurance Manual.

14.2.1 The DOC study results are evaluated against the LCS acceptance limits.

14.3 Daily instrument Quality Control checks for the alpha spectrometer must be completed following the instructions detailed in the current revision of Pace SOP PGH-R-020, “Alpha Spectroscopy Instrument Operations.”

14.4 Daily instrument Quality Control checks for the liquid scintillation counter must be completed following the instructions detailed in the current revision of Pace SOP PGH-R-022, “Liquid Scintillation Instrument Operations.”

14.5 Daily instrument Quality Control checks for the gas flow proportional counters must be completed following the instructions detailed in the current revision of Pace SOP PGH-R-002, “Gas Flow Proportional Instrument Operations.”

14.6 See Appendix II for performance indicator evaluation calculations and criteria. Numerical performance indicators may be used to assess QC for non-drinking water samples when the default assessment indicates a QC failure. The numerical performance indicator must be within +/- 3 for all other matrices. The z-score for precision assessment may be used for drinking waters with the approval of the Department Manager/Supervisor using the +/- 2 specification.

14.7 Sample Tracer Recovery/Tracer Peak Energy

14.7.1 Sample tracer is added to each sample and used to calculate sample recovery. Tracer recovery is required to be within 30%-110% for samples analyzed under jurisdiction of the DOD QSM. Pace’s default acceptance criteria for tracer recovery requires recovery between 30%-110%; however, recoveries may be acceptable between 10% and as high as 130% with the documented permission of the Department Manager/Supervisor or specified designee.

14.7.2 Samples with tracer recoveries outside of this range should be re-prepped with direction from the Department Manager or Manager-specified designee after determining possible causes.

14.7.3 Sample tracer counts should be sufficient to minimize the expanded uncertainty of the count and any potential for tailing of the tracer counts into other regions of interest. A minimum of 400 tracer counts is recommended, however, in some instances, the sample matrices may prohibit achieving a minimum 400 tracer counts, and the sample data must be evaluated prior to extending or recounting samples solely to achieve a minimum 400 tracer counts.

14.7.4 Excluding samples analyzed for isotopic thorium content using Th-229 as a tracer, samples analyzed under jurisdiction of the DOD QSM, the tracer energy must be within 40 keV of the known tracer isotope peak energy.

14.7.5 For samples associated with the DOD QSM, if the tracer energy is not within 40 keV of the known peak energy, this may indicate an issue with the detector used for analysis. The sample may be reanalyzed using an alternate detector. If re-analysis results indicate an acceptable tracer energy peak location, results may be reported. If, following re-preparation and re-analysis, the tracer peak is not within 40 keV of the known peak energy, results may be reported with appropriate “J” flagging.

14.7.6 If the tracer is a non-Th-229 alpha emitter, its peak full width half maximum (FWHM) value must be evaluated and generally should be less than 100 keV. When the FWHM is greater than 100 keV the sample data must be inspected using professional judgment to determine if the detector was functioning properly and if the procedure was adequate. Data that has FWHM up to 125 keV may be reported with qualification, except for samples analyzed under the requirements of the DOD QSM. For DOD-associated samples, if the tracer FWHM exceeds 100 keV, the affected sample must either be re-prepared and re-analyzed or the alpha count source may be “re-purified” in accordance with the process outlined in this SOP. If count source re-purification is attempted but fails the FWHM criteria, the sample must be re-prepared and re-analyzed. If upon re-preparation and re-analysis the sample exhibits a tracer peak energy outside of established control criteria, analysis results may be reported with the appropriate “J” flag.

14.7.7 Unlike other radioactive tracers used for yield monitoring, the alpha peak energies associated with Th-229 are of lower abundance and greater energy distribution over the region of interest associated with Th-229. For this reason, there is a greater uncertainty in calculating peak centroid energies and FWHM values when using spectroscopy systems that perform alpha spectral analysis of. When it is observed that the Th-229 centroid energy is calculated to be greater than 40 keV from the average Th-229 peak energy or when the Th-229 peak resolution is determined to be greater than 100 keV, analytical spectra must be reviewed and approved by the Department Manager or a

Department Manager specified designee. For approval, analytical spectra must document clear de-markation between the Th-229 tracer and adjoining analyte regions of interest.

14.8 Method Blank (MB)

14.8.1 One MB must be prepared for each analytical batch. The purpose of the MB is to monitor for cross contamination during the analytical process. When available, the MB should be prepared from a similar matrix as samples contained in the analytical batch. If appropriate blank matrix material is not available, ASTM Type II DI water (Reagent Blank) must be carried through the procedure. A reagent blank may be used for sample correction purposes following approval of affected clients.

14.8.2 The MB result must be less than the MDC. If the method blank result is greater than MDC, individual sample results may still be reportable.

14.8.2.1 PASI's default criteria allows reporting of sample results less than the CRDL (contract required detection limit) or greater than 10 times the blank result. Relative sizes of the sample and blank aliquots must be factored when making this determination (raw counts).

14.8.2.2 For samples analyzed under the DoD QSM, the Method Blank result must be less than ½ the detection limit. Corrective action is necessary for any MB result greater than ½ the detection limit.

14.9 Sample Duplicate (DUP)

14.9.1 One Duplicate Sample (DUP) must be randomly assigned within each batch. The purpose of the sample DUP is to measure precision of the analytical process. Laboratory duplicates are not intended to assess precision related to the sample collection process. Sample collection precision can only be assessed through collection of duplicate samples at the time of sample collection. The sample DUP is a duplicate volume of sample processed identically as other samples in the analytical batch.

14.9.2 For batches with drinking water samples originating from the state of Arizona, Duplicate Samples (DUP) must be randomly assigned within each batch at a frequency of no less than 10%. A batch of ten samples or fewer must contain at least one duplicate sample. A batch of greater than 10 samples up to 20 samples must contain a minimum of two duplicate samples if the batch contains samples originating from Arizona.

14.9.3 Calculation

$$\%RPD = \frac{\left|\left(R_S - R_D\right)\right|}{\left(\left(R_S + R_D\right)/2\right)}$$ Where:

R_S = sample activity concentration
R_D = duplicate activity concentration

14.9.4 Duplicate sample performance is acceptable when the %RPD is <25%.

14.10 Sample Matrix Spikes (MS)

14.10.1 Because this analytical method requires the use of radiotracers for yield determination, PASI's default QC policy is that a sample matrix spike (MS) is not required for alpha spectrometry analyses except for drinking water analysis.

14.10.2 A matrix spike is prepared by spiking a known amount of spike solution (Am-241, Cm-244, Pu-239, Th-232, U-238, Np-237, etc.) into a portion of one sample in the batch, and it must be processed identically as for other samples. The purpose of the MS is to assess the effect of sample components on the analytical process. The volume of sample used for the MS must be equivalent to the volume used for sample analysis. The spike amount should be approximately 10 times the detection limit and not less than 20% of the anticipated sample concentration.

14.10.3 Matrix Spike Recovery Calculation

$$\%REC = \frac{(x - x_0)}{c} x100$$ Where:

x = measured concentration of the spiked sample
x_0 = measured concentration of the unspiked sample
c = spike concentration added

14.10.4 MS performance is acceptable when agreement of the measured value and the expected value is within ±25% of the true value.

14.11 Sample Matrix Spike Duplicates (MSD)

14.11.1 A sample Matrix Spike Duplicate (MSD) is not required for this analysis. When required by the customer/contract, a MSD must be prepared for each analytical batch. The MSD must be prepared as a duplicate of the MS.

14.11.2 For all matrices the MSD must pass the criteria established for the MS. Additionally, the MS and MSD must pass the criteria established for duplicate precision.

14.12 Laboratory Control Sample (LCS)

14.12.1 One LCS must be prepared for each analytical batch and is a reference material that contains a known concentration of spike (Am-241, Cm-244, Pu-239, Th-232, U-238, Np-237, etc.) in a matrix that is similar to the samples within the batch. If this material is not available, a well-characterized material (WCM) may be used. If neither of these is available, DI may be spiked with a spike solution greater than 2 times the detection limit.

14.12.2 LCS Recovery calculations

$$\%REC = \frac{x}{c} x100$$ Where:

x = Analytical result of the LCS
c = Known concentration of the LCS

14.12.3 LCS performance is acceptable when the %REC is within ±25% of the known value.

14.13 Laboratory Control Sample Duplicate (LCSD)

14.13.1 A LCSD must be analyzed to measure batch precision whenever adequate sample volume is not available for sample DUP analysis. The LCSD must be prepared in an identical fashion as the LCS and processed identically as for other samples.

14.13.2 The LCSD must pass the criteria established for the LCS.

14.13.3 Additionally, the LCS and LCSD must pass the criteria established for duplicate precision.

14.14 Summary of QC related Activities:

Method Blank	One per Batch
Reagent Blank	One per Batch (as required by client)
Duplicate Sample	One per Batch (frequency of 1 per 10 samples for batches containing DW originating from AZ)
Matrix Spike	One per Batch (for drinking water analysis or as required by client)
Matrix Spike Duplicate	One per Batch (frequency of 1 per 10 samples for batches containing DW originating from AZ or as required by client)
Laboratory Control Sample	One per Batch
Laboratory Control Sample Dup	One per Batch for samples in the absence of Duplicate sample.

14.15 Corrective Actions for Out-Of-Control Data

14.15.1 Method Blank (Reagent Blank) (MB/RB) – Individual samples that do not meet the acceptance criteria must be reanalyzed. If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

14.15.2 Duplicate (DUP) – DUP analysis that fails the replicate test must be reanalyzed to determine if analytical failure or sample heterogeneity was the cause of the problem.

14.15.3 Matrix Spike Recovery (MS) – MS recoveries that fail high and outside of control criteria with a sample result that is less than the reporting limit may be reported with narration. Additionally, MS recoveries that fail low and outside of control criteria for

Drinking Water samples with a sample result that is greater than the MCL must be reported with comment as potentially biased low due to matrix interference. Otherwise, MS recoveries that do not meet the acceptance criteria must have that sample reanalyzed. If a Matrix Spike Duplicate is also analyzed and the recovery is comparable to the MS, the results are reported and noted in the final report. Matrix effect must be determined by re-analysis of the MS/Sample pair or demonstration of acceptable precision between a MS/MSD pair.

14.15.4 The analyst must evaluate the MS results to attempt to determine the cause of the failure and the appropriate action to take based on that evaluation. All decisions made must be documented.

14.15.5 Matrix Spike Duplicate (MSD) - If an MSD is analyzed and the recovery is comparable to the MS, the results are reported with qualification in the final report.

14.15.6 Laboratory Control Sample (LCS) – If an LCS analysis does not meet the acceptance criteria, the entire analytical batch must be re-prepped and reanalyzed.

The results of the batch may be reported, with qualification in the final report, if the LCS recoveries are high and the sample results within the batch are less than the reporting limit.

14.15.7 Laboratory Control Sample Duplicate (LCSD) – If an LCSD does not meet the recovery acceptance criteria, the entire analytical batch must be reanalyzed.

The results of the batch may be reported, with qualification, if the LCS recoveries are high and the sample results within the batch are less than their the reporting limit, and duplicate precision meets the acceptance criteria.

14.15.8 If there is no additional sample available for reanalysis, evaluate the usefulness of the data in the final report.

14.16 Contingencies for handling Out-of-Control or Unacceptable Data

14.16.1 Method Blank (Reagent Blank): If the sample is exhausted, evaluate the usefulness of the data in the final report.

14.16.2 Duplicates: If the sample is exhausted, evaluate the usefulness of the data in the final report.

14.16.3 Matrix Spike Recovery: If a Matrix Spike is analyzed and the spike recoveries are not comparable, and the sample is exhausted, evaluate the data usefulness in the final report.

14.16.4 Matrix Spike Duplicate: If a Matrix Spike Duplicate is analyzed and the spike recovery is not comparable to the Matrix Spike and the sample is exhausted, evaluate the data usefulness in the final report.

14.16.5 Tracer recovery: If the tracer recovery is less than 30% but greater than 10% with more than 400 tracer counts, the sample may be reported with supervisor permission. Tracer recovery above 110% but below 130% may be reported but must be narrated. Samples with tracer recovery below 10% or above 130% must be re-analyzed. If the sample is exhausted, evaluate the usefulness of the data in the final report.

15. Method Performance

15.1 Each analyst must read and understand this procedure with written documentation maintained in their training file on the Learning Management System (LMS).

15.2 An initial demonstration of capability (IDOC) study must be performed. A record of the IDOC will be maintained on file in each analysts training file in the LMS.

15.3 On an annual basis, each analyst will complete a continuing demonstration of capability (CDOC).

15.4 Laboratory Control Samples are analyzed with each batch, the results are charted to monitor control limits and trending.

16. Pollution Prevention and Waste Management

16.1 Place radioactive waste into the appropriate receptacles.

16.2 Discard acidified samples and unusable standards into the proper waste drains.

16.3 Dispose of waste materials in accordance to type: (Non-hazardous, hazardous, non-radioactive, radioactive or mixed).

17. References

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17.3 Krieger, H. L. and Whittaker, E. L., Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, “Uranium-Radiochemical Method,” Method 908.0, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH, August, 1980.

17.4 ASTM E181-93, Standard Test Methods for Detector Calibration and Analysis of Radionuclides, ASTM Standards, Vol. 12.02.

17.5 ASTM D-3972-90, Test Method for Isotopic Uranium in Water by Radiochemistry, ASTM Standards, Vol. 12.04.

17.6 Table of Radioactive Isotopes, Brown and Firestone, Shirley editor, John Wiley & Sons, 1986.

17.7 Currie, L., Limits for Quantitative Detection and Quantitative Determination, Analytical Chemistry, Vol. 40. No. 3, Pg 586-593, 1968.

17.8 Currie, L., Lower Limit of Detection: Definition and Elaboration of a Proposed Position for Radiological Effluent and Environmental Measurements, NUREG/CR - 4007, USNRC, 1984.

17.9 "American National Standard Calibration and Usage of Alpha/Beta Proportional Counters", ANSI N42.25-1997.

17.10 "Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)", July 2004, Final.

17.11 Eichrom Industries, Various Actinide Procedures. Darien, Illinois, 1995

17.12 EML Procedures Manual, HASL-300 28th Edition.

17.13 Pace SOP PGH-R-001, current revision (Analysis of Samples for Gross Alpha and Gross Beta content).

17.14 Pace SOP PGH-R-002, current revision (Gas Flow Proportional Counter Operation).

17.15 Pace SOP PGH-R-020, current revision (Alpha Spectroscopy Instrument Operations).

17.16 Pace SOP PGH-R-022, current revision (Liquid Scintillation Counter Operations).

17.17 Pace SOP PGH-R-024, current revision (Radiochem Sample Preparation).

17.18 Pace SOP PGH-C-027, current revision (Deionized Water Quality and Suitability).

17.19 Pace SOP PGH-C-025, current revision (Reporting SDWA MCL Violations).

17.20 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, Program Policy and Structure (most recently approved revision).

17.21 TNI Standard, Requirements for Laboratories Performing Environmental Analysis, current version.

17.22 Pace Analytical Services, LLC. - Pittsburgh Laboratory Quality Assurance Manual, current version.

17.23 Department of Defense (DOD), Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories; DOD QSM version 5.1, DOE Quality Systems for Analytical Services Version 3.1, 2017.

18. Tables, Diagrams, Flowcharts, Appendices, etc.

18.1 Attachment No 1: Concentration Calculation & Counting Uncertainty

18.2 Attachment No 2: Evaluation of QC using Numerical Indicators.

18.3 Figure No 1: Analysis Flowchart for Sequential Analysis of Am, Pu, U, and Th

18.4 Figure No 2: Analysis Flowchart for Sequential Analysis for Am, Pu, U (Regular Water or Solids)

18.5 Figure No 3: Analysis Flowchart for Sequential Analysis of Am and Pu in Solids >1 Gram

18.6 Figure No 4: Analysis Flowchart for Sequential Analysis of U and Th in solid or water

19. Method Modifications

19.1 This method for uranium in water is substantially compliant with ASTM Method D-3972-90 and HASL 300 Method U-02 for uranium with the following exceptions:

19.2 ASTM D3972-90 Modifications:

19.2.1 Ammonium iodide or HNO_3/H_2O_2 rinses have been substituted for the HI rinse to strip plutonium from the column prior to eluting uranium.

19.3 HASL U-02 Modifications:

19.3.1 U-02 foresees initial pre-concentration by simple evaporation. This can often lead to analyte loss due to poorly soluble residues. The method has been modified to employ a ferric hydroxide precipitation which both avoids formation of insoluble residues while providing additional decontamination.

19.3.2 Method HASL 300 U-02 specifies the use of AG 1X4 anion exchange resin. PASI utilizes AG 1X8 anion exchange resin due to increased loading capacity as well as enhanced selectivity in the separation of uranium from competing/interfering elements.

19.3.3 Method HASL 300 U-02 indicates the use of 7N HCL solution in the loading of uranium onto the anion exchange column with 1 N HCL as the eventual eluant for uranium. PASI utilizes a 9N HCL for loading and 0.1N HCL for elution to enhance analyte recovery.

19.3.4 Additionally, PASI utilizes intermediate stripping reagents to selectively remove plutonium (9N HCl / 0.05N NH_4I) and neptunium (6N HCl / 0.52N HF), if present in the samples.

19.3.5 Additional rinses have been added to HASL 300 U-02 to provide efficient decontamination from alpha emitting interferences.

19.3.6 Quality Control requirements have been modified to conform to PACE Analytical Services, LLC. QAP requirements and procedures.

19.4 This method for plutonium, americium and curium isotopes in all matrices and uranium in non-drinking water matrices is a PACE Analytical Services, LLC. proprietary method and is based in parts on EPA or other promulgated methods.

20. Revisions

Document No.	Reason for Change	Date
PGH-R-008-6	1. Updated cover sheet to include Periodic Review. Updated Cover Page, Headers and Footers for this revision. Added periodic review signature lines to the cover page. 2. Updated Table of Contents section to include Attachments	31Dec2012

Document No.	Reason for Change	Date
	and Flowcharts. Made TOC updateable and linked to all the primary sections. 3. Updated Flow charts for the current thorium procedure after column separation. 4. Updated flowcharts with "Count to meet MDC and obtain 400 tracer counts", removing any suggested time requirement. 5. Section 12.7.6.6 – added to include addition of HCl to match flowchart directions. 6. Corrected section 12.8.7 to direct to the correct section for Pu analysis. 7. Section 14.4.3– expanded suggested minimum tracer counts requirements. 8. Section 15 – Removed annual MDL study requirement. Not performed for radiochemical methods. 9. Section 17 – Added TNI reference 10. Removed all references allowing approval of a "senior analyst" and replaced with "approval of Department Manager or Manager-specified designee."	
PGH-R-008-7	1. Updated cover page for this revision and to update the copy right footnote. Also updated to include the Methods Referenced 2. Section 1.3 corrected location of Method Deviations section. 3. Section 6: Updated to specify not targeting weights, recording observed measurements, and not removing sample from beakers once transferred from bottle. 4. Section 10: DI reference to ASTM Type II, and SOP reference. 5. Section 12: Updated to specify spiking and tracing preceding all chemical additions beyond initial preservation. 6. Section 17: Updated to include ASTM D 3972-90 and PASI-PGH QAM. 7. Section 19: Updated deviations from HASL 300 U-02 method.	07Nov2013
PGH-R-008-8	1. Annual SOP review and update. 2. Section 2 – Added references to applicable instrument operation SOPs. Included these references where instrumentation is discussed in other areas of the document. 3. Section 8.1.1 – Included pH verification requirements and recording. 4. Section 8.1.4 – Added maximum hold time requirement. 5. Section 9 – Included references to Pace SOP for instrument operations and listed instrumentation. 6. Section 12.1 – Clarified aqueous sample analysis amounts and instructions for diluting samples. 7. Section 12.6 – Urine samples measured with graduated cylinders not weighed. 8. Section 14.5 – Changed to be consistent with other SOPs for discussion of numerical indicator application. 9. Section 14 – Included duplicate requirement for Arizona drinking water samples in applicable sections.	13Jul2014

Document No.	Reason for Change	Date
	10. Section 15 – added CDOC requirements 11. Section 17 – Added applicable instrument SOP references. 12. Reformatted document.	
PGH-R-008-9	1. Removed from section 5.1: Analysts must be trained as radiation workers and personal dosimeter worn. 2. Section 14 modified to add tracer peak quality control requirements for USDOD-related sample analysis. 3. Section 17 modified to add reference to the US DOD/DOE QSM version 5.0.	20Feb15
PGH-R-008-10	1. Annual review and update. 2. Updated section references throughout document. 3. Updated section 12.4.7 to discuss the process for treating highly oxidized solids to improve analyte recovery.	27Jul2015
PGH-R-008-11	1. Periodic review and update. 2. Section 2.3 – All analytes are decay corrected to the supplied collection date and time. 3. Section 3.2 – Removed reference to a Rapid Extraction Method, not performed. 4. Sections 12.6, 12.11, 12.12, 12.13, and 12.14 – Removed the instructions for Urine analysis, and all instructions referring to specialized methods which are not being performed. 5. Section 10 – Removed reagents associated with the removed methods/sections. 6. Section 12.10.3 and 12.10.4 removed since they are not used. 7. Methods 1 and 2 – Removed the “additional” volumes on each step, since various sized columns are not utilized.	15Mar2017
PGH-R-008-12	1. Periodic review and update. 2. Section 4.9- Inserted comment regarding MAPEP soil series interferences and enhanced dissolution process outlined in section 12.11 3. Section 9- Included additional apparatus for performing the enhanced dissolution procedure. 4. Sections 10- Included additional reagents needed for performing enhanced dissolution. 5. Section 12.1 – Removed precipitate rinse using pH 10 DI water. 6. Section 12.4.2.2 – Added due to section 12.11. 7. Section 12.6.6- Changed wording to reflect when it is necessary to perform this step. 8. Section 12.7.4.5, 12.7.5- Adjusted to reflect volumes used for these steps. 9. Section 12.11- Added to provide instruction for performing enhanced dissolution by fusion method for Uranium/plutonium analysis of MAPEP solid samples. 10. Section 17 – Included references for fusion method.	26Sep2017
S-PGH-R-008-rev.13	1. Section 8.1.1.1 samples must be held minimum of 24 hours. 2. Section 2.1 updated to remove biota as a matrix. 3. Sections 8.1.2 and 8.1.3 updated to remove urine as a matrix. 4. Section 11.33 updated to broaden instructions for	08Feb2018

Document No.	Reason for Change	Date
	calibration verifications. 5. Section 12.3 deleted to remove reference to biota samples which are not analyzed by the lab. All subsequent sections moved up in the SOP. 6. Line references updated to adjust for removal of section 12.3. 7. Numerous sections updated to remove reference to urine and biota. 8. Section 12.5.6 and 12.5.7, updated to current practice with explanation. 9. Section 13.2 and 14.1 added to discuss uranium MCL violation determination and actions. 10. Section 14.8.2 updated to include DoD QSM method blank requirements. 11. Section 14.14.3 modified to enhance assessment of failing MS/MSDs.	

Attachment I - Calculations

The **radioactivity concentration** of a sample is calculated according to the following equations:

Eq. 1 $$A = \frac{(S_S / T_s)}{Denom}$$

Eq. 2 $$E_T = R * E$$

Eq. 3 $$E_i = \frac{S_E}{T_E * C_E * F_E * D_E}$$

Eq. 4 *E = average of individual isotope efficiencies*

Eq. 5 $$D = e^{-\lambda t}$$

Eq. 6 $$\lambda = \frac{\ln 2}{T_{1/2}}$$

Eq. 7 $$Denom = E_T * V * 2.22 * D * F$$

Eq. 8 $$R = \frac{(S_T / T_S)}{(E * C)}$$

Where:

A = The radioactivity concentration for the radioisotope being measured in units of pCi per Liter, gram, filter, or sample. ***"Activity."***

S_S = Represents the background corrected net counts for the radioisotope being measured. ***"Peak Net Cts."***

B = Represents the Bkg Cts acquired in the applicable region of interest (ROI) referenced to the sample count time. "***Bkg Cts (ref to Sample ct time)".***

T_S = Represents the count time for the sample. ***"Sample time (min)."***

T_B = Represents the count time for the background. ***"Bkg Time (min)."***

E_T = Represents the total system efficiency for the counted sample. This represents the detector efficiency corrected for chemical recovery. ***"Total Eff."***

V = Represents the sample volume (in liters), mass (in grams), filter portion analyzed (fractional), or sample portion analyzed (fractional). ***"Aliquot."***

2.22 = Represents the factor to convert from disintegrations per minute (dpm) to picocuries (pCi). ***"Act. Conv. From dpm to pCi."***

D = Represents the fraction of analyte remaining after decay time T. ***"Fract. Remain."***

F = Represents the summed branching ratio for all alpha particles emitted in the region of interest. In most cases this is 100% (+/- 1%). If the branching ratio varies significantly (greater than associated uncertainty) from 100%, this correction should be applied. ***"abnd."***

R = Represents the analytical chemical recovery for the tracer analyte. ***"Chemical Yield."***

S_T = Represents the background corrected net tracer peak counts in the applicable region of interest (ROI). Located in the **"Peak Net Cts"** column for the tracer nuclide row.

B_T = Represents the background counts for the tracer analyte acquired in the tracer region of interest (ROI) referenced to the sample count time. Located in the **"Bkg Cts (ref to Sample Ct time)"** column for the tracer nuclide row.

E = Represents the efficiency for the detector used for sample counting. The efficiency is a fixed number for each detector representing the fractional percent of radioactive events that are measured by the counting system. ***"Det. Eff. (cpm/dpm)."***

C = Represents the dpm of tracer (decay corrected to the time of sample counting) added to the sample. ***"Spike dpm."***

$T_{1/2}$ = Represents the half-life of the radionuclide being measured. ***"T1/2 (y)."***

t = Represents the elapsed time between the reference and count dates in the same units as $T_{1/2}$.

The sample specific **counting uncertainty** is calculated as follows.

Eq. 9 $$\textit{Counting Uncertainty} = \frac{1.96 * \sqrt{((S_S/T_S)/(T_S)) + ((B/Ts)/(T_B))}}{\text{Denom}}$$

As summed background and analyte count rates approach zero, assumptions underlying the uncertainty calculation are violated and it will return an unrealistic value of zero (0) uncertainty when zero summed counts are observed. The following equation provides a more accurate estimate of count uncertainty at zero and near-zero count rates.

Eq. 10 $$\text{ZeroUnc} = \frac{1.96 * \sqrt{zaf / T_S / T_S + zaf / T_B / T_B}}{Denom}$$

Where:

zaf = zero activity factor

and T_S, T_B, and Denom were previously defined

Note 1: Depending on sample type and contract requirements the zero activity factor may be either 3.0 or 2.71. PASI's default is 2.71 consistent with the current version of ANSI N42.23. Bioassay samples must be calculated using 3.0 to be consistent with ANSI N13.30.

Note 2: The Zero Count Uncertainty is compared to the count uncertainty above. The larger of the two is used as the counting uncertainty in subsequent total error calculations.

The error term is further evaluated to provide an estimate of total error hereafter referred to as the *Combined Standard Uncertainty* (CSU a.k.a. TPU).

Eq. 11 $$\text{CSU (pCi/unit)} = \sqrt{(\text{CountingUncertainty})^2 + (UE1 * A)^2 + (UE2 * A)^2 + (UE3 * A)^2 + (UE4)^2}$$

UE1, UE2, UE3, and UE4 represent partial derivatives estimating the relative uncertainty at the ***95% confidence interval*** for various factors in the activity calculation as follows:

UE1 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with preparation (e.g., sample aliquot or transfers and splits prior to addition and equilibration of tracer).

UE2 represents combined factors estimating routine maximum relative uncertainty (fractional) associated with analysis (e.g., peak integration, peak overlap, tracer contaminants).

UE3 represents combined factors estimating relative uncertainty (fractional) associated with yield correction (e.g., count uncertainty for tracer peak, SRM known value, tracer volume or mass aliquot, tracer equilibration efficiency).

UE4 represents the factor estimating additional uncertainty (activity) associated with an individual sample -- to be used in exceptional circumstances with approval of the Department Manager or Manager-specified designee and with appropriate documentation and narration only.

The Minimum Detectable Concentration (MDC), Decision Level (DL) activity and Critical Level are calculated per guidance of ANSI N42.23 and N13.30 as:

Eq. 12 $$\mathrm{MDC}=\frac{4.65 * \sqrt{(\mathrm{B/Ts}) * T_S} + ZeroActFact}{\mathrm{Ts} * \mathrm{Denom}}$$

The Critical Level (LC), is calculated per guidance of ANSI N42.23 as:

Eq. 13 $$LC=\frac{1.65 * \sqrt{(\mathrm{B/Ts}) * (1/T_S + 1/T_B)}}{\mathrm{Denom}}$$

Attachment II - (Numerical Performance Indicators)

1. **Method Blank (MB)**

1.1 The numerical performance indicator for the method blank is calculated by:

$$Z_{Blank} = \frac{x}{u(x)}$$

Where:
x = Measured blank activity
u(x) = Combined standard uncertainty (1 sigma) in the blank measurement

1.2 MB performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. MB performance indicator values should be recorded on a control chart.

2. **Laboratory Control Sample (LCS)**

2.1 The numerical performance indicator for a laboratory control sample is calculated by:

$$Z_{LCS} = \frac{x - c}{\sqrt{u^2(x) + u^2(c)}}$$

Where:

x = Analytical result of the LCS
c = Known concentration of the LCS
$u^2(x)$ = Combined standard uncertainty (1 sigma) of the result squared.
$u^2(c)$ = Combined standard uncertainty (1 sigma) of the LCS value squared.

2.2 LCS performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to +2. Performance indicator values should be recorded on a control chart.

3. **Duplicates (DUP)**

3.1 These criteria are applicable for the evaluation of the Duplicate, Matrix Spike Duplicate and Laboratory Control Sample Duplicates.

3.2 The numerical performance indicator for laboratory duplicates is calculated by:

$$Z_{Dup} = \frac{x_1 - x_2}{\sqrt{u^2(x_1) + u^2(x_2)}}$$

Where:
x_1, x_2 = Two measured activity concentrations
$u^2(x_1)$, $u^2(x_2)$ = The combined standard uncertainty (1 sigma) of each measurement squared.

3.3 Duplicate sample performance is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. DUP performance indicator values should be recorded on a control chart for each QC sample type (Dup, MSD, LCSD)

4. **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**

4.1 The numerical performance indicator for a matrix spike sample is calculated by:

$$Z_{MS} = \frac{x - x_0 - c}{\sqrt{u^2(x) + u^2(x_0) + u^2(c)}}$$

Where:
x = measured concentration of the spiked sample
x_0 = measured concentration of the unspiked sample
c = spike concentration added
$u^2(x)$, $u^2(x_0)$, $u^2(c)$ = the squares of the respective combined standard uncertainties (1 sigma) of these values.

4.2 MS performance for all matrices is acceptable when the numerical performance indicator calculation yields a value between –3 and 3. Warning limits have been established as –2 to 2. MS performance indicator values should be recorded on a control chart.

Figure 1. Analysis Flowchart for Sequential Analysis of Am, Pu, U, and Th (Method 2)

BOX 1___Precondition a small anion column with 20mL 8N HNO_3 – discard
___Dissolve sample in 15mL 8N HNO_3 - heat to aid in dissolution as necessary – samples must be clear – centrifuge if suspended solids are present
___Load sample onto conditioned columns – collect in clean c-tubes labeled **ALL**
___Rinse sample c-tube with 15mL 8N HNO_3 and add to columns – collect for **ALL**
___Rinse column with 15mL 8N HNO_3 – collect for **ALL**
___Pour contents of **ALL** c-tubes into clean glass beakers and heat to dryness. **Proceed to Box 3.**
___Place new c-tubes under columns labeled **TH** – elute and collect thorium by adding 25mL 9N HCl to columns. **Proceed to Box 2.**
___Save columns for re-use

BOX 2___Transfer **TH** fraction to new labeled 250mL disposable cup.
___Add 0.80ml of iron carrier and dilute to the 220mL mark with DI water
___Precipitate the thorium as a hydroxide by adding 22mL of NH4OH and stirring with a disposable pipette.
___Stir the samples vigorously after 15 minutes.
___Allow the precipitate to settle for 30 minutes, decant the supernate, and transfer the rest to the original c-tube with pH 10 DI water.
___Centrifuge and discard the supernate.
___Dissolve **TH** residue in 3mL 9N HCl
___Dilute the dissolved **TH** with DI water to a final volume of 25mL
___Microprecip **TH** with Neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 3___Add 15mL conc HCl to beakers labeled **ALL** and heat to dryness
___Dissolve contents of beakers labeled **ALL** in 15mL 9N HCl / 0.1% H_2O_2 – heat to aid in dissolution
___Load samples onto columns previously used – collect in c-tubes labeled **AM**
___Rinse **ALL** beakers with 10mL 9N HCl / 0.1% H_2O_2 and pour on columns – collect for **AM**
___Rinse columns with 20mL 9N HCl – collect for **AM. Proceed to Box 4**
___Place new c-tubes under columns labeled **PU –** elute and collect plutonium by adding 20mL 9N HCl / 0.05N NH_4I. **Proceed to Box 5**
___Rinse columns with 15mL 6N HCl / 0.52N HF – discard to waste
___Rinse columns with 10mL 9N HCl – discard
___Elute uranium into c-tubes labeled **U** by adding 20mL 0.1N HCl
___Microprecip **U** with neodymnium, titanous chloride to persistent purple color, and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 4___Transfer contents of c-tubes labeled **AM** to new glass beakers, add 3 drops Fe carrier and heat to dryness
___Add 5mL conc HNO_3 and heat to dryness
___Prepare Tru resin columns with pre-filter resin on bottom
___Condition columns with 5mL 2N HNO_3 - discard
___Dissolve contents of beaker in 10ml 2N HNO_3 / 0.5N $AlNO_3$
___Add 1 drop KSCN indicator and 6-8 drops of fresh 1N ascorbic acid – samples should be clear
___Pour sample onto columns – discard
___Rinse sample beaker with 5mL 2N HNO_3 – add to column - discard
___Rinse with 5mL 2N HNO_3 - discard
___Rinse with 5mL 2N HNO_3 - discard
___Elute AM into c-tubes with 2mL 9N HCL followed by 10mL 4N HCl
___Transfer to beakers and heat to dryness
___Add 1.0mL formic acid – heat to dryness
___Condition TEVA columns with 5mL fresh 3N Ammonium Thiocyanate / 0.1N Formic acid
___Dissolve samples in 15mL 3N AThio/ 0.1N formic
___Load sample onto columns – discard
___Rinse columns with 2mL 1N AThio/ 0.1N formic – discard
___Rinse with 3mL 1N AThio/ 0.1N formic – discard
___Rinse with 5ml 1N AThio/ 0.1N formic – discard
___Elute **AM** into new c-tubes with 15mL 2N HCl
___Microprecip **AM** with neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 5___Transfer **PU** fraction to new glass beakers
___Add 10mL conc HNO_3 and 3 drops iron carrier
___Heat until reactions subside
___Add H_2O_2 dropwise to destroy NH_4I – about 15 drops
___Heat to dryness
___Add 5mL conc HCl
___Heat to dryness
___Dissolve **PU** residue in 10mL 9N HCl
___Transfer to new c-tubes with DI water to a final volume of 20mL
___Microprecip **PU** with neodymnium, 10-12 drops 25% dihydrazine dihydrochloride, and HF
___Count to meet MDC and obtain 400 tracer counts.

Figure 2: Analysis Flowchart for Sequential Analysis for Am, Pu, U (Water or Solids) Method 1

Box 1___Prepare samples in accordance with the applicable section based on the matrix (12.1 for aqueous samples, 12.4 for solid samples). Solids must undergo complete digestion with HF.
___Condition prepared anion columns with 10mL 9N HCl.
___**Proceed to Box 2.**

BOX 4___Transfer **PU** fraction to new glass beakers
___Add 10mL conc HNO_3 and 3 drops iron carrier
___Heat until reactions subside
___Add H_2O_2 dropwise to destroy NH_4I – about 15 drops
___Heat to dryness
___Add 5mL conc HCl
___Heat to dryness
___Dissolve **PU** residue in 10mL 9N HCl
___Transfer to new c-tubes with DI water to a final volume of 20mL
___Microprecip **PU** with neodymnium, 10-12 drops 25% dihydrazine dihydrochloride, and HF
___Count for to meet MDC and obtain 400 tracer counts.

BOX 2___Dissove sample residue/precipitate in 15mL 9N HCl / 0.1% H_2O_2 – heat to aid in dissolution
___Centrifuge samples if necessary
___Load samples onto columns collect in c-tubes labeled **AM**
___Rinse sample c-tube with 10mL 9N HCl / 0.1% H_2O_2 and pour on columns – collect for **AM**
___Rinse columns with 20mL 9N HCl – collect for **AM.**
Proceed to Box 3
___Place new c-tubes under columns labeled **PU –** elute and collect plutonium by adding 20mL 9N HCl / 0.05N NH_4I. **Proceed to Box 4**
___Rinse columns with 15mL 6N HCl / 0.52N HF – discard to waste
___Rinse columns with 10mL 9N HCl – discard
___Elute uranium into c-tubes labeled **U** by adding 20mL 0.1N HCl
___Microprecip **U** with neodymnium, titanous chloride to persistent purple color, and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 3___Transfer contents of c-tubes labeled **AM** to new glass beakers, add 3 drops Fe carrier and heat to dryness
___Add 5mL conc HNO_3 and heat to dryness
___Prepare Tru resin columns with pre-filter resin on bottom
___Condition columns with 5mL 2N HNO_3 - discard
___Dissolve contents of beaker in 10ml 2N HNO_3 / 0.5N $AlNO_3$
___Add 1 drop KSCN indicator and 6-8 drops of fresh 1N ascorbic acid – samples should be clear
___Pour sample onto columns – discard
___Rinse sample beaker with 5mL 2N HNO_3 – add to column - discard
___Rinse with 5mL 2N HNO_3 - discard
___Rinse with 5mL 2N HNO_3 - discard
___Elute AM into c-tubes with 2mL 9N HCL followed by 10mL 4N HCl
___Microprecip **AM** with neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

Figure 3: Analysis Flowchart for Sequential Analysis of Am and Pu in Solids >1g

Box 1___1. Aliquot 3-4 grams of dry pulverized solid into clean labeled ceramic crucible.
___2. Add 1.0mL of iron carrier. Add tracers to all samples, and spike solution to LCS and MS.
___3. Cover sample with crucible lid and place in oven at 550C overnight (minimum of 4 hours).
___4. Remove samples from oven, re-label, and carefully loosen solid with a disposable pipette.
___5. Transfer loosened solid to a labeled glass beaker with a minimal amount of 8N HNO_3.
___8. Add 5mL 16N HNO_3 and 5mL 12N HCl to the crucible and place on hotplate at medium heat to loosen/remove the remaining solid.
___9. Transfer this solution to the appropriate glass beaker with 9N HCl. Repeat the above step as necessary to remove solid from the crucible.
___10. __ __Add 15mL 16N HNO_3 and 15mL 12N HCl to each sample beaker.
___11. __ __Cover sample with a watch glass and leach for 30 minutes on hotplate at medium heat.
___12. __ __Cool sample and transfer to a centrifuge tube using DI water. Centrifuge the sample.
___13. __ __Transfer the supernate to a clean labeled glass beaker and heat to dryness.
___14. __ __Transfer the solid back to the original beaker with 9N HCl.
___15. Repeat steps 10-14 two additional times adding the supernate from each centrifuge cycle to the beaker in step 13. The solid should be lighter in color indicating complete leaching of iron metal from the sample.
___16. Heat the supernate to dryness.
___17. Add 10mL 12N HCl to each sample beaker and heat to dryness. **Proceed to Box 2.**

BOX 4___Transfer **PU** fraction to new glass beakers
___Add 10mL conc HNO_3 and 3 drops iron carrier
___Heat until reactions subside
___Add H_2O_2 dropwise to destroy NH_4I – about 15 drops
___Heat to dryness
___Add 5mL conc HCl
___Heat to dryness
___Dissolve **PU** residue in 10mL 9N HCl
___Transfer to new c-tubes with DI water to a final volume of 20mL
___Microprecip **PU** with neodymnium, 10-12 drops 25% dihydrazine dihydrochloride, and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 2___Precondition small anion column with 10mL 9N HCl.
___Dissolve contents of beakers in 15mL 9N HCl / 0.1% H_2O_2 – heat to aid in dissolution – centrifuge sample
___Load samples onto columns – collect in c-tubes labeled **AM**
___Rinse beakers/c-tubes with 10mL 9N HCl / 0.1% H_2O_2 and pour on columns – collect for **AM**
___Rinse columns with 20mL 9N HCl – collect for **AM.**
Proceed to Box 3
___Place new c-tubes under columns labeled **PU –** elute and collect plutonium by adding 20mL 9N HCl / 0.05N NH_4I.
Proceed to Box 4
___Rinse columns with 15mL 6N HCl / 0.52N HF – discard to waste
___Rinse columns with 10mL 9N HCl – discard
___Elute uranium into c-tubes labeled **U** by adding 20mL 0.1N HCl
___Microprecip **U** with neodymnium, titanous chloride to persistent purple color, and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 3___Transfer contents of c-tubes labeled **AM** to new glass beakers, add 3 drops Fe carrier and heat to dryness
___Add 5mL conc HNO_3 and heat to dryness
___Prepare Tru resin columns with pre-filter resin on bottom
___Condition columns with 5mL 2N HNO_3 - discard
___Dissolve contents of beaker in 10ml 2N HNO_3 / 0.5N $AlNO_3$
___Add 1 drop KSCN indicator and 6-8 drops of fresh 1N ascorbic acid – samples should be clear
___Pour sample onto columns – discard
___Rinse sample beaker with 5mL 2N HNO_3 – add to column - discard
___Rinse with 5mL 2N HNO_3 - discard
___Rinse with 5mL 2N HNO_3 - discard
___Elute AM into c-tubes with 2mL 9N HCL followed by 10mL 4N HCl
___Transfer to beakers and heat to dryness
___Add 10mL HCl and heat to dryness
___Add 1.0mL formic acid – heat to dryness
___Condition TEVA columns with 5mL fresh 3N Ammonium Thiocyanate / 0.1N Formic acid
___Dissolve samples in 15mL 3N AThio/ 0.1N formic
___Load sample onto columns – discard
___Rinse columns with 2mL 1N AThio/ 0.1N formic – discard
___Rinse with 3mL 1N AThio/ 0.1N formic – discard
___Rinse with 5ml 1N AThio/ 0.1N formic – discard
___Elute **AM** into new c-tubes with 15mL 2N HCl
___Microprecip **AM** with neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

Figure No 4: Analysis Flowchart for Sequential Analysis of U and Th in solid or water utilizing Anion Resin.

BOX 1___Precondition a small anion column with 20mL 8N HNO_3 – discard
___Dissolve sample in 15mL 8N HNO_3 - heat to aid in dissolution as necessary – samples must be clear – centrifuge if suspended solids are present. An extra 5mL of 8N HNO_3 may be used if transferring solids to a centrifuge tube.
___Load sample onto conditioned columns – collect in clean c-tubes labeled **U**
___Rinse sample c-tube with 15mL 8N HNO_3 and add to columns – collect for **U**
___Rinse column with 15mL 8N HNO_3 – collect for **U**
___Pour contents of **U** c-tubes into clean glass beakers and heat to dryness. **Proceed to Box 3.**
___Place new c-tubes under columns labeled **TH** – elute and collect thorium by adding 25mL 9N HCl to columns. **Proceed to Box 2.**
___Save columns for re-use

BOX 2___Transfer **TH** fraction to new labeled 250mL disposable cup.
___Add 0.80ml of iron carrier and dilute to the 220mL mark with DI water
___Precipitate the thorium as a hydroxide by adding 22mL of NH4OH and stirring with a disposable pipette.
___Stir the sample vigorously after 15 minutes.
___Allow the precipitate to settle for 30 minutes, decant the supernate, and transfer the rest to the original c-tube with pH 10 DI water.
___Centrifuge and discard the supernate.
___Dissolve **TH** residue in 3mL 9N HCl
___Dilute the dissolved **TH** with DI water to a final volume of 25mL
___Microprecip **TH** with Neodymnium and HF
___Count to meet MDC and obtain 400 tracer counts.

BOX 3___Add 5mL conc HCl to beakers labeled **U** and heat to dryness
___Dissolve contents of beakers labeled **U** in 15mL 9N HCl– heat to aid in dissolution
___Load samples onto columns previously used
___Rinse **U** beakers with 10mL 9N HCl and pour on columns
___Rinse columns with 20mL 9N HCl
___Rinse columns with 20mL 6N HCl / 0.52N HF – discard to waste
___Rinse columns with 10mL 9N HCl – discard
___Elute uranium into c-tubes labeled **U** by adding 20mL 0.1N HCl
___Microprecip **U** with neodymnium, titanous chloride to persistent purple color, and HF
___Count to meet MDC and obtain 400 tracer counts.

ATTACHMENT C-35

MCL STANDARD OPERATING PROCEDURES

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE		
Project Management: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President	5/2/12 Date
	Quality Assurance Officer	5/1/12 Date

1.0. INTRODUCTION

The purpose of this standard operating procedure (SOP) is to provide guidance for the establishment of a quality project. This SOP discusses project initiation, sample management, project document control, and waste management planning. This SOP applies to all projects unless specifically excluded in a project specific Quality Assurance (QA) Plan or project work plan.

2.0. GENERAL RESPONSIBILITIES

There are four types of responsibilities:

2.1. Project Manager (PM) or Project Leader (PL):

The PM or leader has ultimate responsibility for all project activities. They:

- Review the contract documents, proposal, or statement of work to assure an understanding of the project requirements (personnel, facilities, methods, etc.) to ensure that MCLinc can meet those requirements.
- Have principle authority to issue test reports and provide the technical opinions and interpretations required.
- Identify any differences/issues and resolve with client.
- Appoint a sample custodian and project team, if appropriate.
- Inform the sample custodian and project team of the client's requirements regarding sample and project specific criteria.

- Ensure that all documentation is inspected, complete, traceable, and placed in the project file at the end of the project.
- Ensure that the project team knows all waste generation and disposal requirements.
- Ensure proper final disposition of all project samples.

2.2. Sample Custodian (SC):

The SC is a designated function of the administrative staff. They are trained to properly:

- Receive samples and document receipt.
- Enter samples into sample tracking system.
- Inform P*M* and project team that samples have arrived.
- Ensure that proper final disposition of samples is determined and indicated in the Project File.

2.3. Document Control Coordinator (DCC):

The DCC or designee is responsible for opening and maintaining the business file for each potential project. The *PM* will notify the DCC or designee of the project approval or start-up and then the DCC assigns the project number and creates a project file folder for the *PM*.

2.4. Project Team Member:

Project Team Members are assigned by management to the project as needed. They:

- Keep accurate and complete documentation of laboratory operations concerning the project in an authorized lab notebook.
- Label all project records with the project number, individual conducting work, date, cross-references, and sample numbers and provide records to the *PM*.
- Report and document all non-conformances related to the project to the *PM*.
- Requisition items as necessary to complete the project.

3.0. PROJECT STARTUP

Prior to the actual start of a project the *PM* should compile the project related information. This information is typically collected/generated during the bid/proposal cycle of the project. This information will begin the project documentation file. This information may include: the client request for proposal, MCLinc proposal, QA documents, and statement of work or work plan. The forms and documentation described in this SOP should be completed as necessary and placed in the project file

3.1. Project Overview

Projects that are priced on a time and materials basis (T&M) with greater than 120 hours budgeted or fixed price non-routine projects of greater than $25K budgeted requires the PM to detail the scope of the project at a "Pre-Project Kick-off Meeting" with the MCLinc President, Chief Operating Officer and Project Team Members. For routine projects and projects smaller than defined above, the *PM* will communicate the information informally in writing to the team members. (Using e-mail, hand written instructions or memos so that each member of the team knows exactly their duties and what is expected.)

3.2. Documentation and Validation of Non-standard Methods

If the project requires development or use of a non-standard method, that method must be documented as a MCLinc SOP and validated. The validation as a minimum should include an evaluation of the method against a known traceable standard, or comparison of the method using standards or samples to another standard method, and producing comparable results within the limits required for the project. Records of this validation must be maintained in the project files.

3.3. Other Project Documents

As project documents are received or generated, the DCC or designee and *PM*, will make sure the documents are placed in the business files or the project files. These document types are defined in the SOP MCL-7729, "QA Records."

4.0. SAMPLE MANAGEMENT

4.1. Receipt of Samples

Upon notification of a sample shipment, the PM must inform the SC of the expected delivery date. The PM will inform the SC of any special hazards or handling protocols that must

be used with the samples. (See MCLinc SOP MCL-7756, "Operator Aid," Appendix BB). If the SC is not available at the time of sample receipt another project team member may substitute for the SC.

Upon receipt, all sample or sample packages must be screened for radioactivity (See note below). The packages should be opened within three (3) hours of receipt and the contents inspected. All packages received by MCLinc via common carrier may contain hazardous materials. These packages must be opened in *Sampling Receiving hood* using proper personal protective equipment (PPE) (safety glasses with side shields, gloves, and lab coat). If the sample containers are intact, they can be logged in and processed as required. Samples hand carried into the lab should be in a secondary container (i.e., poly bag, box or cooler, etc.). If not, place in a poly bag prior to log in. If the samples are in sealed secondary containers that require opening to identify the containers should be safely opened as described above. All chain of custody (COC) forms should be checked and completed. Appendix A shows an example sample receipt checklist and assignment of project number that must be completed for COC sample projects unless a client specific document is required. This form is to be filled out prior to the samples being logged into the system.

Make sure there are no reasons to reject the samples, such as: broken/open containers, insufficient sample size, improper collection media, or received at wrong temperature. If any aspect of the sample shipment condition raises concern with the SC, the project leader and the QAM should be notified immediately and the issue resolved prior to sample log-in.

If the sample package is labeled as containing radioactive materials, packages should be rad screened and opened in a radiological buffer area. The external surfaces of the package shall be monitored within three (3) hours of receipt of the package or, if received during nonworking hours, no later than three (3) hours after the beginning of the next working day. Notify the Radiological Safety Officer if contamination is found in the package or if the radiation level exceeds the background. See Appendix *1 MCL-7775* for further information on receipt of radiological samples.

After the samples have been received and verified including samples received as non-COC, the samples should be logged into the sample database. Appendix B is a sample log-in template showing the information that can be entered into the sample database. After all of the sample information has been entered, print out the inventory of samples. Verify data entry on the print out. Label all samples with proper sample ID number. The SC will inform the project team of sample receipt.

Note 1 For sample *receipt* radiological screening, use a portable survey instrument that is annually third party calibrated. Check instrument daily using a Coleman Lantern Mantle source. Check battery and instrument response to the source (alarm generated). Record the daily checks on data sheet in green notebook on sample cart.

CAUTION: **All samples received suspected or marked containing beryllium, beryllium oxide and asbestos, shall only be opened or removed from secondary container in an operating laboratory hood by an experienced analyst.**

4.2. Chain of Custody

Depending upon the type of project or client MCLinc may receive samples that are under a Chain of Custody (COC). The COC provided by MCLinc is shown as Appendix *C**; however, MCLinc may receive other forms specific to a client. In all COC cases, MCLinc keeps the **original COC form.** Chain of custody (COC) samples will be maintained within the MCLinc facility. No internal chain of custody is required while the samples are under MCLinc control. Whenever the sample control is transferred to non-MCLinc personnel, the COC form must reflect the transfer and acceptance of the sample. *Use only the numbered copies of the MCLinc COC Form (Appendix *C*) which are available at the sample receiving cart.

COC samples are considered controlled when at least one of the following is true:

- Samples are maintained within the controlled - access MCLinc facility.
- Samples are locked in a storage cabinet outside the building.
- Samples are under direct supervision of a MCLinc employee, or
- Samples are contained inside of an instrument.

4.3. Sample Storage

Samples routinely will be stored in accordance with the customer objectives. Any samples for volatiles analysis shall be stored in a refrigerator free of chemical and with a refrigerator blank (DI Water in 40ml VOA vial). The following are general guidelines, which should be followed for other sample types within the MCLinc facility.

4.3.1. Classified Samples

Classified samples are stored in the Classified Repository in the Limited Area. These samples will be handled in accordance to the Facility Security Plan (MCL-7706).

4.3.2 Radiological Samples

When not in use, the samples will be stored in an appropriate sample storage cabinet in C-corridor. Samples may be stored in the laboratory during use but caution must be used to ensure that any COC and QA requirements are met at all times. Storage and handling requirements of the MCLinc "Radiological Protection Plan" (MCL-7715) must be followed.

4.4. Sample Identification

The sample will be marked with the MCLinc project number and sample number generated during sample login. See MCLinc SOP MCL-7756, "Operator Aids," Appendix O for further

discussion of sample receiving procedures.

4.5. Sample Handling

If not known otherwise, samples, sub-samples, and treatment residuals should be handled as potentially hazardous using the minimum personal protective equipment according to the "Chemical Hygiene Plan" (MCL-7702). The project specific sample and QA guidance, and all applicable MCLinc requirements and related regulations must be met.

4.5.1 Receipt of Soil from Foreign and Domestic Sources

Soil received from a foreign or domestic sources for chemical or physical analysis falls under the United States Department of Agricultures, Animal and Plant Health Inspection Service (APHIS) Permit to Receive Soil for MCLinc. All regulatory and specifically listed permit conditions apply (See MCLinc's Permit to Receive Soil for sample labeling, storage, and disposal). The APHIS Permit is located in the QA file.

4.5.2 APHIS Sample Disposal

After analysis is complete and the report has been submitted and reviewed, samples may be returned to the client, used up during analysis, or sterilized using the techniques listed in the APHIS permit. Samples returned to the client will follow all shipping requirements including the requirements in the APHIS permit. If the sample is sterilized, a memo to the file will state the material was sterilized.

4.6. Sample Return

The customer should be notified for return of samples and residuals (if applicable) at the closure of the project. The *PM* is responsible for all samples relating to their projects.

4.7. Sample Archival

With customer approval, a sample and related residuals can be placed into MCLinc archives. The sample should be permanently marked, protected from cross-contamination, and stored in an appropriate sample archival area.

4.8. Disposal of Samples and Related Residuals

Upon completion of the project, samples designated for disposal by MCLinc will be processed per SOP "Waste Management Plan," MCL-7718.

5.0. DOCUMENTATION

The use of the project number and sample numbers will provide a consistent means of tracking project activities and ensuring that all QA objectives are traceable.

5.1. Lab Notebooks

Laboratory notebooks are controlled documents and will be used by all personnel working on a project. Laboratory notebooks should contain legible, clear, concise information describing all aspects of work performed. References should be made to any instrument logbook or other source of QA documentation which would be considered part of the project's overall performance factors. The lab notebook is the sole responsibility of each individual project team member and is to maintain per SOP MCL-7724, "Good Notebook Keeping Practices."

5.2. Raw Data

All raw data will be clearly marked and/or logged to provide direct linkage to the project. The MCLinc sample number should always be referenced at a minimum. The lab notebook and/or the instrument logbook should provide a clear description of operating parameters, associated QA references, sample preparation methods, file/data storage locations. A copy of the raw data should be provided to the PM for review and inclusion in the project files. (Refer to Note above)

5.3. Report Data

All data collected during the analyses, calculated or graphically formatted (i.e., put into a more acceptable, report-style, format) must be documented as instrumental print out or referenced in the lab notebook. The file name(s) and location should be clearly documented. An original copy should be provided to the PM. (Refer to Note)

5.4. Final Report

All final reports must be released to the client through the Office Manager for formality check and proper record keeping.

The final report should be reviewed by at least one other person, other than the primary author(s), prior to being given to the DCC. A copy of the full final report, identical to the customer's version, should be labeled as such and placed in the project files. Any changes or additions to the final report must be clearly marked as an addendum or revision to the original report.

5.5. Customer Feedback

Any comments, questions, or follow-up by the customer through out the duration of the project should be documented in the lab notebook. A summary of the customer feedback should be compiled and placed in the project file. Any client complaints must be addressed and documented.

A customer feedback survey will be administered on an annual basis. See Appendix D for the customer feedback form. All forms will be compiled in the QA files. If there are negative

feedback comments, they will be handled via the Non-conformance SOP MCL-7722.

Note 1: Electronic files should be backed up either with a paper copy or an electronic copy, i.e., CD or floppy disk. Electronically transmitted data must be checked prior to sending and be sent in an electronic file, i.e., Word, Excel, etc. These files must be protected from unauthorized access or improper amendment per MCL-7728 Verification of Data Software.

Appendix A - Sample Receipt Checklist

MCLinc Project Number Assignment

SOW #:____________P.O. #:____________MCLinc Project #_____________________

Date Samples Received:____

Client:____

Sample(s)
Received From:___________________________ Received By:_________________________

Phone:________________________________ Fax: ____________________________

# of Samples:________________ Turnaround Time:_____________________________

____Routine (7 working days)
____Rush (2 working days)
____Priority (1 working day)

Analysis (Method):____

____

Special Handling:____

Project Leader: __________________Team:___________________________________

Sample is:

____Not Flammable	____Not Toxic	____Not Radioactive	Other	____Non-Hazardous
____Suspect Flammable	____Suspect Toxic	____PIC		
____Known Flammable	____Known Toxic	____Suspect Radioactive	____________	
		____Known Radioactive		

◊◊

1. Shipping Bill Present? ☐ Yes ☐ No ☐ N/A Shipping Bill No.________________
2. Client Chain-of-Custody present? ☐ Yes ☐ No ☐ N/A Chain of Custody No.______________
3. Shipping container intact? ☐ Yes ☐ No ☐ N/A Container Type________________
4. Custody seals present? ☐ Yes ☐ No ☐ N/A Seals intact? ☐ Yes ☐ No
   Seal location:________________
5. Client C-O-C properly filled out, signed, and dated? ☐ Yes ☐ No ☐ N/A
6. Does Client C-O-C indicate sample type? ☐ Yes ☐ No ☐ N/A
7. Are all samples properly labeled and accounted for? ☐ Yes ☐ No ☐ N/A
8. Were samples preserved? ☐ Yes ☐ No ☐ N/A
9. Was preservation checked? ☐ Yes ☐ No ☐ N/A
10. Radiological check? ☐ Yes ☐ No ☐ N/A
11. Sample Condition Upon Receipt: ☐ Okay ☐ Other:_______________________
12. Sample storage location ____

Appendix B- Sample Login Template

Materials and Chemistry Laboratory Sample Tracking Sheet

Project Information

Project Number:

Project Leader:

Project Information Input By:

Client/Contact:

Sample Received From:

Telephone:

FAX:

Sample Information

MCLinc Sample Number:

Customer Sample Number:

Sample Information Input By

Date Received:

Composition/Description:

Date Requested:

Date Completed:

Hazards:

Final Disposition:

Sample Location:

Archive Number:

Source:

Requested Analysis

Requested Analysis:

Q/A Plan:

Comments:

Appendix C – Chain of Custody (COC)

Materials and Chemistry Laboratory, Inc.

Chain of Custody/Request for Analysis

MCLinc COC No. 810

Sample ID	Description (Matrix/Container)	Analysis Requested	Customer Information
			Company:
			Report To:
			Address:
			Phone:
			Fax:
			Project:
			Billing:
			PO #
			Required Turnaround Time
			☐ Routine (7 Working Days)
			Other:
			Known or Suspected Hazards
			☐ Radioactive ☐ Asbestos
			☐ Toxic ☐ Flammable
			☐ Nonhazardous
			Other:
Relinquished by:	**Date:**	**Time:**	**Relinquished by:**	**Date:**	**Time:**

Appendix D – Customer Feedback Form

Materials and Chemistry Laboratory, Inc. (MCLinc) is dedicated to providing you with the best laboratory services possible. MCLinc would like to make sure you are getting the quality of service you deserve, so in order for us to continue to improve our services we would like to know how we are doing. Please take a moment to answer this brief survey. If your answer to questions 1-4 is no, please explain in question 5.

1. Was the quality of work what you expected? ☐ yes ☐ no

2. Was our staff professional and courteous? ☐ yes ☐ no

3. Was the turnaround time prompt and as scheduled? ☐ yes ☐ no

4. Did we accommodate all of your needs? ☐ yes ☐ no

5. Do you have any questions or comments on how we can improve and serve you better?

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE	
Operation Guidance Electron Microscopy: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President — Date: 2/17/2009 Quality Assurance Officer — Date: 2/16/09

1.0 INTRODUCTION

This document and the documents referenced herein provide a framework for the safe and consistent operation of electron microscopes. It is accepted that operating personnel have an understanding of the instrumentation and theory of operation. This guideline will identify the hazards associated with the operation and ensure the safe usage along with providing a high level of confidence in the results obtained.

2.0 GENERAL RESPONSIBILITIES

2.1 Principle Operator

The Principle Operator is responsible for the routine operation, upkeep of the instrumentation, documentation, and work area associated with the instrumentation. The appointment of the Principle Operator for each instrument is made by the *Chief Operating Officer.*

2.2 Secondary Operator

The Secondary Operator should be able to assist the Primary Operator in routine operation and maintenance. The Secondary Operator may be able to perform all operations at the same level of expertise as the Primary Operator, but this is not a requirement. Secondary Operators may be certified by either the *Chief Operating Officer* or the Principle Operator.

2.3 *Chief Operating Officer*

The *Chief Operating Officer* represents the first level of line management which is responsible for supplying the resources for proper upkeep of the required instrumentation.

3.0 EQUIPMENT AND MATERIALS

3.1 Major Components

This table lists the major equipment covered by this guideline. The property number is the property number associated with the main instrument component. It is recognized that additional property numbers may exist for accessories and other secondary components.

Manufacture	Model #	Property #	Room #
Hitachi	S-4500	K333391	A104
JEOL	JXA-840	K322408	A106
JEOL	2000FX	K331267	E101
Hitachi	S-5000	K333393	E102
FEI	ESEM-2020	K333395	E103

3.2 Basic Process Description

Electron microscopy (EM) impinges a focused electron beam on a solid surface to produce electron *images* and x-rays which contain information about the sample. The electrons are used to create electron micrographs (images) and the x-rays are used to obtain elemental information about the sample *with associated x-ray analyzers*. EMs vary by the nature and relative position of their electron optic components with respect to the sample. EMs can optimize various electron-sample interactions (i.e. scanning, transmission, and diffraction) to obtain various types of materials characterization. The "output" is typically an electron micrograph *from a secondary electron detector (SEI), backscattered electron detector (BEI), or transmitted onto a fluorescent screen*, electron diffractogram, or elemental *composition by x-ray spectroscopy* (qualitative or quantitative). The following are brief overviews of typical operational aspects of the instrumentation:

The electron guns operate at very high voltage (1,000 to 200,000 volts) but at very low current (nA to pA range). EMs operate in a vacuum with the electron gun typically being at 10^{-6} to 10^{-7} torr and the sample being between 50 and 10^{-5} torr; hence, sample exchange and manipulation are done via sample exchange interlocks and mechanical stages.

Each instrument has associated equipment required for the vacuum system, cooling, and valving (compressors). Operators are required to understand the interaction of each component and perform routine, preventive maintenance on each component according to the vendor operating manual.

Each scope has an associated x-ray analyzer used to determine elemental composition. X-ray emission is shielded by the metal construction of each instrument.

3.3 *Basic Operating Process*

This describes the *general* guidelines for *sample preparation, instrument operation, and collecting & transferring data for interpretation.*

3.3.1 *Sample Preparation*

Sample preparation for SEM and TEM investigation is the key for a successful investigation. The following notes should be considered prior to loading a sample into the electron scopes:

SEM: Loose powders are not acceptable in the SEMs. SEM preparations are typically mounted on adhesive carbon tape on top of graphite planchets (ie. Ted Pella, Inc.). Because of sample charging, samples are typically carbon coated to reduce the effect of charging on the images. Feature mapping under a stereoscope prior to analysis is strongly recommended to help navigate on the sample at the higher SEM magnifications.

TEM: Loose powders are not acceptable in the TEM. TEM copper grids with a Formvar film layer can be purchased from Ted Pella, Inc. Three microliter samples can be mounted directly on these grids, dried, and loaded into the TEM. A dispersion in ethanol with gentle sonication works well. The technique for preparing a TEM grid for NIOSH 7402 is outlined in the NIOSH 7402 procedure and MCLinc SOP 7742.

3.3.2 *Instrument Operation*

Each instrument has a unique start-up/shutdown procedure outlined in each vendor manual. Instruments must be operated according to the vendor operating manual which outlines procedures for loading/unloading samples, operation, data collection, maintenance, and troubleshooting.

Note that the EMs should never be left unattended when the electron source is activated. When not in use, the EMs should be left in the shutdown condition outlined by the principle operator.

3.3.3 *SEM Data Collection and Transfer*

The SEMs can collect electron images from 20x to 1,000,000x magnification. Both SEI and BEI images can be collected and stored. Images can be transferred for reporting by:

- Polaroid film: Each SEM unit has been set up to collect images by Polaroid type 52 or 57 land film. Film development takes less than 1 minute and has excellent resolution.

- Printer: Each associated x-ray analyzer has the capability to grab the image from the SEM's CRT and print to a printer. The image can then be scanned and converted to a electronic data file.

- Electronic Data File: The Hitachi 4500 has an EDAX x-ray analyzer that is capable of storing and saving images in various formats including bmp, tif, and jpg formats. Data is readily transferred by memory card or CD.

X-ray spectra can be transferred for reporting by:

- Printer: Each associated x-ray analyzer has the capability to print to a printer. The spectra can then be scanned and converted to a electronic data file.

- Electronic Data File: The Hitachi 4500 has an EDAX x-ray analyzer that is capable of storing and saving spectra in various formats including bmp, tif, and jpg formats. Data is readily transferred by memory card or CD.

3.3.4 TEM Data Collection and Transfer

The JEOL 2000FX TEM can collect electron images from 20x to 1,000,000x magnification. Images can be collected and stored only by Kodak film. Follow manufacturer's instructions for using Kodak D-19 Developer and Kodak Rapid Fixer.

X-ray spectra can be transferred for reporting by a printer associated with the x-ray analyzer. The spectra can then be scanned and converted to a electronic data file.

3.4 Laboratory Supplies

This non-inclusive listing provides a baseline for the types of supplies as well as engineering and administrative controls that should be available, as needed, to ensure a safe (personnel and environmental) work place.

Disposable lint-free or powder-free gloves
Lint-free cloths
Disposable laboratory waste bags
Fume hoods equipped to provide a well-ventilated workspace
Protective eyewear
Protective laboratory coat/apron
Spill cleanup material
Emergency eyewash station
Emergency shower station
Fire extinguisher
Access to MSDS sheets for all chemicals used

Ted Pella, Inc and SPI, Inc. are good sources for various EM supplies for sample preparation such as TEM grids and graphite planchets.

3.5 Standards

The following components, or equivalent ones, should be available for quality control and performance evaluation of the various electron microscopes. The selection and use of the particular standard is based upon operator preference. The standard used should be documented in the appropriate logbook and should be used in agreement with the methods outlined in this document.

Magnification standards:

- NIST 484A Specimen ID JY-55-OJ (2 each)
- NIST 484E Specimen ID-SH
- 2160 lines per millimeter cross grating (E. F. Fullam, Inc., Cat. #60021)

Elemental standards:

- C. M. Taylor Corp. #1 Element STD 202-52
- C. M. Taylor Corp. #2 Element STD 202-52
- C. M. Taylor Corp. #4 Element STD 230-27
- C. M. Taylor Corp. #5 Element STD 230-30
- SPI STD 87-103
- Tousimis 8026 103-S

X-ray performance (FWHM) standards:

- X-checker, Small World (#1)
- X-checker, Small World (#2)
- C. M. Taylor Corp. #1 Element STD 202-52

Resolution standards:

- Prickly gold grid Type D

These standards are centrally located, in dry boxes where applicable. Control is maintained through storage in manufacturers labeled containers or in labeled sample storage containers. The standard certification papers are filed with the QA Officer.

4.0 SAFETY PRECAUTIONS

4.1 General Laboratory Safety

Follow guidance outlined in the Chemical Hygiene Plan for the Materials and Chemistry

Laboratory, Inc. (MCL-7702) and the Quality Assurance Plan to the Materials and Chemistry Laboratory, Inc. (MCL-7701).

Develop and encourage safe laboratory habits.
Food will not be stored or consumed in lab areas.
All work areas are to be kept clean and uncluttered.
Safety glasses are required to be worn as posted.
The appropriate personal protective equipment must be worn when required by the job.
Report accidents and near-miss accidents to your supervisor.
On-the-job injuries must be reported immediately.

4.2 Specific Hazards

The electron guns operate at very high voltage (1,000 to 200,000 volts). When changing a filament or performing maintenance, the vendor operating procedure must be followed exactly to prevent high voltage exposure.

For specific hazards of the instruments see the operator's manual and MCL-7717 for Health and Safety approaches to handling the hazards properly. Do not operate unless you understand potential hazards involved with the instrument.

4.3 Emergency Shutdown

The safest, most direct method of shutting the instrument off should be posted in clear plain sight on the front of the instrument. The instructions should be in large print, signed, dated, and laminated.

5.0 ENVIRONMENTAL AND WASTE MANAGEMENT CONCERNS

5.1 Waste Minimization Methods

Kodak Rapid Fixer - Used fixer will be sent out for resource recovery of silver.
Polaroid Film Packs - Digital images will minimize film waste.

Sample Preparation - Use of smallest possible beaker or test tube for cleaning samples or equipment (e.g. tweezers, spatula). Use only a portion of a paper towel or wipe as needed.

Reuse sample planchets by using small amount of double sticky carbon tape. The carbon tape and sample can be peeled off after the analysis and disposed of as solid waste. The planchet can then be reused to mount samples without being added into the waste stream.

5.2 Waste Disposal Methods

All RCRA/TSCA/RAD waste generated by this process shall be disposed of in accordance with the MCLinc Waste Management Plan MCL-7718.

5.3 Environmental Risks

Routine operation of this equipment poses no environmental risks.

6.0 QUALITY AND PERFORMANCE DOCUMENTATION

6.1 Quality Assurance Documentation

The following information should be documented at a minimum of the time period stated and after maintenance activities have been performed. This information will provide direct documentation of the performance (calibration) parameters affecting the quality of the output (results) of the instrumentation. Documentation is the responsibility of the Principle Operator and will be kept with the instrument.

Image magnification (at least semiannually): A determination of the magnification of applicable image source(s) shall be performed.

Energy calibration (at least semiannually): The energy calibration shall be checked. Standards such as Cu and/or Al should be used.

EDS energy resolution - FWHM (at least semiannually): A Mn Ka peak shall be used to measure the full width at half maximum peak intensity (FWHM).

WDS performance (as needed): The position and FWHM of peaks of interest will be documented.

Performance Documentation

The following information shall be documented in the time period stated.

Instrument usage (every time): Logbooks shall be kept for each EM to record instrument usage, operator and project number.

Scheduled instrument maintenance (per event): A copy of the paper work provided by the service provider should be kept in chronological order. Any information or work which has been provided in response to questions or operational abnormalities that is not clearly documented in the paperwork should be documented and attached.

Non-scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or system work which is not clearly documented on the vendor's paperwork or work instructions provided over the telephone should be documented.

Instrument calibration non-conformance (per event): The actions required to bring the instrument back into compliance with operating specifications as noted in section 6.1 should be documented.

6.3 Vendor Manuals

Vendor manuals form the basis of documentation for operating information. These manuals in combination with vendor/professional training and on-the-job training should allow the principle operator to safely, properly, and fully operate the instrumentation.

Vendor manuals shall be readily available during instrument operation.

6.4 Data Tracking

Data documentation and archival information is the responsibility of the originator and should be recorded in the laboratory notebook.

7.0 REFERENCES

MCLinc *Chemical Hygiene Plan* (MCL-7702)

MCLinc *Quality Assurance Plan* (MCL-7701)

MCLinc *Waste Management Plan* (MCL-7718)

Materials and Chemistry Laboratory, Inc. Standard Operating Procedure		
Operation Guide X-Ray Diffraction: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President	1/4/17 Date
	Quality Assurance Officer	1/3/17 Date

1.0 PURPOSE

This document and the documents referenced herein provide a framework for the safe and consistent operation of the x-ray diffractometer (XRD). It is accepted that operating personnel have an understanding of the instrumentation and theory of operation. This guideline will identify the hazards associated with the operation and ensure the safe usage of the instrumentation. This guideline will provide a high level of confidence in the results obtained and provide the foundation for quality control and quality assurance. *Details for analyzing samples are presented in Operator Aid 35 in MCL-7775 Standard Operating Procedure.*

2.0 ROLES AND REFERENCES

2.1. Responsibilities

2.1.1. Principle Operator

The principle operator is responsible for the routine operation, upkeep of the instrumentation, and work area associated with the instrumentation. The appointment of the principle operator for each instrument is made by the *Laboratory Manager*.

2.1.2. Secondary Operator

The secondary operator should be able to assist the primary operator in routine operation and maintenance. The secondary operator may be able to perform all operations at the same level of expertise as the primary operator, but this is not a requirement. Secondary operators may be certified by either the *Laboratory Manager* or the principle operator.

2.1.3. Laboratory Manager

The *Laboratory Manager* represents the first level of line management which is responsible for supplying the resources for proper upkeep of the required instrumentation.

3.0 EQUIPMENT AND MATERIALS

3.1. Major Components

This table lists the major equipment covered by this guideline. The property number is the manufacturer's serial number associated with the main instrument component. It is recognized that additional property numbers may exist for accessories and other secondary components.

Manufacture	Model #	Property #	Room #	Principle Operator	Secondary Operator
Rigaku	MiniFlex II	GD40045	D103	M.R. Colberg	A.B. Dunaway

3.2. Basic Process Description

X-ray diffraction measures the intensity of x-rays (i.e. Cu Kα) that diffract off a powder sample at discrete angles. The relative angle-intensity relationship provides crystallographic information about the sample. The diffraction pattern serves as a "fingerprint" of the phases of crystalline species present. The following is a brief overview of typical operational aspects of the instrumentation:

- The water cooled x-ray tube operates at high power (2000 watts maximum). Typical operating conditions are 30 kV and 15 mA (e.g. 1050 watts).
- XRD operates at atmospheric conditions.
- X-ray yield is contained/shielded by the instrument.

3.3. Laboratory Supplies

This non-inclusive listing provides a baseline for the types of supplies as well as engineering and administrative controls that should be available, as needed, to ensure a safe (personnel and environmental) work place.

- Disposable gloves
- Disposable laboratory waste bags
- Protective eye wear (during maintenance)
- Spill cleanup material
- Emergency eyewash station
- Emergency shower station
- Fire extinguisher
- Access to MSDS sheets for all chemicals used

-

3.4. Standards

The following components should be available for quality control and performance evaluation of XRD. The selection and use of this particular standard is based upon operator preference. The standard used should be documented in the appropriate logbook.

- Position/Intensity/Resolution Standard: Quartz (Novaculite) (Supplied by Rigaku)

4.0 SAFETY PRECAUTIONS

4.1. General Laboratory Safety

- Abide all guidance outlined in the Chemical Hygiene Plan (MCL-7702, *current revision*) and the Quality Assurance Plan (MCL-7701, *current revision*).
- Develop and encourage safe laboratory habits.
- Food will not be stored or consumed in lab areas.
- All work areas are to be kept clean and uncluttered.
- Safety glasses are required to be worn as posted.
- The appropriate personal protective equipment must be worn when required by the job.
- Report all accidents and near-miss accidents to your supervisor.
- All on-the-job injuries must be reported immediately.

4.2. Specific Hazards

These hazards have been identified by the MCLinc. The following sections will list the hazard, its description, what the possible consequences are, and what controls are in place to mitigate the hazard. It is believed that the controls in place are adequate for all hazards identified in this section.

4.2.1. High Voltage

High voltage is required for instrument operations. Exposure to a high voltage source could lead to electric shock of personnel, destruction of equipment, and possible fire. This hazard is controlled by engineering and administrative controls. Engineering controls exist since these instruments have been manufactured to meet all safety and electrical codes. These instruments provide various safety interlocks to ensure that all sources of high voltage are properly shielded and that unintentional contact with high voltage sources is not possible. Administrative control exists since maintenance of the equipment is covered by maintenance agreements with the vendor. This provides highly specialized and skilled personnel to perform all necessary maintenance. These maintenance sub-contractors are also monitored and made to comply with all MCLinc safety rules and regulations. It is recognized that the highest risk from the high voltage can occur during non-routine operational conditions. These conditions are when a water leak is present at or near the instrument. Sources of water leaks can be water-cooling lines for the x-ray source or from drainage from the piping located above the ceiling panel in the room. Whenever uncontained water is detected all operations should cease. This off-normal incident should be reported to the MCLinc *Laboratory* Manager.

4.2.2. Off-shift Operations

Laboratory work being performed outside of normal shift may create a situation where backup support or help is not immediately available. This may lead to a lack or delay of emergency notification in case of an accident. This hazard is controlled by engineering controls and administrative controls. Engineering controls such as emergency pull boxes, telephones, fire sprinkler system, fire extinguisher, building public address system can be used to notify others that of an off-normal situation. Administrative controls exist in that personnel are required to notify the PSS office (574-3282) if they will be occupying laboratory or office facilities during off-shift hours. This notification will help support the emergency response personnel in the event of an off-normal event. The performance of new (i.e., first time) activities is not permitted during off-shift hours. These controls and the use of training and on-the-job experience mitigate the hazards associated with this scenario.

4.2.3. Radioactive Materials

The possibility exists that the samples that are being analyzed will be radioactive. (See MCL-7710 for guidance on sample preparation.) Handling radioactive materials could lead to personnel exposure and/or contamination of equipment/property. This hazard is controlled by engineering, administrative and PPE controls. The engineering controls are the methods by which the samples are prepared. (All RAD samples are prepared in a radiological area and are surveyed prior to removal from the area.) The sample is firmly secured onto the sample platform. For proper analysis the sample has to be stable and firmly in place. The administrative controls are enacted by the HP organization. The HP technician must first establish a radioactive materials storage area (RMSA). This involves the survey of the instrument before and after the sample has been analyzed and the surrounding area. This HP support ensures that radiological material has not come loose during the analysis. The PPE containment involves the use of a

sample transfer box from the radiological area to the temporary RMSA at the instrument, the use of gloves while handling the sample, the skirting of the instrument area with yellow (RAD) plastic or tyvek, and radiological disposal of all PPE and lab supplies used in the RMSA.

4.2.4. Ionizing Radiation

This hazard recognizes the fact that x-rays are produced by the x-ray tube. These x-rays could be a potential source for personnel exposure. This hazard is mitigated by engineering controls. The basic requirements for the production of x-rays require the safety interlock system of the instrument to be operational. This instrument is surveyed for x-ray leakage. This means that it is not possible for a person to place their hand in, at, or near the source of x-ray production. It is also recognized that the source of the ionizing radiation can be totally removed by shutting off the x-ray gun.

4.3. Classified Work

Follow all guidance provided in the MCLinc Facility Security Plan (MCL-7706) for performing classified work.

4.4. Emergency Shutdown

The safest, most direct method of shutting the instrument off should be posted in clear plain sight on the front of the instrument. The instructions should be in large print and laminated.

5.0 ENVIRONMENTAL AND WASTE MANAGEMENT CONCERNS

5.1. Waste Minimization Methods

Sample preparation — use of smallest possible beaker or test tube for cleaning samples or equipment (e.g., tweezers, spatula). Use only a portion of a paper towel or wipe as needed.

5.2. Waste Disposal Methods

All RCRA/TSCA/RAD waste generated by this process shall be disposed of in accordance with MCLinc policies (MCLinc Chemical Hygiene Plan, MCL-7702).

5.3. Environmental Risks

No appreciable environmental risks are noted at this time for XRD operation.

6.0 QUALITY AND PERFORMANCE DOCUMENTATION

6.1. Quality Assurance Documentation

The following information shall be documented in the time period stated. This information will provide direct documentation of the performance (calibration) parameters affecting the quality of the output (results) of the instrumentation. The documents resulting from these QA procedures will be kept in Room A108.

Diffraction Calibration (monthly): The three most intense peaks of the quartz (novaculite) standard will be used to track position and linearity of the goniometer. Here, the difference between the measured 2-theta positions for the quartz (novaculite) standard are compared to a standard quartz diffractogram (ICDD 46-1045). The differences are tracked over time. A regression analysis is performed on the resulting data. A more detailed discussion can be found in Appendix A.

Diffraction Resolution (monthly): A plot of the degrees two theta range from 65 to 70 will be observed for the split of the five peaks that make up this region of the quartz standard diffractogram. An example of this region is in Appendix A.

Detector Performance - (monthly): The measured intensities (in counts per second) of the three most intense peaks of the quartz (novaculite) standard are tracked over time. A regression analysis is performed on the resulting data as a monitor of intensity drift. An example of this tracking is in Appendix A.

6.2. Performance Documentation

The following information shall be documented on the time period stated and after shutdown periods and maintenance. This information will document the scheduled maintenance, non-scheduled maintenance, and root cause for the instrumental non-conformance. The documents resulting from these performance procedures will be kept in room A108.

Scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or work which has been provided by the vendor in response to questions or operational abnormalities that is not clearly documented in the vendor's paperwork should be documented and attached to the vendor's paperwork.

Non-scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or system work which is not clearly documented on the vendor's paperwork or work instructions provided over the telephone should be documented.

Instrument calibration non-conformance (per event): The actions required to bring the instrument back into compliance with operating specifications as noted in Section 6.1 should be documented.

6.3. Vendor Manuals

Vendor manuals form the basis of the documentation for operating information. These manuals in combination with vendor/professional training and on-the-job training should allow the principle operator to safely, properly, and fully operate the instrumentation.

Vendor manuals shall be kept in good condition and be readily available during instrument operation.

6.4. Data Tracking

Diffraction patterns collected should be stored on disk in the raw data format. All diffraction patterns should be given a unique filename. The file name should be logged with information concerning the sample ID number, the operating conditions, the disk storage ID number, and the date.

7.0 REFERENCES

MCLinc *Chemical Hygiene Plan* (MCL-7702)

MCLinc *Quality Assurance* (MCL-7701)

MCLinc *Sample Preparation Guide* (MCL-7710)

MCLinc Operator Aids: SOP#2 (MCL-7775)

Appendix A
Quality Assurance Documentation

Quality assurance documentation for the XRD is obtained on a monthly basis. The quartz (Novaculite) standard from Rigaku should be run at 30 kV and 15 mA. The program used to do the standard run covers the range 5 to 85 degrees two theta in 0.025 degree steps with a scan speed of 1.0° per minute. The resulting diffraction data is converted into an ASCII format and loaded into the XPowder software for analysis. Peak positions and intensities for the three most intense quartz reflections (see below) are measured manually on "zoomed" peaks. Peak positions are compared a standard quartz diffractogram (ICDD 46-1045) from the International Center for Diffraction Data (ICDD) PDF-2 database. The plots of the diffractogram and the computer printout are archived in Room A108. Subsequent computer analysis of the data is done using Microsoft Excel and a scientific graphics software package (SciDavis).

There are three areas of calibration interest. The first of these is the two theta position of the diffractometer. To evaluate this parameter of XRD operation, the positions of the three most intense reflections from a standard quartz pattern (ICDD 46-1045) are compared to the corresponding peaks in the diffractogram determined from the Novaculite standard. 2θ, d-spacing, and intensity data for the three peaks from the standard pattern are listed in table 1.

Table 1: Diffraction data for the three most intense reflections in ICDD 46-1045).

Peak	2θ	d-spacing	Relative intensity (I/I_{100})
1	20.86	4.255	16
2	26.64	3.343	100
3	50.14	1.818	13

The differences between the monthly run and the standard peak locations for each of the three peaks are calculated and recorded. A linear regression analysis is performed on the recorded results (Figure 1). Should the regression analysis show an unacceptable amount of drift, the root cause will be determined and actions will be taken to correct the inconsistency.

The second area of interest is the check of x-ray tube and detector performance. Intensities measured using the Novaculite standard are recorded over time. Drift is assessed using linier regression on the dataset recorded over time. A series of low and high angle diffraction peaks is used to track any variation in peak intensity. An example of this tracking is shown in Figure 2. If

the intensity falls below an acceptable level, the root cause will be determined and actions will be taken to correct the variance from acceptable operating conditions.

The third area of interest is the check of detector performance. A plot of the 65 to 70 degrees two theta region versus intensity will be observed for the split of the five peaks that make up this region of the quartz standard diffractogram. An example of this region is shown in Figure 2. In addition, the Ka1 peak FWHM at 59.2 degrees two theta will be determined and plotted for variation. If the resolution increases to an unacceptable level, the root cause will be determined and actions will be taken to correct the variance from acceptable operating conditions.

Figure 1
Example of a plot tracking drift in the 2θ position of a selected line in measured quartz (Novaculite) diffractograms recorded at different times.

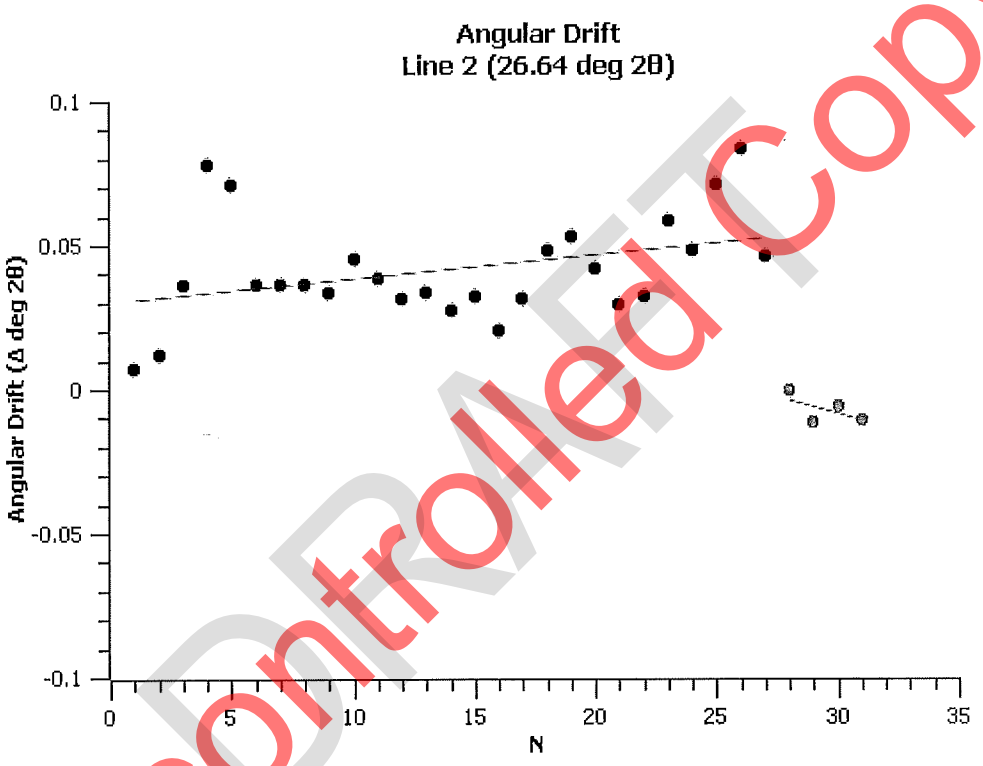


Figure 2
Example of a plot tracking changes in the intensity of a selected line in measured quartz (Novaculite) diffractograms recorded at different times.

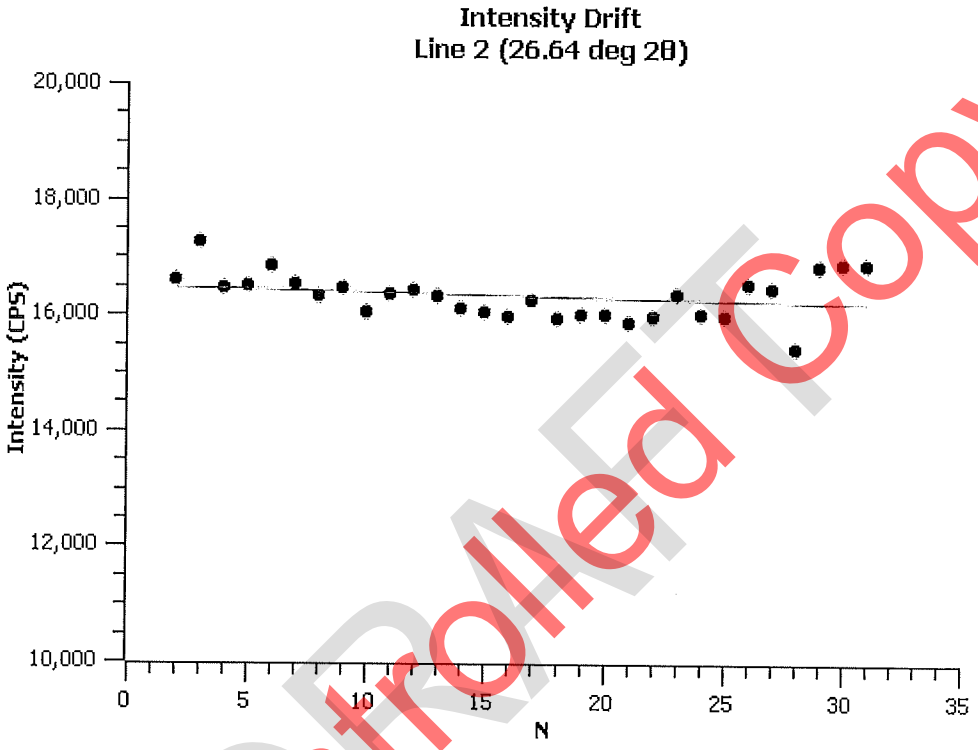
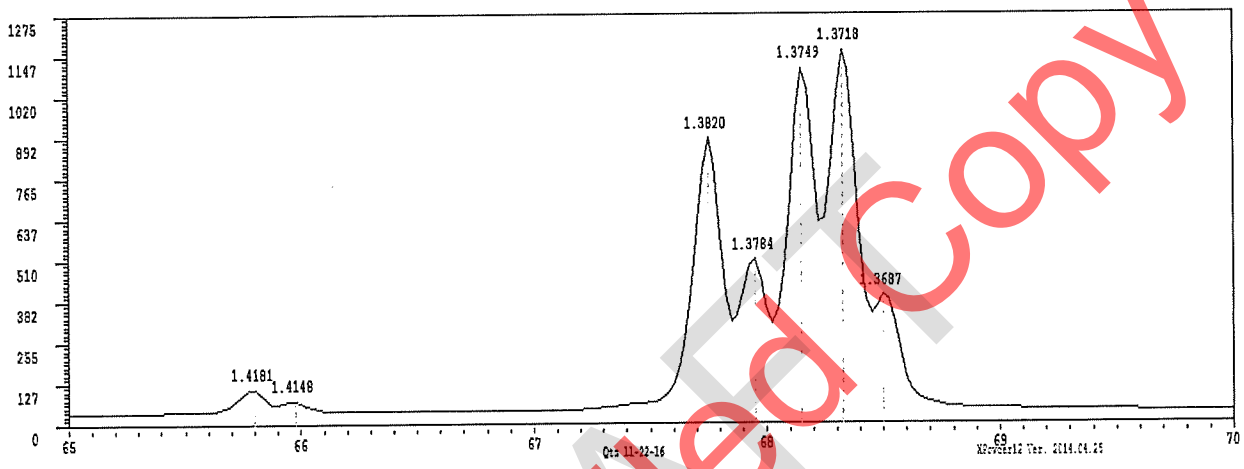


Figure 3
An example of a resolution check.



MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE		
Determination of Uranium by a Modified Davies-Gray Titration: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President	9/15/11 Date
	Quality Assurance Officer	9/12/11 Date

1.0 PURPOSE

This procedure applies to samples of uranium compounds of the nature UxFz,, UxOyFz, UxOy and others relating to uranium contaminated scrap materials where interfering elements are kept to a minimum.

2.0 SCOPE

This procedure may also be applied to determine levels of uranium in aqueous and solid samples.

3.0 ROLES AND RESPONSIBILITIES

MCLinc analyst is responsible for performing the analysis on the samples per this procedure, reviewing the results, and reporting any problems.

The Operations Manager or Project Manager represents the first level of management and provides project oversight.

4.0 MATERIALS AND APPARATUS

- Platinum wire: 12"
- Orion Ag-AgCl Half-Cell Single Junction Reference Electrode
- *Thermo Scientific Orion Star pH/ISE Benchtop Meter* or equivalent
- Micro buret-2 ml, 0.002 ml graduations, Gilmont GS-1200A
- Magnetic stirrer
- Teflon coated stir bars
- Hot plate
- Fume hood
- Muffle or tube furnace

- Assorted laboratory glassware – cleaned in laboratory detergent solution and rinsed well in DI H_20
- Platinum or quartz boats
- Thermometer

5.0 REAGENTS

- Sulfuric acid (H_2SO_4): 96%, concentrated.
- Sulfamic acid (H_2NSO_3H): reagent grade.
- Phosphoric acid (H_3PO_4): 85%, concentrated.
- Ferrous sulfate ($FeSO_4.7H_2O$)), granular or crystal.
- Nitric acid (HNO_3): 70%, concentrated.
- Ammonium molybdate [$(NH_4)_6Mo_7O_{24}.4H_2O$], crystals.
- Vanadyl sulfate ($VOSO_4.nH_2O$), 99% pure.
- Potassium dichromate ($K_2Cr_2O_7$), Primary Standard Grade.
- Triuranium octaoxide, (U_3O_8), highly pure.
- Distilled or deionized water.
- Chromic acid for glassware cleaning.
- Sodium hydroxide, (NaOH), pellets or other caustic chemical for acid neutralization
- Laboratory detergent.

6.0 REAGENT PREPARATION

A. 1 M Sulfuric acid solution:

1. To a 2-liter volumetric flask, add ~ 1000 ml DI H_2O.
2. Carefully, while holding flask under the cold water faucet, add 110 ml of concentrated H_2SO_4 while swirling.
3. Allow to cool and then dilute to volume with DI H_2O.

Shelf life: ~ 6 months.

B. 1.5 M Sulfamic acid:

1. To a 1-liter volumetric flask, add 145.5 g of sulfamic acid and ~ 800 ml of DI H_2O.
2. Stir on a magnetic stirrer with gentle heat sufficient to dissolve the solids.
3. Cool and dilute to volume with DI H_2O.

Note: 100 ml of this solution is used in the preparation of the reagent in 6.0 D.

Shelf life: ~ 6 months.

C. 1 M Ferrous sulfate solution:

1. To a 100 ml volumetric flask, add ~65 ml DI H_2O.
2. Carefully add 10 ml of concentrated H_2SO_4.
3. Add 28 g of $FeSO_4.7H_2O$.
4. Carefully shake to dissolve, cool, and dilute to volume with DI H_2O

Shelf life: 2 days

D. Nitric-sulfamic acid solution with ammonium molybdate:

1. To a 1 liter storage bottle, add 400 ml of DI H_2O.
2. Add 4.0 g of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ and dissolve.
3. Add 500 ml of concentrated HNO_3 and mix.
4. Add 100 ml of the sulfamic acid solution previously prepared in 6.0 B and mix well.

Shelf life: ~ 6 months

E. 0.027 N Potassium dichromate standard solution:

1. Dry *NIST* SRM 136*F* $K_2Cr_2O_7$ or equivalent primary standard grade for *2* hrs at 110 °C.
2. Cool in dessicator.
3. Weigh out about 1.325 g (accurately to 4 decimal places) of the dichromate for 1 liter of solution corrected for the assay.
4. Add the weighed dichromate to a 1-liter volumetric flask (calibrated), dissolve and dilute to volume with DI H_2O.
5. Transfer the solution to a 1 liter glass bottle.
6. Calculate the normality of the solution.

Normality ($K_2Cr_2O_7$) = mass(g) x assay x 1 mol/294.1844 g x 6 eq/mol x 1/vol. flask (L)

Shelf life: indefinite.

F. 0.008 N Potassium dichromate solution:

1. Dissolve 0.39 g $K_2Cr_2O_7$ in a 1-liter volumetric flask with DI H_20.
2. Bring to volume with DI H_2O.
3. This solution is used to oxidize impurities in the phosphoric acid, its normality does not have to be precise.

Shelf life: indefinite

G. Uranium standard solution, ~ 340 ppm U in 10% HNO_3.

1. Place a small quantity of NBS 950b U_3O_8 in a quartz or platinum boat.
2. Insert the boat into the tube furnace at 800 deg C for 1 h.
3. Cool in a dessicator to room temperature.
4. Weigh out ~0.4 g of U_3O_8 in a 200 ml tall form beaker.
5. Add 50 ml 10% HNO_3 and 50 ml conc. HNO_3.
6. Heat gently on a hot plate to dissolve.
7. Cool and transfer to a 1 liter calibrated volumetric flask using DI H_2O.
8. Add 45 ml of conc. HNO_3.
9. Dilute to volume with DI H_2O.
10. Transfer the solution to a 1 liter glass bottle.

Shelf life: indefinite

Calculation of uranium concentration in the standard solution.:

0.4000 g U_3O_8/1 L x 0.99968 g U_3O_8/1.00000 g U_3O_8 x 0.848001 g U/ 1.000000 g U_3O_8 x 1000 mg U/1 g U = 339.09 mg U/L

Standards are reanalyzed when the deviation from the accepted value exceeds 0.1 mg.

H. Uranium standard solution, ~ 42 ppm U in concentrated H_3PO_4.

1. Place a small quantity of NBS 950b U_3O_8 in a quartz or platinum boat.
2. Insert the boat into the tube furnace at 800 deg C for 1 h.
3. Cool in a dessicator to room temperature.
4. Weigh out ~0.05 g of U_3O_8 in a 200 ml tall form beaker.
5. Add ~25 ml concentrated H_3PO_4.
6. Heat gently on a hot plate to dissolve.
7. Cool and transfer to a 100 ml volumetric flask using concentrated H_3PO_4.
8. Dilute to volume with concentrated H_3PO_4.

Shelf life: U+4: Analyze within a week, total U indefinite

Calculation of uranium concentration in the standard solution.:

0.0500 g U_3O_8 x 0.99968 g U_3O_8/1.00000 g U_3O_8 x 0.848001 g U/ 1.000000 g U_3O_8 x 1000 mg U/1 g U = 42.386 mg U/L

Standards are reanalyzed when the deviation from the accepted value exceeds 0.1 mg.

7.0 PROCEDURE

A. Total U for samples dissolved in dilute HNO_3

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).
   c) 3 ml conc. H_2SO_4 and swirl.
   d) 5 ml 1.5 M sulfamic acid and swirl.
   e) 40 ml conc. H_3PO_4 down the beaker walls and swirl.
   f) 3 ml DI H_2O and swirl.
   g) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   h) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   i) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   j) 100 ml 1 M H_2SO_4 (wash down thermometer).
   k) 100 mg – 120 mg vanadyl sulfate.

2. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on uranium concentration, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

3. Place the remaining solution in the appropriate waste container.

B. Total U for samples dissolved in concentrated H_3PO_4.

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).
   c) 3 ml conc. H_2SO_4 and swirl.
   d) 5 ml 1.5 M sulfamic acid and swirl.
   e) 28 ml conc. H_3PO_4 down the beaker walls and swirl.
   f) 11 ml DI H_2O and swirl.
   g) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   h) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   i) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   j) 100 ml 1 M H_2SO_4 (wash down thermometer).
   k) 100 mg – 120 mg vanadyl sulfate.

2. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on uranium concentration, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

3. Place the remaining solution in the appropriate waste container.

C. Procedure for U^{+4} (sample must be dissolved in concentrated H_3PO_4)

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).

Allow the beaker to stand while the rest of the reagents are added to a separate clean beaker.

2. To a separate clean 250 ml beaker, add:
   a) 15 ml conc. H_3PO_4 and swirl.
   b) 3 ml conc. H_2SO_4 and swirl.
   c) 5 ml 1.5 M sulfamic acid and swirl.
   d) 13 ml conc. H_3PO_4 down the beaker walls and swirl.
   e) 11 ml DI H_2O and swirl.
   f) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   g) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   h) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   i) 100 ml 1 M H_2SO_4 (wash down thermometer)..
   j) Add this solution to the beaker containing the pipetted sample (steps 1a-1b).
   k) Add 100 mg – 120 mg vanadyl sulfate.

3. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on sample size, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

4. Place the remaining solution in the appropriate waste container.

8.0 CALCULATIONS

Sample Titration Data showing 2nd derivative method:

A Volume (ml)	B Potential (mV)	C d(B)/ d(A)	D d^2(B)/ d^2(A)
0.000	559		
0.008	576		
0.010	583		
		\ 7,500 / \	
0.012	598		+ 4,500
		\ 12,000 / \	
0.014	622		- 2,500
		\ 9,500 /	
0.016	641		

Endpoint = 0.012 ml + 0.002 ml [4,500 / (4,500 + 2,500)] = 0.01329 ml

Sample Calculation for the Amount of Uranium:

[0.01329 ml - 0.0024 ml (blank)] x 0.027039 meq/ml x 1 mmol/ 2 meq x 238.03 mg U/ 1 mmol U = 0.035 mg U

Reporting limit – The reporting limit for this procedure is the amount of uranium corresponding to 0.005 ml of titrant after blank correction.

9.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Because all materials utilized in this procedure are potentially radioactive sources, all samples, waste, and standards will be appropriately labeled and handled according to MCL-7718 and MCL-7715.

The waste will be minimized by using small volumes and minimizing quantities utilized for sample preparation and standards preparation. Materials for disposal will be segregated and properly labeled. Where possible, the waste will be reduced by known treatment methodologies.

Rad waste will be measured and documented and where necessary turned over to an approved commercial handling and disposal service.

10.0 REFERENCES

1. W. Davies and W. Gray, "A Rapid and Specific Titrimetric Method for the Precise Determination of Uranium Using Iron (II) Sulfate as Reductant,"Talanta" 11, (1964), p. 1203.

2. Eberle et al., "Titrimetric Determination of Uranium in Product, Fuel, and Scrap Materials After Ferrous Ion Reduction in Phosphoric Acid," New Brunswick Laboratory Progress Report No. 252, July, 1970.

3. R.J. Jarabek, Transport Measurements of UF_5 Using a Precision Analysis for U^{+4}, K/PS-5017, Martin Marietta Energy Systems, Inc., Oak Ridge Gaseous Diffusion Plant, April 2, 1984.

4. D.A. Skoog and D.M. West, Fundamentals of Analytical Chemistry, Holt, Reinhart, and Winston, Inc., pp. 550-554, 2nd ed., 1969.

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE		
Acid Digestion for Metals Based on EPA Method 3050B: **Materials and Chemistry Laboratory, Inc.**	Approved: MCLinc President	5/9/14 Date
	Quality Assurance Officer	5/6/14 Date

1.0. PURPOSE

This document provides the procedural steps and materials necessary to digest air filters, wipes, and other industrial hygiene samples, and solid samples including soils for total environmentally available metals for subsequent analysis by Flame atomic Absorption, GFAA, and Inductively Coupled Plasma

2.0. SCOPE

This procedure is based on the USEPA metals preparative Method 3050B as defined in SW-846 and NIOSH Method 7300 Elements by ICP.

3.0. ROLES AND RESPONSIBILITIES

MCLinc Analyst is responsible for following this procedure and reporting any anomalies that may occur and reviewing the results and properly documenting all elements as required in the procedure.

MCLinc Project Manager provides project oversight and is responsible to assure all users of this procedure on the project are trained and understand the procedure. The MCLinc Technical Director and QA Officer will provide support as needed.

4.0. REAGENTS/MATERIALS/EQUIPMENT

4.1. Reagents

Nitric Acid – concentrated, trace metals grade
Hydrogen peroxide – 30%
Nitric Acid Solution – 1:1 concentrated trace metals grade nitric/DI water.
Spiking Solutions
Hydrochloric Acid – concentrated, trace metals grade

4.2.Equipment

Glass beakers
Ribbed watch glasses capable of covering the beakers
Electronic Balance
Hot plate with temperature monitoring capabilities (i.e. thermometer in beaker of water)

Assorted laboratory glassware (volumetric flasks, graduated cylinders, pipets, etc.)
Funnels and Whatman # 41 filter paper or MCLinc approved equivalent.
Hot Block with temperature monitoring capability (i.e., thermometer in digestion tube of water *[For recording the Hot Block used and the location of the thermometer tube location, see Appendix A.*)
50mL digestion tubes to fit hot block
50mL centrifuge tubes

4.3. Miscellaneous

Latex/nitrile gloves
Tongue depressor or metal spatulas
DI water bottle
Paper towels
Sample Prep/lot Sheet
Polyethylene bottle

5.0. PROCEDURE

Note: Clean all glassware per MCL SOP for Glassware Cleaning, MCL-7753.

5.1. Sample Preparation

1. Identify beaker and tare on the balance.
2. Transfer 2g±0.1g using a clean unused tongue depressor to the tared beaker.
3. Record the MCL Sample No. and weight on the prep/lot sheet.
4. Add 10 ml of the 1:1 nitric acid solution to the beaker mix, cover with a watch glass and reflux at 95°C±5°C for a minimum of 10min without boiling.
5. Allow to cool, add 5 ml of concentrated nitric acid, replace the watch glass and reflux at 95°C±5°C for a minimum of 10 min without boiling.
6. If brown fumes appear repeat step 5 until there are no more brown fumes being generated.
7. Once the generation of brown fumes has stopped allow to heat until the volume has been reduce to approximately 15-20 ml. **Do not allow to go to dryness.**
8. Allow the sample to cool, add 2 ml of DI water and 3 ml of 30% peroxide. Add the peroxide slowly being careful not to allow the sample to effervesce out of the beaker. Continue to add peroxide in 1ml aliquots until all effervescing has stopped. Do not add more than 10ml total volume of peroxide.
9. Cover the sample and reflux at 95°C±5°C again reducing the volume to approximately 15-20 ml. **Do not allow to go to dryness.**
10. After cooling, filter using a funnel and Whatman #41 filter paper. Quantatively transfer the filtered sample to a 100 ml volumetric flask with DI water and bring up to volume.
11. Pour sample solution into properly labeled polyethylene sample bottle.

5.2. Sample Prep with Hot Block Digestion

1. Weigh Sample (2.0+/-0.1g in tared vials or carefully place air filters, wipes directly into the vials for hotblock digestion.)
2. Turn on hot block @ set point = 115 per manual *(95 °C ±5°C)* temperature. *Check temperature for each set of samples.*

3. Weigh samples in tared vials for hot block digestion.
4. Add 10ml 8N HNO_3 and 3ml HNO_3 .
5. Digest for 1 hour.
6. Remove from hot block and cool.
7. Add 3-10ml H_2O_2.
8. Digest for 30 minutes.
9. Remove from hot block and allow to cool.
10. Filter into 50mL centrifuge tube containing 5mL conc HNO_3 for ICP-OES analyses. For ICP/MS omit the 5mL conc. HNO_3.
11. Bring to volume and run on ICP.

5.3. Special Instructions – Antimony

1. This procedure is for preparation for solid sample(s) requiring antimony analysis. If sample(s) require the analysis of other metals, use this digestion procedure for the preparation for all metals.
2. Add 2.5 mL conc. HNO_3 and 10 mL conc. HCl to a 1-2 g sample (wet weight) or 1 g sample (dry weight) and cover with a watchglass or vapor recovery device. Place the sample in the hot block at *95 ℃ ±5°C* and reflux for 15 minutes.
3. If the sample has not dissolved in the acid solution, proceed to Step 4. If the entire sample has dissolved in the digestate acid solution, filtration is not necessary. The final sample volume will be 50mL if 10 or fewer metals will be analyzed or 100mL if more than 10 metals are analyzed. Allow the digestate solution to cool. Quantitatively transfer the digestate solution to the proper size container and dilute to volume with reagent water.
4 Filter the sample/digestate solution through Whatman No. 41 filter paper or equivalent and collect filtrate in a clean 100mL volumetric flask. Wash the filter paper while still in the funnel with 5mL of hot (95±5°C) HCl, then with 5-10mL of hot reagent water. Collect the wash solutions in the same flask.
5. Remove the filter paper and residue from the funnel and place back in the digestion vessel. Add 5mL of hot HCl and place the digestion vessel back in the hot block. Heat at 95±5°C until the filter paper dissolves. Remove the tube from the hot block and rinse the watch glass and sides of tube with reagent water. Filter the residue and collect the filtrate in the same flask as in Step 4. Allow the filtrate to cool. If there is no precipitate present after cooling, dilute to volume with reagent water. If there is a precipitate present, see NOTE and proceed to Step 6. NOTE: High Concentrations of metal salts with temperature-sensitive solubilities can result in the formation of precipitates in these solutions upon cooling. If precipitation occurs, do not dilute to volume.
6. If a precipitate forms, add up to 10mL HCl. After precipitate has dissolved, dilute the sample to volume with reagent water.

5.4. *Special Instructions – Beryllium Oxide (BeO)*

For samples requiring the analysis of BeO, prepare samples per Operator Aid UU in McLinc SOP MCL-7756.

5.5. Other Special Instructions

Radiological Screening Samples

1. Label the top of a 20 ml scintillation vial with the sample number.
2. Transfer 0.5- 2 ml of the final digestate solution from Step 10 above.
3. Proceed per MCL 7733 Section 6.6.1.11

Shipping metals digested sample

1. Label 250 ml plastic sample bottle with the MCL sample number, TM (for Total Metals) designator and Batch ID.
2. Fill bottle with final digestate solution; seal and stage for shipping

6.0. QUALITY CONTROL (QC)

Each batch of 20 or fewer samples will contain a minimum of *two* Laboratory Control Sample*s* (LCS *and LCSD*) and a Method Blank (MB). Matrix Spike/Matrix Spike Duplicate (MS/MSD) will be added if required by project.

6.1. LCS

Since the metal requests vary by project, select appropriate standard spiking solutions for LCS*s* that match sample request. Spike levels should be within the calibration range of the metal. For the digestion of air filter samples or wipe samples, add a new filter or wipe to the LCS sample*s* before digestion.

6.2. Method Blank

1. Run with each set of samples digested.
2. Using a clean beaker begin at Step 4 above in 5.1 and process with the rest of the samples.

6.3. MS/MSD

Matrix spike (MS) and matrix spike duplicate (MSD) are project specific and are used to determine accuracy. If required, one set of MS/MSD is included with each batch of 20 or fewer samples processed.

7.0. REPORTING

All recording of information shall be done on the Metals Sample Preparation Log Sheet. An example is presented in Appendix A.

8.0. POLLUTION PREVENTION AND WASTE MANAGEMENT

Because all materials utilized in this procedure are potentially radioactive sources, all samples, waste, and standards will be appropriately labeled and handled according to MCL-7718 and MCL-7715.

The waste will be minimized by using small volumes and minimizing quantities utilized for sample preparation and standards preparation. Materials for disposal will be segregated and properly labeled. Where possible, the waste will be reduced by known treatment methodologies.

Radioactive waste will be measured and documented and where necessary turned over to an approved commercial handling and disposal service.

9.0. REFERENCES

USEPA SW-846 Third Edition Revision 2, December 1996 Method 3050B Acid Digestion of Sediments, Sludges, and Soils.

NIOSH Method 7300 Elements by ICP, Fourth Edition, Issue 2, August 15, 1994.

APPENDIX A. HOT BLOCK LABELS

The hot blocks used for metals digestion are not moved from the laboratory or within the laboratory. The two large hot blocks are identified as A and B from left to right within the fume hood they are located within. The two small hot blocks are identified as C and D within the fume hood they are located within. Record on the prep sheet the Hot Block number and the well used for temperature determination. Move the thermometer to the next location with every set of samples digested.

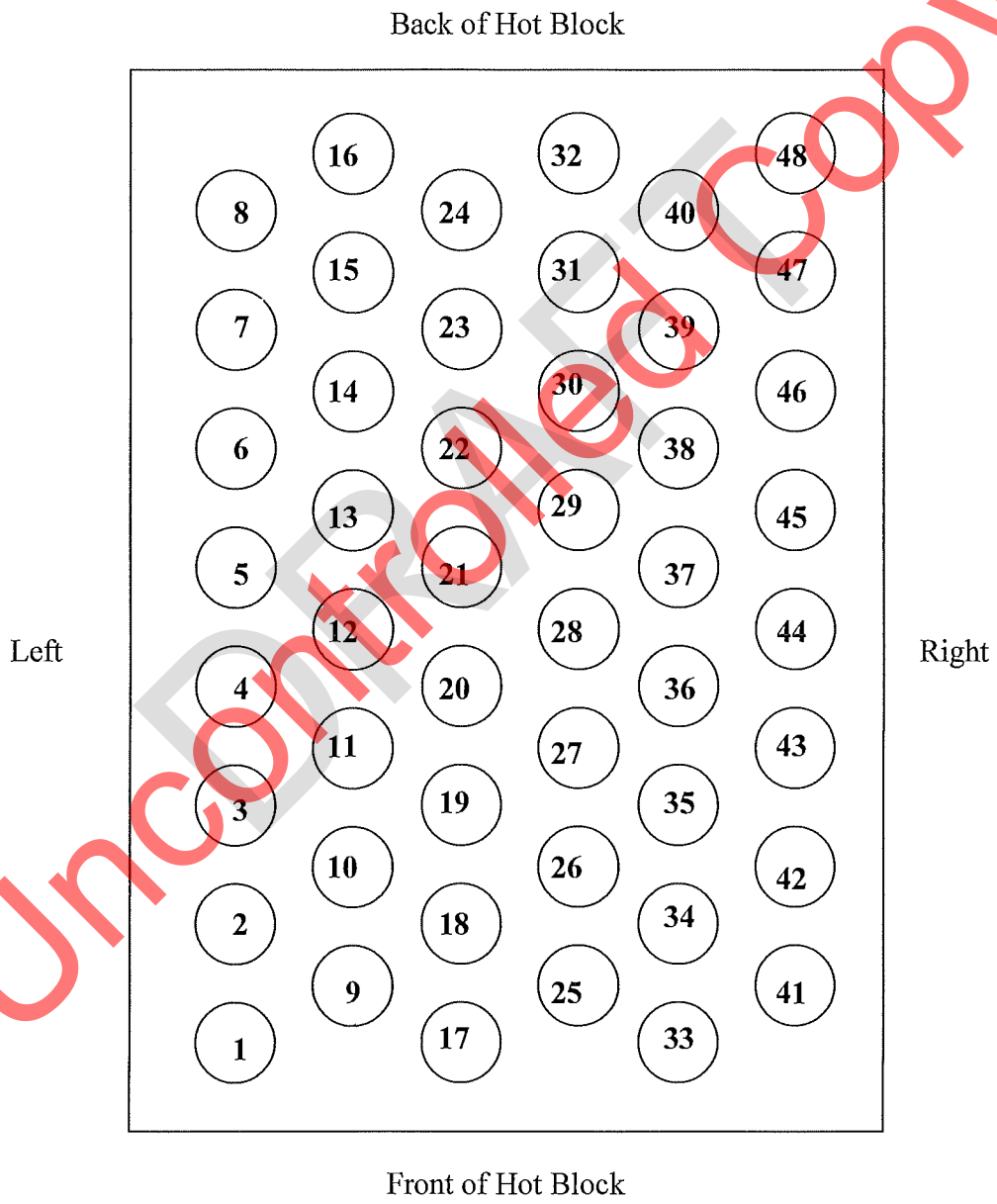


Figure 1. ***The wells within the Large Hot Blocks (A and B)are numbered from Left to Right, Front to Back in numerical order starting with 1.***

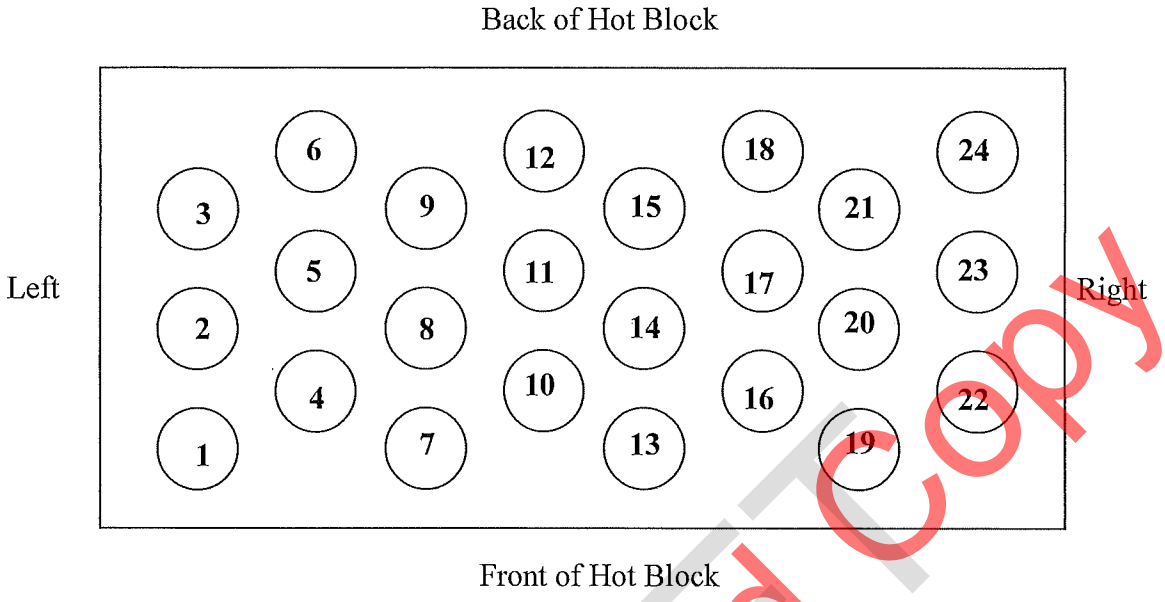


Figure 2. ***The wells within the Small Hot Blocks (C and D) are numbered from Left to Right, Front to Back in numerical order starting with 1***

Procedure: MCL-7746 or Be MCL-7756, App. UU Sample Type: Air Filters, Wipes, Solids, Soils, Oils

Date: _____ Prepared By: _____ Project ID: _____

Spike ID(s):A_____B_____C_____D_____

Used (check one): Class A Glass Pipets? ☐ Autopipet? ☐ Autopipet #: _____Verification Wt. _____ (DI H_2O) (g)

QA/QC Information	QC Sample ID	Matrix (soil, wipe, filter, DI H_2O)	Aliquot (g) or (mL)	Volume (mL) & Spike ID	Final Vol. (mL)	Comments

Sample Information	MCL Sample ID	Matrix (soil, filter, etc.)	Aliquot (g) or (mL)	Final Vol. (mL)	Digestion Temp (°C)	Comments	Chemicals/Reagents Used (include Prep Log Date)

Balance ID		Weight (g)				
Calibration ID		Actual (g)				
Weight Set ID		Actual (g)				
		Avg (g)				

Hot Block Used (circle one): **A** **B** **C** **D** Thermometer ID: _____

Temp. Blank Location:_____ Signature _____

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE	
Inductively Coupled Plasma-Atomic Emission Spectrometry Metals Analysis: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President — Date 12/20/18 Quality Assurance Officer — Date 12/20/18

1.0 PURPOSE

This document describes the procedures to determine elements/metals in properly prepared samples based upon NIOSH Method 7300 and USEPA SW-846 Method 6010 Metals using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

2.0 SCOPE AND APPLICATION

2.1 Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) determines trace elements/metals, in solution. This method can be used for all elements listed in Table 1. All matrices, excluding filtered acid preserved groundwater samples; other aqueous samples, (i.e. TCLP/EP extracts; unfiltered groundwater), industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Both non-digested and digested samples must be matrix matched with the same type and concentrations of acids as found within the standards.

Table 1 lists the three analytical wavelengths to be measured per element and method detection limits for the elements. Elements other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest is demonstrated.

2.2 Users of this method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using this method for analysis.

2.3 Use of this method is restricted to chemist/qualified operators who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method. They must also have been appropriately trained on the instrumentation and its software, along with use of Attachment 1 for determining results.

3.0 RESPONSIBILITES

MCLinc Project Manager is responsible for seeing that a Total Radiological Activity Screening analysis is performed on radiochemical samples as received in a timely manner prior to ICP analysis. The Project Manager is also responsible for assuring project QA/QC is clearly defined to the ICP operator and sample preparation.

The MCLinc Analyst is responsible for routine operation, inventory of all required materials, upkeep of equipment, reviewing and reporting of results, and the housekeeping of the work area associated with the equipment.

The Operations Manager represents the first level of management and provides project oversight and is responsible for supplying the resources for proper upkeep of the required instrumentation.

4.0 SUMMARY OF METHOD

4.1 Prior to analysis, samples, except filtered and acid preserved groundwater, must be digested using appropriate sample preparation methods. This includes all total and "acid-leachable" analyses.

4.2 This method describes multi-elemental determinations by ICP-AES using a sequential optical systems and axial/radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry at the defined wavelengths. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required particularly for trace elements. A minimum of one background measurement must be measured at a wavelength adjacent to all analyte wavelengths on all samples and QA/QC during analysis. (Note two point background measurements are the preference and to be done routinely. The selection of one point is for unusual measurements.) The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In the selection of the reported wavelength the analyst should select the best representation of the measured wavelength that is determined to be as free as possible from spectral interference and appropriately compensated for background intensity. Background corrections are made as needed to compensate for excessive interferences if they occur on all three of the calibrated monitored wavelengths.

The logic for the selection of the reporting wavelength and affiliated concentration is shown in Attachment 1.

For each element - two primary and a secondary wavelength are measured. These wavelengths are predetermined (Ref. Table 1) based upon their response and the relative absence of spectral interferences.

5.0 DEFINITIONS

5.1 Applicable definitions are located throughout this SOP.

6.0 INTERFERENCES

6.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra, and instrumental noise (i.e. plasma flutter, pump pressure/nebulizer flutter etc.).

6.2 Background emission and stray light can usually be compensated for by subtracting the background intensity on either side of the analyte wavelength peak. The use of multiple wavelengths for an analyte allows the selection of the wavelengths with the least amount of interference and/or background emission for reporting. The locations selected for the measurement of background intensity are determined by the complexity of the spectrum adjacent to the analyte wavelength peak. The placement of the wavelength peak baseline can be made during method set up before an analysis or during analyst data review after the analysis. The wavelength peak baseline used for routine measurement must be free of off-line spectral interference or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

6.2.1 The analyst in establishing the method and reviewing the resulting spectra will verify presence or absence of spectral interference by:

- Evaluating scanned wavelength on either side of the analyte wavelength peak
- Determining the shape of the analyte wavelength peak of a sample compared to a calibration standard
- Evaluating the analyte wavelength peak integration

6.2.2 Samples that show a elevated background emission/interferences across the range for all three defined wavelengths may be background corrected by applying the instrumental software correction program that uses algorithms to compensate and interpolate contributions from adjoining interfering spectra (i.e. interelement interference etc.). Individual spectra that show interference will be corrected only if deemed necessary due to problems with the other spectra for the affected analyte.

6.2.3 To determine the appropriate location for background correction, the user must scan the area on either side adjacent to the specified wavelength and define these areas appropriately in the establishment of the analytical methods files or during data review.

6.2.4 The potential for spectral overlaps are avoided/greatly reduced by measuring multiple wavelengths for each of the target elements.

6.2.5 Because interelement corrections vary depending upon the choice of background correction points and the complexity of the sample, multiple wavelength measurements are being used in this SOP for routine operation instead of interelement correction factors and corrections. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences that can only be

compensated for by interelement manipulation, use of multiple wavelength, or software algorithms or methods of standard additions.

6.2.6 The interference effects must be evaluated when instrument parameters are changed. Intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). Even though these variables are compensated for by the calibration of each defined wavelength for each target element, the analyst is required to review the results for each wavelength. This review will determine and document per wavelength the effect of the interferences and the selection of the wavelength to be reported.

6.2.7 When the instrumental software interelement correction algorithms are applied, their accuracy should be verified, by analyzing the appropriate spectral interference check solutions.

6.2.8 When interelement corrections are used, verification of absence of interferences is required or proof that the interference is not included in the data. To demonstrate this absence of interference, an Interference Check Solution (ICS) containing similar concentrations of the major components of the interference contributing elements at > 10mg/L must be run with each new project; the resulting data must be kept on file with the sample analysis data and the affected element (those elements with > 20% variability from expected value) flagged appropriately.

6.3 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. A Yttrium internal standard is not used routinely but will be used if the analyst and QC deem necessary to allow for appropriate correction if physical interferences are present.

6.4 Another physical interference that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. Routine maintenance and operational awareness/data review will minimize the occurrence of this interference.

6.5 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample (e.g. the addition of competitive ionization potentional compounds), by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte.

6.6 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and are minimized by high flow flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times.

7.0 SAFETY

7.1 General laboratory protection (safety glasses, lab coat, and disposal latex/nitrile gloves) should be worn at all times when handling standards or samples.

7.2 Stock metal standards and acid solutions may pose potential health risks. Extreme care should be utilized when handling these solutions.

8.0 APPARATUS AND MATERIALS

8.1 Perkin Elmer 2000 Model Inductively Coupled Argon Plasma Atomic-Emission Spectrometer with both axial and radial measurement capability.

8.2 Sequential multiple wavelengths per analyte with affiliated computer-controlled emission spectrometer and background correction.

8.3 Radio-Frequency generator compliant with FCC regulations.

8.4 Mass flow-controller for argon nebulizer gas supply. (Geminheart nebulizer and cyclonic spray chamber)

8.5 Peristaltic pump.

8.6 Perkin Elmer Autosampler.

8.7 Argon gas supply: high-purity grade (99.99%).

8.8 Nitrogen, dry – 99% purity

9.0 REAGENTS

9.1 Acids used in the preparation of standards and for sample processing must be of high purity. Nitric acid (conc), HNO_3, trace metals grade

9.2 Reagent water: All references to water in the method refer to ASTM Type II (>1Mohm-cm) water.

9.3 Standard stock solutions are either purchased commercially as certified standards or prepared from ultra-high purity grade chemicals or metals (99.99 or greater purity) within the lab.

Mixed calibration standard solutions - Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to polyethylene or polypropylene bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentration can change on aging. Standards greater than 100ug/L are stable for one year from preparation date in a 10% acid solution. Standards less than 100ug/L are stable for only 6 months in a 10% acid solution. Stock standards are per manufactures expiration date and in-house at greater than 1,000mg/L are stable for 3yrs in 10% acid.

9.4 Two types of blanks are required for the analysis of samples. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample preparation processing.

9.4.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. The calibration blank will also be used for all initial and continuing calibration blank determinations. The calibration blank is also analyzed prior to calibration and immediately after all CCV's. The resulting spectral values for each measured wavelength are automatically subtracted from the calibration standards measurements.

9.5 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. This result is not subtracted from the samples measurements but reported as a separate QC result.

(OPTIONAL) Working ICS Solutions for checking interferences and case-by-case interference correction if required. The stock solutions for the ICS solutions will be procured certified from a commercial source.

9.6 The quality control standard is a second source standard used for Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV).

The second source solution is an independent standard near the midpoint of the calibration linear range at a concentration other than that used for instrument calibration for the majority of the calibration analytes. This standard will contain each analyte found in each of the stock solutions used to prepare the commercial standard. An independent standard is defined as a standard composed of the analytes from either a source different from those used in the standards for instrument calibration or from the same vendor but a different lot.

10.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

10.1 Sample collection procedures should address all considerations described in Quality Assurance Project Plan.

10.2 Plastic or glass containers are acceptable for use in Method 6010B.

10.3 Aqueous samples should be preserved with 1:1 HNO_3 to a pH < 2.

11.0 QUALITY CONTROL

11.1 The type and frequency of the quality control program will be defined by the project. Dependant on the project defined program the following quality control data, and as defined in Table II may be included The resulting data should be maintained and be available for easy reference or inspection.

11.2 Lower Instrument Detection Limits (IDLs) in µg/L can be estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined annually and kept on file. The IDL will be determined by the multiplication of the average standard deviation for each of the three days analysis measurement for each analyte wavelength by 3.14. The IDL will be defined by the least sensitive wavelength as defined in Table 1 for each measured element unless lower limits are required by the specific project.

11.3 The upper detection Limit is defined by the point where data results are not reportable due to either:
1) The calibration line is no longer linear
2) For non linear lines it is the point where the line curvation is lost and the line becomes relatively flat.

11.4 The reporting limits will be defined by the limits of the upper and lower calibration standard. All values outside the calibration range (i.e. the upper and lower reporting limit) will either be diluted to be within the calibration range or reported as estimated values. Table 1 list routine reporting limits based on least sensitive of the three wavelengths except where noted. Lower; lower reporting limits can be achieved on numerous analytes based on one or two lines if need by specific project.

11.5 A minimum three (3) point calibration curve will be developed prior to sample analysis. Two points will be the concentrations defining the upper and lower calibration limit for every wavelength being used in the analytical run (i.e. 3 wave lengths for each target analyte)

11.6 Dilution Test: This test may be applied for unusual matrices. If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times greater than the concentration in the method blank) an analysis of a

fivefold (1+4) dilution must agree within ±15% of the original determination. If not, an interference effect must be suspected. One dilution test, if applicable, would be included for each twenty samples (or less) of each matrix in a batch.

11.7 Post-Digestion Spike Addition: This test may be applied for new or unusual matrices. An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125 percent of the known value or within the laboratory derived acceptance criteria. The spike addition should be based on the indigenous concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, the sample must be diluted and reanalyzed to compensate for the matrix effect. Results must agree to within ±25% of the original determination. The use of a standard-addition analysis procedure may also be used to compensate for this effect.

11.8 There will be two different Laboratory Control Samples (LCS) analyzed with each batch of samples. These being:

1) LCS shall be prepared and analyzed in duplicate and are solutions spiked to yield concentrations in the lower to mid-range of the calibration curve with all target analytes.
2) LCSL may be requested by the Quality Manage for special projects and are solutions spiked to yield concentrations in the 2-4X the lower calibration standard for each target analyte.

The acceptance criterion for the LCS is the average of the accepted spectral lines 100±30% of the known value or within the laboratory derived acceptance criteria, should that be determined.

11.9 Calibration Verification

11.9.1 Initial Calibration Verification (ICV) verifies the instrument calibration.

The ICV will be prepared from either a second commercial source or a different lot than the primary standard used for calibration from the same commercial supplier.

The ICV will be analyzed immediately after the calibration.

The results of the ICV must agree 100% ± 10% on at least one of the analytical line not exceed 100% ± 20% on the other two analytical lines where there are no known interferences present in the ICV standard solution. The average of the interference lines must not exceed 100 % ± 17%. The 100 % ± 20% is not applicable for the few analytes where a really viable and stable second or third line (noted in Table 1) does not exist.

11.9.2 Continuous Calibration Verification (CCV) solution verifies the calibration during and after sample analysis.

The CCV solution will be analyzed after every ten (10) samples including non-blank QC samples (LCS,MB,MS/MSD samples).CCV results must agree within

100±10%R on at least one elemental line and not exceed 100±20% on the other two (2) elemental lines unless there is interference with one or both lines or if one or two other viable lines do not exist for analyte. Average recovery should not exceed 100±17%R for 3 lines.

11.10 A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) is project specific and is prepared and analyzed at a rate of every batch of 20 or fewer samples of the same matrix.

11.10.1 The Percent (%) Recovery is calculated as follows:
(Acceptance Criteria ± 25%)

$$\%R = \frac{(MS-S)}{TV} \times 100$$

Where:
MS = value of the Matrix Spike
S = value of the sample (unspiked)
TV = Theoretical Value of the Spike (Concentration of spiked solution))

11.10.2 The relative percent difference (RPD) between duplicate determinations must be calculated as follows: (Acceptance Criteria ± 20%)

$$RPD = \frac{|D_1 - D_2|}{\frac{(D_1 + D_2)}{2}} \times 100$$

Where:
RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (duplicate)

A control limit of 35% RPD should not be exceeded for analyte values greater than 100 times the instrumental detection reporting limit. If this limit is exceeded, the reason for this situation must be investigated and corrected if appropriate, and if any samples are affected, they should be reanalyzed.

11.11 Dilute and reanalyze samples that exceed the linear calibration range or use an alternate, less sensitive line or plasma viewing angle for which quality control data is established.

11.12 MDL's are performed annually; results will be on file in MCLinc's QA/QC files.

12.0 CALIBRATION

12.1 Initiate appropriate operating configuration of the instruments computer according to the instrument manufacturer's instructions.

12.1.1 Turn on the Argon flow (80 PSI minimum)

12.1.2 Turn on the water chiller.

12.1.3 Connect all pump tubing.

12.1.4 Ignite Plasma and allow for warm-up and performance of automated initialization sequence.

12.2 Perform torch alignment.

12.3 Set up the instrument with the proper operating parameters according to the methods development defined parameters.

12.4 During calibration perform a blank and wavelength correction for all of the elements wavelengths in the method preferably using the three lower standards.

12.5 Calibrate the instrument for the analytes of interest using the calibration blank and calibration standards, at the beginning of every run. Flush the system with the rinse blank between each standard solution. Use the average of at least three plasma readings per analyte for both calibration and sample analyses.

13.0 PROCEDURE

13.1 Solubilization and digestion procedures are presented in the sample preparation methods (e.g., EPA Methods 3005 - 3050). See SOP# MCL-7746, MCL-7752, and MCL-7753. For dissolved metals analysis, take an appropriate aliquot of the filtered sample and acidify with concentrated HNO_3 acid so that the final concentration of HNO_3 is 10%.

13.2 Initiate appropriate operating configuration of the instruments method file defining reporting units (ug/L liquid and mg/kg solids) calibration parameters, re-sloping parameters and frequency, CCV frequency, acceptance criteria and corrective actions, LCS Duplicate and matrix spike criteria. In the method file also define by element the 3 wavelengths to be used, axial or radial measurement and the specific plasma operational parameters.

13.2 Set up the Sequence window, which defines the methods to be used, the sample information file to be used, the samples to be analyzed by each method and the Results file name. The Results file is the file in which the data is stored.

13.3 Save the sample and method files, and run the sequence.

13.4 The sample run sequence will have an Instrument Blank (Inst. Blank) Cal Blank immediately following every CCV i.e.:

Calibration Blank
Calibration Standards-Low to High
ICV
Inst. Blank
LRL
LCS
samples including MB, MS, MSD
CCV
Inst. Blank
samples
CCV
Inst. Blank
Etc.

13.6 Flush the system with the rinse blank solution until the signal levels return to the method's levels of quantitation (defined in the established method based on time and flow rate) before the analysis of each sample. Nebulize each sample until a steady-state signal is achieved (defined by the method, depending on flow rate and tubing length etc.) prior to collecting data.

13.7 Dilute and reanalyze samples that have concentrate ions exceeding the linear range for an analyte.

14.0 CALCULATIONS

14.1 Calculations: The quantitative values shall be reported in appropriate units, such as micrograms per liter (µg/L) for aqueous samples and micrograms per gram (ug/g) for solid samples. If dilutions were performed, the appropriate corrections must be applied to the sample values

14.2 For dissolved metals analyses:

ug/L = C x DF

Where:
C = Digest concentration (ug/L)
DF = Dilution Factor

14.3 For digested aqueous samples:

$$ug/L = \frac{C \times DF \times V}{W}$$

Where:

C = Digest concentration (ug/L)

DF = Dilution Factor
V = Final volume in L after sample preparation
W = Initial volume in L of sample before sample preparation

14.4 Soil/Solid concentrations may be reported on the basis of the dry weight of the sample (A separate determination of percent solids must be performed):

$$\text{ug/g (dry weight)} = \frac{C \times DF \times V \times S}{W}$$

Where:

C = Digest concentration (ug/L)
DF = Dilution Factor
V = Final volume in L after sample preparation
W = Weight in g of wet sample
S = 100 / % Solids

14.5 Air filter samples may be reported as total microgram or milligrams per filter or if air volume is given as mg/cubic meter

Total micrograms = C x DF x V
Total milligrams = micrograms / 1000
Total mg/cubic meter = $\frac{C \times DF \times V}{\text{Air volume in } M^3}$
Where:

C = Digest concentration (ug/L)
DF = Dilution Factor
V = Final volume in L after sample preparation

14.6 All results should be reported with up to three significant figures.

15.0 METHOD PERFORMANCE

15.1 Refer to Table 1 for Method Detection Limit information.

16.0 POLLUTION PREVENTION

16.1 To minimize hazardous materials generated with this method, minimal quantities of samples are digested (50 mls final volume), and minimal quantities of standards are prepared.

17.0 WASTE MANAGEMENT

17.1 It is the laboratory's responsibility to comply with all applicable federal, state, and local regulations governing waste management.

18.0 REFERENCES

18.1 SW 846 3rd Edition, Method 6010B, Inductively Coupled Plasma – AES

18.2 NIOSH Method 7300, Fourth Edition, Issue 2, August 15, 1994

18.3 EPA SW846, Method 6010C, Inductively Coupled Plasma - AES

Table 1: Wavelength and Reporting Limits

Analyte Element	View	Wave Length #1	Wave Length #2	Wave Length #3	Lower Cal/Reporting Limit (ug/L)[1]	Upper Cal/Reporting Limit (ug/L)[1]
Al	Attn. Axial	396.153	394.401	237.313	100	3000
Ca	Radial	422.673	317.933	315.887	100	3000
Mg	Radial	279.553	280.271	285.213	100	3000
K	Attn. Axial	766.490		769.896	100	3000
K	Axial		404.721		100	3000
Cr	Axial	267.716	205.560	206.158	10	1500
Ni	Axial	227.022	221.648	231.604	10	1500
Ag	Axial	238.068	338.289	233.137[2]	10	1500
Zn	Axial	206.200	213.857	202.548	10	1500
As	Axial	193.696	188.979	197.197	8	4000
Tl	Axial	190.801	276.787	351.924	8	4000
Cd	Axial	214.440	228.802	226.502	4	2000
Se	Axial	196.026	206.279[2]	203.985[2]	25	6000
Pb	Axial	220.353	217.00	283.306	50	6000
Fe	Radial	238.204	239.562	259.939	100	10000
Co	Axial	228.616	238.892	236.380	20	6000
Ba	Axial	455.403	493.408	233.527	4	1000
Mn	Attn. Axial	257.610	259.372	260.568	4	1000
Be	Attn. Axial	313.107	234.861		4	1000
Be	Axial			313.042	4	1000
Cu	Axial	324.752	327.393	224.700	20	5000
V	Axial	292.402	311.071	270.093	20	5000
U	Axial	385.358	367.007	409.014	20	20000
Sb	Axial	206.836	217.582[2]	231.146[2]	50	6000
Ti	Axial	334.940	336.121	337.279	34	8000
Li	Radial	670.784			10	2400
Li	Axial		413.256	610.362[2]	10	2400
Mo	Axial	202.031	203.845	204.597	10	2400
Sr	Radial	407.771	421.552	460.733	7	1600
P	Axial	214.914	177.434	178.221[2]	124	11000
B	Axial	249.677	249.772	208.957	20	8000
Sn	Axial	189.927	235.485[2]	283.998[2]	40	4000
Th	Axial	283.73	401.913	339.204	20	4000
Zr	Axial	343.823	339.197	257.139	10	4000
Si	Axial	251.611	212.412	288.158	50	8000
Cs	Axial	455.531	459.320	None	5000	200000

[1] Based on Standards preparation as of 01/01/05

[2] Line is not strong due to either poor response or strong interference; but best available

Note: these parameters are subject to change based upon further evaluation by the operator

Table 2: Wavelengths and Method Detection Limits

Analyte/ Element	Primary Wave length A (nm)	Primary Wave length B (nm)	Secondary Wave length (nm)	Lower Cal/ Report Limit (ug/L) Single Line	Upper Cal Report Limit (ug/L) Three Line	IDL (ug/L)	MDL Soil (ug/g)	MDL Water (ug/L)
Al								
Ag[2]	328.98	338.289	243.778	10	100	TBD*	TBD*	TBD*
As	228.812	188.979	193.696	10	160	TBD	TBD	TBD
Ba	455.403	493.408	233.527	0.5	2	TBD	TBD	TBD
Be	313.107	234.861	313.042	2	10	TBD	TBD	TBD
Cd	214.440	228.802	226.502	2	5	TBD	TBD	TBD
Cr	267.716	205.560	284.325	1	5	TBD	TBD	TBD
Cu	324.752	327.393	224.700	15	20	TBD	TBD	TBD
Hg	184.886	194.168	253.652	20	75	TBD	TBD	TBD
Mn	257.610	260.568	259.372			TBD	TBD	TBD
Mo	202.031	203.845	204.597	1	5	TBD	TBD	TBD
Ni	231.604	221.648	227.022	4	10	TBD	TBD	TBD
Pb	220.313	217.00	283.306	40	100	TBD	TBD	TBD
Sb	252.851	206.836	217.582	2	20	TBD	TBD	TBD
Se[2]	196.026	206.279	203.985	20	50	TBD	TBD	TBD
Sr	407.771	421.512	460.733	0.1	25	TBD	TBD	TBD
U	385.958	367.007	409.014	20	40	TBD	TBD	TBD
V	292.402	309.310	311.071	1	10	TBD	TBD	TBD
Zn	206.200	213.865	202.548	5	20	TBD	TBD	TBD

Note: The project will define the target concentration and reporting limit; the reporting limit for this SOP will be defined by the least sensitive of the three wavelengths used; unless otherwise noted.

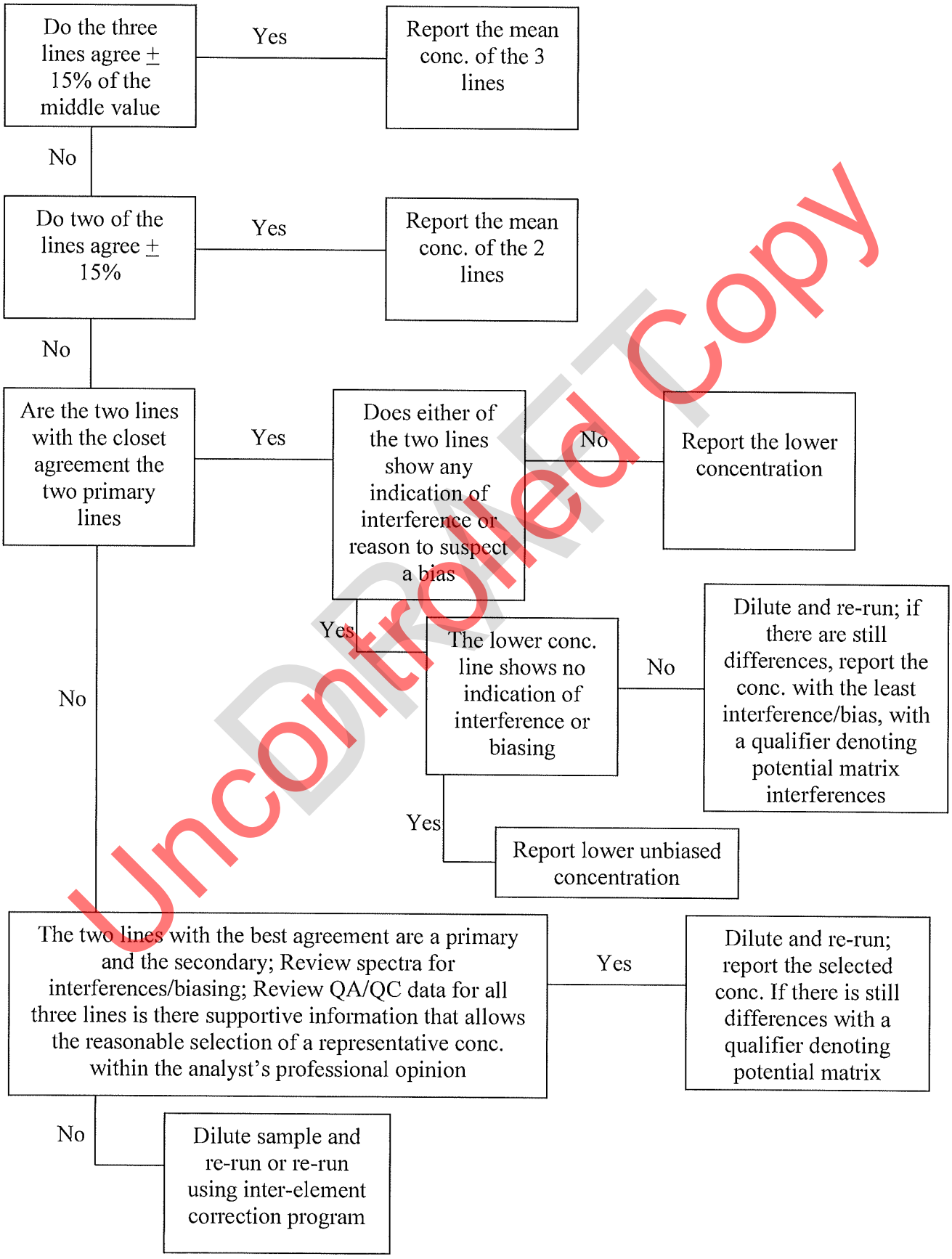
* To be determined and place in instrument log and QA files.

Table 3: QA/QC

Description	Frequency	Acceptance criteria*	Corrective Action
Calibration Curve Defining linear calibration range. 3 points minimum lower and upper defining reporting limits. 3rd point preferably near the lower	Before the analysis of the sample where after instrumental repair or maintenance and when a CCV shows and existing calibration failed	Correlation Coef(R) > 0.998	Check Standards, calibration range, operational parameters ; rerun calibration
Initial Calibration Verification Std. (ICV) Standard containing all target analytes prepared from a second source solution	Immediately after development of calibration curve & before sample analysis	100%R±10% for at least 1 of 3 lines; 100%±20% on other 2 lines except for interference or existence of other viable lines for element.	Reanalyze solution; check solution preparation; recalibrate
Continuing Calibration Verification Std. (CCV) Standard containing all the target analytes prepared from a same source solution	After every 10 samples including non-blank QC's and at the end of analysis sequence	100%R±10% for at least 1 of 3 lines; 100%±20% on other two lines except for interference or existence of other viable lines for element.	Check both CCV and calibration standards preparation; re-calibrate
Calibration Blk.	Immediately following every CCV	Analyte concentration less than half of LRL	Check tubing and acid; remake solution
Laboratory Control Samples Spiked clean material containing all or defined analytes processed through the entire preparation and analysis process according to EPA SW846 6010C	*Two* per batch of sample exceeding no more than 20 samples per batch	%R 100%±30% or as defined by developing recovery studies also is dependent on sample preparation methodology. Criteria according to EPA SW846 6010C	Re-run and if fails again, review all affiliated QA/QC and if deemed necessary by QAM re-prepare and analyze all affiliated samples
Method Blank (MB) Sample composed of all the reagents and process through the entire method	One per batch of sample exceeding no more than 20 samples per batch	No detected compounds exceeding 1/2 the lower reporting limit	Qualify reported data
Lower Reporting Limit (LRL) Reanalyze the lowest concentration calibration standard as a sample	Once per day per metal	100 ± 50%	Internal MCLinc requirement – Review with QAM

*For Air and Wipes, See AIHA Criteria

Attachment 1



ICP Metals
CALIBRATION SOLUTION STANDARD #1
PREPARATION LOG

Prepared By: _____ Date Prepared: 7/17/18 Exp. Date: 1/18/2019

Matrix: 5% HNO3 / 5% HCl

Analyte	Conc. (ug/ml)		Standard ID	032218 ICP-STD #5	032218 ICP-STD #1	032218 ICP-STD #2	032218 ICP-STD #3	032218 ICP-STD #4
			Final Vol (ml)	1000	1,000	500	500	50
			STD 5 Transfer vol.(ml)	4.000	10.000	25.000	25.00	
ICP0461 Exp. 10/2018				5.00				
Na*	1,000	ug/L		5,000	20	100	250	2,500
Al	1,000	ug/L		5,000	20	100	250	2,500
Ca	1,000	ug/L		5,000	20	100	250	2,500
Mg	1,000	ug/L		5,000	20	100	250	2,500
K	1,000	ug/L		5,000	20	100	250	2,500
Cr	500	ug/L		2,500	10	50	125	1,250
Ni	500	ug/L		2,500	10	50	125	1,250
Ag	500	ug/L		2,500	10	50	125	1,250
Zn	500	ug/L		2,500	10	50	125	1,250
ICP0427 Exp. Date 6/2019				2.00				
As	1,000	ug/L		2,000	8	40	100	1,000
Tl	1,000	ug/L		2,000	8	40	100	1,000
Cd	500	ug/L		1,000	4	20	50	500
Pb	1000	ug/L		2,000	8	40	100	1,000
Se	500	ug/L		1,000	4	20	50	500
ICP0468 Exp. Date 04/2024				5.250				
Se	1,000	ug/L		5,250	21	105	263	2,625
Total Se	Total	ug/L		6,250	25	125	313	3,125
ICP0455 Exp. Date 08/2024				10.50				
Pb	1,000	ug/L		10,500	42	210	525	5,250
Total Pb		ug/L		12,500	50	250	625	6,250
ICP0443 Exp. Date 08/2019				10.00				
Fe	1,000	ug/L		10,000	40	200	500	5,000
Ba	100	ug/L		1,000	4	20	50	500
Mn	100	ug/L		1,000	4	20	50	500
Be	100	ug/L		1,000	4	20	50	500
Cu	100	ug/L		1,000	4	20	50	500
Co	200	ug/L		2,000	8	40	100	1,000
V	100	ug/L		1,000	4	20	50	500
ICP0420 Exp. Date 07/2021				3.00				
Co	1,000	ug/L		3,000	12	60	150	1,500
Total Co	Total	ug/L		5,000	20	100	250	2,500
ICP0449 Exp. Date 4/2023				9.00				
Cu	1,000	ug/L		9,000	36	180	450	4,500
Total Cu	Total	ug/L		10,000	40	200	500	5000
ICP0419 Exp. Date 08/2023				4.00				
V	1,000	ug/L		4,000	16	80	200	2,000
Total V	Total	ug/L		5,000	20	100	250	2,500
ICP0437 Exp. Date 05/2024				5.0				
U	1,000	ug/L		5,000	20	100	250	2,500
ICP0448 Exp. Date 02/2019				5.00				
Ti	1,000	µg/L		5,000	20	100	250	2,500
Li	300	µg/L		1,500	6	30	75	750
Mo	300	µg/L		1,500	6	30	75	750
Sr	200	µg/L		1,000	4	20	50	500
P	1000	µg/L		5,000	20	100	250	2,500
ICP0469 Exp. Date 02/2024				5.0				
P	1,000	µg/L		5,000	20	100	250	2,500
Total P		µg/L		10,000	40	200	500	5,000
ICP0450 Exp. Date 09/2022				6.0				
B	1,000	ug/L		6,000	24	120	300	3,000
ICP0423 Exp. Date 03/2021				10.00				
Sn	1,000	ug/L		10,000	40	200	500	5,000
ICP0446 Exp. Date 01/2019				5.00				
Th	1,000	ug/L		5,000	20	100	250	2,500
ICP0458 Exp. Date 10/2023				2.50				
Zr	1,000	ug/L		2,500	10	50	125	1,250
ICP0436 Exp. Date 05/2021				12.50				
Si	1,000	ug/L		12,500	50	250	625	6,250
ICP0444 Exp. Date 01/2019				2.50				
Nb	1,000	ug/L		2,500	10	50	125	1,250
ICP0438 Exp. Date 6/2023				12.50				
Sb	1,000	ug/L		12,500	50	250	625	6,250

Room#		Balance/ Weight Set	Check Mass, g	Weight #1, g	Weight #2, g	Average Wt., g	Tolarance Acceptance
Humidity							± 2%
Temperature							± 2%

Pipette s/n	Volumn, mL	Weight #1, g	Weight #2, g	Average Wt., g	Tolarance Acceptance
					± 2%
					±2%
					±2%
					±2%
					±2%
					±2%

ICP Metals

ICV/CCV - 2nd SOURCE STANDARDS PREPARATION LOG

Prepared By: _____ Date Prepared: 7/18/2018 Exp. Date: 1/14/2019

Matrix: 5% HNO3 / 5% HCl

Stock Solution		Standard ID	071118-SS1						
Analyte	Conc. (ug/ml)	Final Vol (ml)	1000	Balance/ Weight Set	Check Mass, g	Weight #1, g	Weight #2, g	Average Wt., g	Tolarance Acceptance
ICP0472 Exp. Date 05/2020		Transfer vol.(ml)	1.00						± 2%
Pb	500	ug/L	500						± 2%
Se	200	ug/L	200						
Cd	150	ug/L	150	Pipette s/n	Volumn, mL	Weight #1, g	Weight #2, g	Average	Tolarance
Mn	100	ug/L	100						± 2%
Be	50	ug/L	50						±2%
Zn	150	ug/L	150						±2%
ICP0413 Exp. Date 07/2019		Transfer vol.(ml)	2.00						±2%
Fe	10000	ug/L	20,000						±2%
Ba	100	ug/L	200						±2%
Co	100	ug/L	200						
Cu	100	ug/L	200	Room#					
V	100	ug/L	200	Humidity					
ICP0451 Exp. Date 08/2020		Transfer vol.(ml)	1.00	Temperature					
As	500	ug/L	500						
Mo	100	ug/L	100						
ICP0473 Exp. Date 01/2022		Transfer vol.(ml)	4.00						
Ca	1000	ug/L	4,000						
K	400	ug/L	1,600						
Al	200	ug/L	800						
Na	200	ug/L	800						
Li	100	ug/L	400						
Cr	20	ug/L	80						
Ni	20	ug/L	80						
Sr	10	ug/L	40						
ICP0452 Exp. Date 08/2020		Transfer vol.(ml)	2.00						
Mg	1000	ug/L	2,000						
Sb	200	ug/L	400						
Tl	200	ug/L	400						
Ag	50	ug/L	100						
ICP0411 Exp. Date 01/2022		Transfer vol.(ml)	0.50						
U	1000	ug/L	500						
ICP0474 Exp. Date 7/2021		Transfer vol.(ml)	0.800						
B	100	ug/L	80						
Mo	1,000	ug/L	800						
Total Mo		ug/L	900						
Si	1,000	ug/L	800						
ICP0471 Exp. Date 09/2024		Transfer vol.(ml)	1.000						
Li	1,000	ug/L	1,000						
Total Li		ug/L	1400						
ICP0409 Exp. Date 03/2021		Transfer vol.(ml)	0.500						
Sn	1,000	ug/L	500						
ICP0390 Exp. Date 09/2020		Transfer vol.(ml)	0.500						
Th	1,000	ug/L	500						
ICP0445 Exp. Date 01/2019		Transfer vol.(ml)	0.500						
Zr	1,000	ug/L	500						
ICP0447 Exp. Date 01/2019		Transfer vol.(ml)	1.000						
P	1,000	ug/L	1000						
ICP0410 Exp. Date 09/2021		Transfer vol.(ml)	0.500						
Sr	1,000	ug/L	500						
Total Sr		ug/L	1440						
ICP0470 Exp. Date 06/2024		Transfer vol.(ml)	0.500						
Ti	1,000	ug/L	500						
ICP0355 Exp. Date 05/2020		Transfer vol.(ml)	0.800						
Nb	1,000	ug/L	800						

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE	
Inductively Coupled Plasma – Mass Spectrometry Element/Metals *Including Tc^{99}* Sample Preparation and Analysis: Materials and Chemistry Laboratory Inc.	Approved: MCLinc President — Date: 7/19/18 Quality Assurance Officer — Date: 7/18/18

1.0 PURPOSE

This document describes the procedures to determine elements/metals in properly prepared samples by inductively-coupled plasma – mass spectrometry (ICP-MS) based upon USEPA SW-846 Method 6020A-*C*. This document is also meant to determine elements/metals in properly prepared samples by ICP-MS based upon USEPA ORD Method 200.8. Any additional or slightly different requirements in Method 200.8 are given in Appendix 1.
The procedure for Tc^{99} is in Section 13.10 - 13.11.

2.0 SCOPE AND APPLICATION

2.1 Inductively coupled plasma – mass spectrometry (ICP-MS) determines trace elements/metals in solution. This method can be used for all elements/metals in Table 1. All matrices excluding filtered acidified groundwater samples and including other aqueous samples, industrial and organic wastes, soils, sludges, sediments and other solid wastes require digestion prior to analysis. Both non-digested and digested samples must be matrix-matched with the same type and concentrations of acids as found within the calibration standards.

Table 1 lists the elements that can be analyzed by this method. Elements other than those can be analyzed by this method if performance at the concentration levels of interest is demonstrated.

2.2 Users of this method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to the use of the method for analysis.

2.3 Use of this method is restricted to chemist/qualified operators who are knowledgeable in the correction of chemical and physical interferences described in this method. They must also have been appropriately trained on the instrumentation and its software.

2.4 An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standard elements are ^{6}Li, ^{45}Sc, ^{74}Ge, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li so that interference from lithium native to the sample is minimized. The listed elements can be used as internal standards and other elements may need to be used as internal standards when samples contain significant native amounts of the recommended internal standard elements. MCL is routinely using ^{6}Li, ^{45}Sc, ^{74}Ge, ^{115}In, ^{159}Tb and ^{209}Bi.

3.0 RESPONSIBILITIES

The **MCLinc Project Manager** is responsible for assuring that project QA/QC is clearly defined to the ICP operator and sample preparation analyst and any health and safety issues are understood.

The **MCLinc Analyst** is responsible for routine operation, inventory of all required materials, upkeep of equipment, reviewing and reporting of results and the housekeeping of the work area associated with the equipment.

The **MCLinc Operations Manager** represents the first level of management, provides project oversight and is responsible for supplying the resources for proper upkeep of the required instrumentation.

4.0 SUMMARY OF METHOD

4.1 Prior to analysis, samples, except for filtered and acid preserved groundwater samples, must be digested using appropriate sample preparation methods. This includes all total and "acid-leachable" samples.

4.2 This method describes the multi-element determination of analytes by ICP-MS in environmental samples. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed, and valid corrections applied, or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents and constituents of the sample matrix.

5.0 DEFINITIONS

Applicable definitions are located throughout this SOP.

6.0 INTERFERENCES

6.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.

6.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. The instrument software used corrects for isobaric and doubly-charged ion interferences. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Examples include $^{75}ArCl^+$ ion on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature, the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1%) counting statistics. Because the ^{35}Cl natural abundance of 75.77% is 3.13 times the ^{37}Cl abundance of 24.23%, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ at m/z 75 is a negligible 0.06% of the $^{40}Ar^{35}Cl^+$ signal):

Corrected arsenic signal (using natural isotopes abundances for coefficient approximations)

= (m/z 75 signal) – (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal)

where the final term adjusts for any selenium contribution at 77 m/z,

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes).

Similarly,

Corrected cadmium signal (using natural isotopes abundances for coefficient approximations)

= (m/z 114 signal) – (0.027) (m/z 118 signal) – (1.63) (m/z 108 signal),

where last 2 terms adjust for any $^{114}Sn^+$ or $^{114}MoO^+$ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108 but use of m/z 111 for Cd is even subject to direct ($^{94}ZrOH^+$) and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients could be improved. The most appropriate coefficients for a particular instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 %) counting precision.

The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary with operating conditions. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. For example, this type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed gas plasmas have been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met. Common isobaric, double charge and oxide formation corrections are included in the instrument software and are performed automatically.

6.3 Additionally, solid phase chelation may be used to eliminate isobaric interferences from both elemental and molecular sources. An on-line method has been demonstrated for environmental waters such as sea water, drinking water and acid-digested samples. Acid-digested samples refer to samples digested by methods similar to SW 846 methods 3052, 3051, 3050, or 3015. Samples with percent levels of iron and aluminum should be avoided. The method also provides a procedure for preconcentration to enhance detection limits simultaneously with elimination of isobaric interferences. The method relies on chelating resins such as imminodiacetate or other appropriate resins and selectively concentrates the elements of interest while eliminating interfering elements from the sample matrix. By eliminating the elements that are direct isobaric interferences or those that form isobaric interfering molecular masses, the mass region is simplified, and these interferences cannot occur. The method has been proven effective for the certification of standard reference materials and validated using SRMs. The method has the potential to be used on-line or off-line as an effective sample preparation method specifically designed to address interference problems.

6.4 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to

correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When intolerable physical interferences are present in a sample, a significant suppression of the internal standard signals (to less than 30% of the signals in the calibration standards) will be observed. Dilution of the sample fivefold (1+4) will usually eliminate the problem (see Section 13.7).

6.5 Memory interferences or carry-over can occur when there are large concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler or skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

7.0 SAFETY

7.1 General laboratory protection (safety glasses, lab coat and disposable latex/nitrile gloves) should be worn at all times when handling standards or samples.

7.2 Acid solutions may pose potential health risks. Extreme care should be utilized when handling these solutions.

8.0 EQUIPMENT AND SUPPLIES

8.1 Inductively coupled plasma-mass spectrometer such as Perkin Elmer Elan 9000 with: (See Appendix 1 for Method 200.8 requirements)

8.1.1 Capability of providing resolution, better than or equal to 1.0 amu at 10% peak height.

8.1.2 Mass range from at least 6 to 240 amu.

8.1.3 Data system that has corrections for common isobaric, double charge and oxide interferences and the application of the internal standard technique.

8.2 Mass flow controller for argon nebulizer gas supply.

8.3 Peristaltic pump for delivery of sample to nebulizer.

8.4 Argon gas supply, high purity.

9.0 REAGENTS AND STANDARDS

9.1 Acids used in the preparation of standards and for sample processing must be of high purity. Nitric acid at less than 2% (v/v) is required for ICP-MS to minimize damage to the interface and to minimize isobaric molecular-ion interferences with the analytes. Many more molecular-ion interferences are observed when hydrochloric and sulfuric acids are used. Concentrations of antimony and silver between 50-500 µg/L require 1% (v/v) HCl for stability. For concentrations above 500 µg/L Ag, additional HCl will be needed. Consequently, accuracy of analytes requiring significant chloride molecular ion corrections (such as As and V) will degrade.

9.2 Reagent Water: All references to reagent water in the method refer to *ASTM Type II (>1.0* Mohms-cm) *water.*

9.3 Standard stock solutions are either purchased commercially as certified standards or prepared from ultra-high purity grade chemicals or metals (99.99% or greater purity).

9.4 Mixed calibration standard solutions are prepared by diluting the stock standard solutions to levels in the linear range for the instrument in a solvent consisting of 1% (v/v) HNO_3 in reagent water. The calibration standard solutions must contain a suitable concentration of an appropriate internal standard for each analyte. Internal standards may be added on-line at the time of analysis using a second channel of the peristaltic pump and an appropriate mixing manifold. Generally, an internal standard should be no more than 50 amu removed from the analyte. Recommended internal standards include ^{6}Li, ^{45}Sc, ^{74}Ge, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, ^{209}Bi. MCL is routinely using ^{6}Li, ^{45}Sc, ^{74}Ge, ^{115}In, ^{159}Tb and ^{209}Bi.

9.5 Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine possible spectral interferences or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. Fresh mixed standards should be prepared, as needed with the realization that concentrations can change on aging. Calibration standards must be initially verified using a quality control standard (see Section 9.7).

9.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The method blank is used to monitor for possible contamination resulting from the sample preparation procedure. The instrument blank is used to flush the system between all samples and standards.

9.6.1 The calibration blank consists of the same concentration(s) of the same acid(s) used to prepare the final dilution of the calibrating solutions of the analytes [often 1% HNO_3 (v/v) in reagent water] along with the selected concentrations of internal

standards such that there is an appropriate internal standard element for each of the analytes.

9.6.2 The method (or preparation) blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions.

9.6.3 The instrument blank consists of 1% to 3% HNO3 (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples. If mercury is to be analyzed, the instrument blank should also contain 2 mg/L $AuCl_3$ solution.

9.7 The interference check solutions A and AB (ICS-A, ICS-AB) are prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. Chloride in the ICS provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^{+}$ on $^{51}V^{+}$ and $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits.

ICP-MS Interference Check Standards A (ICP0298) and AB (ICP0299) are to be analyzed with all runs that include any of the following metals: As, Cd, Cr, Co, Cu, Mn, Ni, Se, Ag, Ti, V, Zn. Dilute the purchased stock Interference Check Standards 10X each in separate vessels to yield concentrations in Table 2. Solution A contains the interference metals (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, Ti) and solution AB contains the interferent metals along with the analytes listed above that can experience isobaric interferences on the ICP-MS. After the closing CCV of analysis run, analyze the Interference Check Standards for all of the analytes listed above that are included for the samples, with the calibration curve used for the analysis run. End with another closing CCV. Percent recovery for each analyte is to be reported to the QAM.

9.8 The quality control standard is the second source standard used for initial calibration verification (ICV) which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard near the midpoint of the linear range at a concentration other than that used for instrument calibration. An independent standard is defined as a standard composed of the analytes from a source different from those used in the standards for instrument calibration or from the same vendor but a different lot.

9.9 Continuing Calibration Verification (CCV) – The CCV is prepared from the same source and same acid matrix as calibration standards at mid-range concentration. The CCV is analyzed after every ten (10) samples including preparation QC samples such as LCS and MB, and at the end of analysis.

9.10 Mass spectrometer tuning solution is a solution containing elements representing all of the mass regions of interest to verify that the resolution and mass calibration of the instrument are within the required specifications (see Section 13.4). This solution is also used to verify that the

instrument has reached thermal stability. For the Elan 9000 ICP-MS, the tuning solution contains 10 µg/L Be, Mg, Co, Rh, In, Ba, Ce, Pb and can also contain Cu, Cd and U.

9.11 Dual detector cross-calibration solution is required for the Elan 9000 ICP-MS for the calibration of the detector in the crossover range between the pulse and analog ranges. This solution will contain 250 µg/L each of Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Tl, Th, U, V, Zn, Na, Ca, Mg, K, Fe, Sc, Y, In, Rh, Tb, Ho, and Bi and 1250 µg/L Ge.

10.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

10.1 Sample collection procedures should address all considerations described in the Quality Assurance Project Plan, (MCL-7701).

10.2 Only polypropylene or fluorocarbon containers are suitable for collection of samples for Method 6020A.

10.3 Aqueous samples should be preserved with 1:1 HNO_3 to a pH <2.

11.0 QUALITY CONTROL

11.1 The type and frequency of the quality control program will be defined by the project. Depending upon the project defined program, the following quality control data, as defined in Table 3 may be included. The resulting data should be maintained and be available for easy reference or inspection.

11.2 Instrument detection limits (IDLs) are a useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. IDLs in µg/L can be estimated as the mean of the blank result plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. (Use zero for the mean if the mean is negative). Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector and/or as designated by a project.

11.3 The intensity of all internal standards must be monitored for every analysis. If the intensity of any internal standard in a sample falls below 30% of the intensity of that internal standard in the initial calibration standard, a significant matrix effect must be suspected. Under these conditions, the detection limit has degraded, and the correction ability of the internal standardization technique becomes questionable. The following procedure is used: First, make sure that the instrument has not just drifted by observing the internal standard intensities in the nearest clean matrix (calibration blank). If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-

standard intensities rise above the 30% limit. Reported results must be corrected for all dilutions.

11.4 To obtain data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. For example, tungsten oxides can be very difficult to distinguish from mercury isotopes. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections is required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Unsuspected interferences may be detected by adding pure major matrix components to a sample to observe any impact on the analyte signals. When an interference source is present, the sample elements impacted must be flagged to indicate (a) the percentage interference correction applied to the data or (b) an uncorrected interference by virtue of the elemental equation used for quantitation. The isotope proportions for an element of molecular-ion cluster provide information useful for quality assurance.

NOTE: Only isobaric elemental, molecular and doubly charged interference corrections which use the observed isotopic-response ratios or parent-to-oxide ratios (provided an oxide internal standard is used as described in Section 6.2) for each instrument system are acceptable corrections for use in this method.

11.5 Dilution test (DT) (serial dilution): This test may be applied for unusual matrices. If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times greater than the concentration in the method blank, refer to Section 9.5.2), an analysis of fivefold (1+4) dilution must agree within ± 10% of the original determination. If not, an interference effect must be suspected. One dilution test is be included for each 20 samples (or less) of each matrix in a batch.

11.6 Post-digestion spike addition (PDSA): An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125% of the known value or within the laboratory derived acceptance criteria. The spike addition should be based on the indigenous concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, the sample must be diluted and reanalyzed to compensate for the matrix effect. Results must agree to within 10% of the original determination. The use of a standard-addition analysis procedure may also be used to compensate for this effect.

11.7 There will be two different laboratory control samples (LCS) analyzed with each batch of 20 or fewer samples using the same sample preparations, analytical methods and QA/QC procedures employed for the test samples:

11.7.1 LCS shall be prepared and analyzed in duplicate and are solutions spiked to yield concentrations in the low to midrange of the calibration curve for each target analyte. The acceptance criterion is 100±30%.

11.7.2 LCSL (low) may be requested by the Quality Manager for special projects and are a solution spiked to yield concentrations 2-4x the concentration of the lowest calibration standard for each target analyte. The acceptance criterion will be determined based on historical results of ICP-MS.

11.8 Check the instrument calibration by analyzing appropriate quality control solutions as follows:

11.8.1 Check instrument calibration using a calibration blank and the ICV.

11.8.2 Verify calibration after every 10 analytical samples with the CCV and the calibration blank and after the last sample.

11.8.3 The results of the ICV and CCV must agree within ±10% of the expected value. If not, terminate the analysis, correct the problem, and recalibrate the instrument. Any sample analyzed under an out-of-range calibration must be reanalyzed.

11.8.4 The results of the calibration blank must be less that 3 times the current IDL for each element. If this is not the case, the reason for the out-of-range condition must be found and corrected and affected samples reanalyzed.

11.9 Verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the interference check solutions. The analyst should be aware that precipitation from the ICS solutions may occur, particularly with silver.

11.10 The analysis of duplicate samples is project specific at the rate of one duplicate for every 20 or less samples. The acceptance criterion is ±20%.

11.10.1 The relative percent difference (RPD) between duplicate determinations is calculated as follows:

$$RPD = 100 \times \frac{|D_1 - D_2|}{(D_1 + D_2)/2}$$

Where:
RPD = relative percent difference
D_1 = initial sample concentration
D_2 = duplicate sample concentration

A control limit of 35% RPD should not be exceeded for analyte values greater that 100 times the instrumental detection reporting limit. If this limit is exceeded, the reason for this situation

must be investigated and corrected if appropriate, and if any samples are affected, they should be reanalyzed.

11.11 Lower Reporting Limit Verification – After the ICV is analyzed and verified, the lower reporting limit (LRL) is verified by analyzing the lowest concentration calibration standard as a sample. The percent recovery should be ±30%.

11.12 A matrix spike (MS) and matrix spike duplicate (MSD) is project specific and is prepared at the frequency of one for every batch of 20 or fewer samples. Acceptance criterion is ±25%.

11.12.1 The % Recovery is calculated as follows:

$$\%R = 100 \times \frac{(MS - S)}{TV}$$

Where:
%R = % recovery
MS = concentration in matrix spike
S = concentration in sample
TV = theoretical concentration of the spike

12.0 CALIBRATION AND STANDARDIZATION

12.1 Conduct mass calibration and resolution checks in the mass regions of interest with the tuning solution. The mass calibration and resolution parameters are required criteria which must be met prior to any samples being analyzed. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be less that 0.9 amu full width at 10% peak height.

12.2 Calibrate the instrument for the analytes of interest (recommended isotopes for analytes are given in Table 3), using the calibration blank and at least a single initial calibration standard according to the instrument manufacturer's procedure. Flush the system with the rinse blank between each standard solution. Use the average of at least 3 integrations for both calibration and sample analyses.

NOTE: Analysts have noted improved performance in calibration stability if the instrument is exposed to the interference check solution after cleaning sampler and skimmer cones. Improved performance is also realized if the instrument is allowed to rinse for 5 to 10 minutes before the calibration blank is run.

12.3 All masses which could affect data quality should be monitored to determine potential effects from matrix components on the analyte peaks. The recommended isotopes to be monitored are listed in Table 3.

12.4 Immediately after the calibration has been established, the calibration must be verified and documented for every analyte by the analysis of the ICV solution. When measurements exceed

±10% of the accepted value, the analyses must be terminated, the problem corrected, the instrument recalibrated, and the new calibration verified with ICV standard. Any samples analyzed under an out-of-range calibration must be reanalyzed. During the course of an analytical run, the instrument may be "resloped" or recalibrated to correct for instrument drift, but resloping must not be used as an alternative to reanalyzing samples following an unacceptable QC sample, such as a CCV. A recalibration must then be followed immediately by a new analysis of an ICV and a calibration blank before any further samples may be analyzed.

13.0 PROCEDURE

13.1 Sample Preparation

Samples should be prepared according to Acid Digestion for Metals (MCL-7746).

13.2 Initiate appropriate operating configuration of the instrument computer according to the instrument manufacturer's instructions.

13.3 Set up the instrument with the proper operation parameters according to the instrument method file defining reporting units (µg/L for liquid and mg/Kg for solids), calibration parameters, ICV and CCV frequency, acceptance criteria and corrective actions, ICS and LCS criteria. In the method file, also define by element the isotope(s) to be used and the specific plasma operational parameters.

13.4 Set up the Workspace file defining the analysis method, sample file, calibration file, data acquisition file, and the instrumental conditions file, which includes tuning, lens calibration and nebulizer calibration.

13.5 Operating conditions: The analyst should follow the instructions provided by the instrument manufacturer. Allow at least 30 minutes for the instrument to equilibrate before analyzing any samples. This must be verified by analyzing a tuning solution (Section 9.8) at least 4 times with relative standard deviations of ≤5% for the analytes contained in the tuning solution.

13.6 Calibrate the instrument following the procedure outlined in Section 12.0.

13.7 The sample run sequence will have an instrument blank immediately following each CCV as shown in this sequence example:

Calibration Blank (Section 9.5.1)
Calibration Standards, Lowest Concentration to Highest (Section 9.4)
ICV (Section 9.7)
LRL (Section 11.11)
Instrument Blank (Section 9.5.3)
ICS-A (Section 9.6)

ICS-AB (Section 9.6) LCSL (Section 11.7.2)
LCSN (Section 11.7.1)
Method Blank (Section 9.5.2)
Up to 10 Samples, including Duplicates and MS/MSD (MS/MSD only if required by project)
CCV (Section 9.7)
Instrument Blank
Up to 10 Samples, including Duplicates
PDSA (Section 11.6)
DT (Section 11.5)
CCV
Instrument Blank

The method preparation quality control samples: LCSL, LCSN, Method Blank, PDSA and DT are counted in the run sequence as samples, i.e., as one of the 10 samples between each ICV – CCV or CCV- CCV sequence.

13.8 Flush the system with the instrument blank solution (Section 9.5.3) until the signal levels return to the levels of quantitation defined in the method (usually about 30 seconds) before the analysis of each sample (see Section 12.3). Nebulize each sample until a steady-state signal is achieved (usually about 30 seconds) prior to collecting data. Analyze the CCV solution (Section 9.7) and calibration blank (Section 9.5.1) at a frequency of at least once for every 10 analytical samples. Flow injection systems can be used as long as they meet the performance criteria of this method.

13.9 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte (or species needed for a correction) or measure an alternate but less abundant isotope. The linearity of the alternate mass must be confirmed by appropriate calibration (see Section 12.2 and 12.4). Alternatively, apply solid phase chelation chromatography to eliminate the matrix interference as described in Section 6.3.

13.10 Final Prep and Analysis of Tc^{99} by ICP/MS. Prior to analysis, samples are prepared according to MCL-7754, Section 5.1 to 5.1.7. In addition to this, the following column separation procedure using Eichrom TEVA resin must be performed on the prepared sample.

13.11 Column Separation

13.11.1 For each sample aliquot analyzed, place a 2mL TEVA column in a column rack.
13.11.2 Place a beaker or tray below each column, remove the bottom plug from each column and allow each column to drain.
13.11.3 Pipet 5mL of 0.1m HNO3 into each column to condition the resin and allow to drain.
13.11.4 Transfer each sample aliquot into the appropriate column and allow to drain.
13.11.5 Rinse each sample container with 50mL 0.1M HNO3 and then transfer the rinse to the appropriate column. Allow the rinse solution to drain.
13.11.6 Discard all drain solutions collected to this point.
13.11.7 Place clean, labeled sample containers below each column.

13.11.8 Pipet 20mL of 11M HNO3 into each column to elute the Tc^{99}. Collect this solution for analysis.

13.11.9 The sample is now ready for analysis per Sections 13.3 – 13.9.

14.0 DATA ANALYSIS AND CALCULATIONS

14.1 The quantitative values shall be reported in appropriate units, such as micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/Kg) for solid samples. If dilutions were performed, the appropriate corrections must be applied to the sample values. All calculations must include appropriate interference corrections (see Section 6.2 for examples), internal-standard normalization, and the summation of signals at 206, 207 and 208 m/z for lead (to compensate for any differences in the abundances of these isotopes between samples and standards).

14.2 For dissolved metals analyses:

$$\mu g/L = C \times DF$$

Where:
C = Sample concentration (µg/L)
DF = Dilution factor

14.3 For digested aqueous samples:

$$\mu g/L = \frac{C \times DF \times V}{W}$$

Where:
C = Digestate concentration (µg/L)
DF = Dilution factor
V = Final volume in L after sample preparation
W = Initial volume in L of sample used for sample preparation

14.4 Soil/Solid concentrations may be reported on the basis of the dry weight of the sample (a separate determination of % total solids must be performed):

$$\mu g/g \text{ (dry weight)} = \frac{C \times DF \times V \times S}{W}$$

Where:
C = Digest concentration (µg/L)
DF = Dilution factor
V = Final volume in L after sample preparation
W = Weight in g of wet sample
S = 100 / % solids

14.5 Air filter sample concentrations may be reported as total µg or total mg per filter or if the air volume sampled is given, as mg/cubic meter (mg/m^3):

$$\text{Total } \mu g = C \times DF \times V$$
$$\text{Total mg} = \mu g / 1000$$
$$mg/m^3 = \frac{C \times DF \times V}{\text{Air volume in } m^3}$$

Where:
C = Digest concentration (µg/L)
DF = Dilution factor
V = Final volume in L after sample preparation

15.0 METHOD PERFORMANCE

15.1 Refer to Table 1 for method detection limit information.

16.0 POLLUTION PREVENTION

16.1 To minimize hazardous materials generated with this method, minimal quantities of samples are digested (50-100 mL final digestate volume) and minimal quantities of standards are prepared.

17.0 WASTE MANAGEMENT

It is the laboratory's responsibility to comply with all applicable federal, state and local regulations governing waste management.

TABLE 1

ELEMENTS THAT CAN BE ANALYZED BY ICP-MS ACCORDING TO EPA METHOD 6020A AND DETECTION LIMITS

Element	Symbol	Lower Reporting Limit (µg/L)	Instrument Detection Limit (µg/L)	Method Detection Limit Soil (µg/g)	Method Detection Limit Water (µg/L)
Aluminum	Al	1.0	TBD*	TBD*	TBD*
Antimony	Sb	0.05	TBD	TBD	TBD
Arsenic	As	0.02	TBD	TBD	TBD
Barium	Ba	0.1	TBD	TBD	TBD
Beryllium	Be	0.02	TBD	TBD	TBD
Cadmium	Cd	0.02	TBD	TBD	TBD
Calcium	Ca	1.0	TBD	TBD	TBD
Chromium	Cr	0.05	TBD	TBD	TBD
Cobalt	Co	0.05	TBD	TBD	TBD
Copper	Cu	0.05	TBD	TBD	TBD
Iron	Fe	1.0	TBD	TBD	TBD
Lead	Pb	0.05	TBD	TBD	TBD
Magnesium	Mg	1.0	TBD	TBD	TBD
Manganese	Mn	0.1	TBD	TBD	TBD
Mercury	Hg	TBD	TBD	TBD	TBD
Nickel	Ni	0.05	TBD	TBD	TBD
Potassium	K	1.0	TBD	TBD	TBD
Selenium	Se	0.02	TBD	TBD	TBD
Silver	Ag	0.05	TBD	TBD	TBD
Sodium	Na	1.0	TBD	TBD	TBD
Thallium	Tl	0.02	TBD	TBD	TBD
Uranium	U	0.01	TBD	TBD	TBD
Vanadium	V	0.02	TBD	TBD	TBD
Zinc	Zn	0.1	TBD	TBD	TBD

* To be determined and placed in the instrument QA files

TABLE 2

RECOMMENDED INTERFERENCE CHECK SAMPLE COMPONENTS AND CONCENTRATIONS

Solution Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	100.0	100.0
Ca	300.0	300.0
Fe	250.0	250.0
Mg	100.0	100.0
Na	250.0	250.0
P	100.0	100.0
K	100.0	100.0
S	100.0	100.0
C	200.0	200.0
Cl	2000.0	2000.0
Mo	2.0	2.0
Ti	2.0	2.0
As	0.0	0.100
Cd	0.0	0.100
Cr	0.0	0.200
Co	0.0	0.200
Cu	0.0	0.200
Mn	0.0	0.200
Hg	0.0	0.020
Ni	0.0	0.200
Se	0.0	0.100
Ag	0.0	0.050
V	0.0	0.200
Zn	0.0	0.100

TABLE 3

RECOMMENDED ISOTOPES FOR SELECTED ELEMENTS

Element of Interest	**Mass**
Aluminum	**27**
Antimony	121, **123**
Arsenic	**75**
Barium	138, 137, 136, **135**, 134
Beryllium	**9**
Bismuth (IS)	209
Cadmium	**114**, 112, **111**, 110, 113, 116, 106
Calcium (I)	42, 43, **44**, 46, 48
Chlorine (I)	35, 37, (77, 82)[a]
Chromium	**52**, **53**, **50**, 54
Cobalt	**59**
Copper	**63**, **65**
Holmium (IS)	165
Indium (IS)	**115**, 113
Iron (I)	**56**, **54**, **57**, 58
Lanthanum (I)	139
Lead	**208**, **207**, **206**, 204
Lithium (IS)	6[b], 7
Magnesium (I)	24, **25**, **26**
Manganese	**55**
Mercury	202, **200**, 199, 201
Molybdenum (I)	98, 96, 92, **97**, 94, (108)[a]
Nickel	58, **60**, 62, **61**, 64
Potassium (I)	**39**
Rhodium (IS)	103
Scandium (IS)	45
Selenium	80, **78**, **82**, **76**, **77**, 74
Silver	**107**, **109**
Sodium (I)	**23**
Terbium (IS)	159
Thallium	**205**, 203
Uranium	**238**
Vanadium	**51**, **50**
Tin (I)	120, **118**
Yttrium (IS)	89
Zinc	64, **66**, **68**, **67**, 70

NOTE: EPA Method 6020 is recommended for only those analytes listed in Table 1. Other elements are included in this table because they are potential interferents (labeled I) in the determination of recommended analytes, or because they are commonly used internal standards (labeled IS). Isotopes are listed in descending order of natural abundance. The most generally useful isotopes are underlined and in boldface, although certain matrices may require the use of alternative isotopes.

a These masses are also useful for interference correction (Section 6.2)

b Internal standard must be enriched in the 6Li isotope (Section 2.4)

TABLE 4
QUALITY ASSURANCE/QUALITY CONTROL

Description	Frequency	Acceptance Criteria	Corrective Action
Instrument Detection Limits (IDLs)	Per Section 11.2	See "Instrument Blank"	
Tuning Solution	Daily	RSD of ≤5%; Mass calibration <0.1 amu from true value; <0.9 amu full width at 10% peak height	Allow to warm up 30 minutes more
Calibration Blank	Before analysis of calibration curve standards	<3X current IDL	Allow instrument blank to flush system for 10-15 minutes and reanalyze
Calibration Curve Defining linear calibration range. 3 points minimum lower and upper defining reporting limits. 3rd point preferably near the lower	Before the analysis of the sample; after instrument repair or maintenance; when an ICV shows an existing calibration curve failed	Correlation Coefficient (R) > 0.998	Check standards, calibration range, operational parameters, Rerun calibration
Initial Calibration Verification Std (ICV) Standard containing all the target analytes prepared from a second source solution	After development of calibration curve	100%R ± 10%	Check both ICV and calibration standards preparation; recalibrate
Continuing Calibration Verification (CCV) Standard prepared from same source as calibration stds.	After every ten (10) samples including QC samples (LCS and MB) and at end of analysis	100%R±20%	Reanalyze CCV; check Preparation and re-prepare if Necessary; recalibrate
Instrument Blank	Immediately following every ICV/CCV	Less than *1/2* the current *LRL* for each element	Check tubing and calibration blank solution; remake solution
Internal Standards	Added to all blanks, calibration standards, samples, QC	In samples, the intensity of all internal standards should be 100±30% of that in the initial calibration solution	Check instrument drift, if drift present, recalibrate; If no drift, dilute sample 5X and reanalyze
Laboratory Control Sample (LCS) Spiked clean material containing all or defined elements processed through the entire preparation and analysis process	Two per batch of samples with a maximum of 20 samples per batch	100%R ± *20%* or as defined by developing recovery studies	Re-prepare if fails review of all related QA/QC, and if necessary re-prepare and reanalyze all associated samples
Method Blank Sample composed of all reagents and processed through entire method	One per batch of samples with a maximum of 20 samples per batch	No analyte concentrations exceeding ½ the lower reporting limit	Qualify reported data
Lower Reporting Limit (LRL) Analysis of lowest concentration calibration standard as a sample	Once per day for each metal analyzed	100%R ± 30%	Internal MCL Inc requirement – Review with QA Manager
MS/MSD	*One set per batch*	*100%±25%*	*Evaluate for matrix effect – QA review*

APPENDIX 1
ADDITIONAL REQUIREMENTS OF EPA METHOD 200.8

TABLE 1-1
ELEMENTS THAT CAN BE ANALYZED BY ICP-MS ACCORDING TO EPA METHOD 200.8 AND DETECTION LIMITS

Element	Symbol	Lower Reporting Limit (µg/L)	Instrument Detection Limit (µg/L)	Method Detection Limit Soil (µg/g)	Method Detection Limit Water (µg/L)
Aluminum	Al	1.0	TBD*	TBD*	TBD*
Antimony	Sb	0.05	TBD	TBD	TBD
Arsenic	As	0.02	TBD	TBD	TBD
Barium	Ba	0.1	TBD	TBD	TBD
Beryllium	Be	0.02	TBD	TBD	TBD
Cadmium	Cd	0.02	TBD	TBD	TBD
Chromium	Cr	0.05	TBD	TBD	TBD
Cobalt	Co	0.05	TBD	TBD	TBD
Copper	Cu	0.05	TBD	TBD	TBD
Lead	Pb	0.05	TBD	TBD	TBD
Manganese	Mn	0.1	TBD	TBD	TBD
Mercury	Hg	TBD	TBD	TBD	TBD
Nickel	Ni	0.05	TBD	TBD	TBD
Selenium	Se	0.02	TBD	TBD	TBD
Silver	Ag	0.05	TBD	TBD	TBD
Thallium	Tl	0.02	TBD	TBD	TBD
Thorium	Th	TBD	TBD	TBD	TBD
Uranium	U	0.01	TBD	TBD	TBD
Vanadium	V	0.02	TBD	TBD	TBD
Zinc	Zn	0.1	TBD	TBD	TBD

18.0 EQUIPMENT AND SUPPLIES

18.1.1 Instrument resolution is 1 amu peak width at 5% peak height.

18.1.2 Mass range from 5-250 amu.

18.1.4 Radio-frequency generator compliant with FCC regulations.

18.1.5 If an electron multiplier detector is used, precautions should be taken, where necessary, to prevent exposure to high ion flux. Changes in instrument response or damage to the multiplier may result with exposure to high ion flux. Samples having high concentrations of elements beyond the linear range of the instrument and with isotopes falling within scanning windows should be diluted prior to analysis.

NOTE: Equipment listed in Method 200.8 Sections 6.2 – 6.10 for the preparation of samples is listed in SOP# MCL-7746, MCL-7752 and MCL-7753.

19.0 QUALITY CONTROL

19.12 Initial Demonstration of Performance

19.12.1 The initial demonstration of performance is used to characterize instrument performance (determination of linear calibration ranges and analysis of quality control samples) and laboratory performance (determination of method detection limits) prior to analyses conducted by this method.

19.12.2 Linear calibration ranges – Linear calibration ranges are primarily detector limited. The upper limit of the linear calibration range should be established for each analyte by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. Care should be taken to avoid potential damage to the detector during this process. The linear calibration range which may be used for the analysis of samples should be judged by the analyst from the resulting data. The upper LDR limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limits must be diluted and reanalyzed. The LDRs should be verified whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

19.12.3 Quality control sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analysis of a QCS. To verify the calibration standards, the determined mean concentration from 3 analyses of the QCS must be within ±10% of the stated QCS value. If the QCS is used for determining acceptable on-going instrument performance, analysis of the QCS prepared to100 µg/L must be within ±10% of the stated value or within the acceptance limits listed in Table 5-1, whichever is greater.

19.13 Assessing Laboratory Performance (mandatory)

19.13.4 Instrument performance – For all determinations the laboratory must check instrument performance and verify that the instrument is properly calibrated on a continuing basis. To verify calibration run the calibration blank and calibration standards as surrogate samples immediately following each calibration routine, after every ten analyses and at the end of the sample run. The results of the analyses of the standards will indicate whether the calibration remains valid. The analysis of all analytes within the standard solutions must be within ±10% of calibration. If the calibration cannot be verified within the specified limits, the instrument must be recalibrated. (The instrument responses from the calibration check may be used for recalibration purposes; however, it must be verified before continuing sample analysis.) If the continuing calibration check is not confirmed within ±15%, the previous 10 samples must be reanalyzed after recalibration. If the sample matrix is responsible for the calibration drift, it is recommended that the previous 10 samples are reanalyzed in groups of five between calibration checks to prevent a similar drift situation from occurring.

19.14 Assessing Analyte Recovery and Data Quality

19.14.1 Sample homogeneity and the chemical nature of the sample matrix can affect analyte recovery and the quality of the data. Taking separate aliquots from the sample for replicate and fortified analyses can in some cases assess the effect. Unless otherwise specified by the data user, laboratory or program, the following laboratory fortified matrix procedure is required.

19.14.2 The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples. In each case, the LFM aliquot must be a duplicate of the aliquot used for sample analysis and for total recoverable determinations added prior to sample preparation. For water samples, the added analyte concentration must be the same as that used in the LFB. For solid samples, the concentration added should be 100 mg/Kg equivalent (200 µg/L in the analysis solution) except silver which should be limited to 50 mg/Kg. Over time, samples from all routine sample sources should be fortified.

19.14.3 Calculate the percent recovery for each analyte, corrected for background concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery of 70-130%. Recovery calculations are not required if the concentration of the analyte added is less than 30% of the sample background concentration.

19.14.4 If recovery of any analyte falls outside the designated range and laboratory performance for that analyte is shown to be in control, the recovery problem

encountered with the fortified sample is judged to be matrix related, not system related. The data user should be informed that the result for that analyte in the unfortified sample is suspect due to either the heterogeneous nature of the sample or an uncorrected matrix effect.

20.0 PROCEDURE

20.1 Sample Preparation

NOTE: If mercury is to be analyzed, the digestion procedure must use mixed nitric and hydrochloric acids through all steps of the digestion. Mercury will be lost if the sample is digested when hydrochloric acid is not present. If it has not already been added to the sample as a preservative, Au should be added to give a final concentration of 2 mg/L to preserve the mercury and to prevent it from plating out in the sample introduction system.

20.1.1 Aqueous Sample Preparation – Dissolved Analytes (from EPA Method 200.8)

20.1.1.1 For the determination of dissolved analytes in ground and surface waters, pipet an aliquot (≥20mL) of the filtered, acid preserved sample into a 50 mL polypropylene (pp) centrifuge tube. Add an appropriate volume of (1+1) nitric acid to adjust the acid concentration of the aliquot to approximate a 1% (v/v) nitric acid solution (e.g., add 0.4mL (1+1) HNO_3 to a 20mL aliquot of sample). If direct addition is being used, add internal standards, cap the tube and mix. The sample is now ready for analysis. Allowance for sample dilution should be made in the calculations.

NOTE: If a precipitate is formed during acidification, transport or storage, the sample aliquot must be treated using the procedure in Section 13.1.2 prior to analysis.

20.1.2 Aqueous Sample Preparation – Total Recoverable Analytes (from EPA Method 200.8)

20.1.2.1 For the "direct analysis" of total recoverable analytes in drinking water samples containing turbidity <1 NTU, treat an unfiltered acid preserved sample aliquot using the sample preparation procedure described in Section 13.1.1.1 while making allowance for sample dilution in the data calculation. For the determination of total recoverable analytes in all other aqueous samples or for preconcentration drinking water samples prior to analysis follow the procedure given in Sections 13.1.2.2 through 13.1.2.8.

20.1.2.2 For the determination of total recoverable analytes in aqueous samples, transfer a 40mL aliquot from a well mixed, acid preserved sample to a 50mL pp digestion tube.

20.1.2.3 Add 2mL (1+1) HNO_3 and 1mL (1+1) HCL to the sample aliquot. Place the tube in a hot block at 95°C and cover with a pp raised watch glass. Reflux sample for 30-60 minutes. Do not boil. Some reduction in sample volume may occur.

20.1.2.4 Allow the sample in digestion tube to cool. Quantitatively transfer the sample solution to a labeled 50mL pp centrifuge tube. Dilute to 50mL with reagent water and mix.

20.1.2.5 Prior to analysis adjust the chloride concentration by pipetting 20mL of the prepared sample solution into a 50mL pp centrifuge tube. If the direct addition method is being used, add appropriate amounts of internal standards. Dilute to 50mL with reagent water and mix. The sample is now ready for analysis. All analyses should be performed as soon as possible after the completed preparation.

20.1.3 Solid Sample Preparation – Total Recoverable Analytes (from EPA Method 200.8)

20.1.3.1 For the determination of total recoverable analytes in solid samples, mix the sample thoroughly to obtain a homogenous aliquot. Weigh 1.0 ± 0.10g of dry sample into a 50mL pp digestion tube.

20.1.3.2 Add 4mL (1+1) HNO_3 and 10mL (1+4) HCL carefully to avoid loss of sample. Place the digestion tube in the hot block (in an appropriate fume hood) at 95°C. Cover with a raised pp watch glass.

20.1.3.3 Reflux the sample for 30 minutes at 95°C. Slight boiling may occur, but vigorous boiling should be avoided to prevent loss of the HCl-H2O azeotrope. Some solution evaporation will occur.

20.1.3.4 Allow the sample to cool and quantitatively transfer the extract to a 100mL volumetric flask. Filter if necessary to remove undissolved solids. Take care to avoid potential contamination from filtration.

20.1.3.5 Prior to analysis, adjust the chloride concentration by pipetting 10mL of the prepared solution into a 50mL pp centrifuge tube. If the direct addition method is being used, add appropriate amounts of internal standards. Dilute to 50mL with reagent water and mix. The sample is now ready for analysis. All analyses should be performed as soon as possible after the completed preparation.

TABLE 5

ACCEPTANCE LIMITS FOR QC CHECK SAMPLE METHOD PERFORMANCE (µg/L)

Element	Symbol	QC Check Sample Concentration (µg/L)	Average Recovery %	Standard Deviation (S_r)	Acceptance Limits (µg/L)
Aluminum	Al	100	100.4	5.49	84-117
Antimony	Sb	100	99.9	2.40	93-107
Arsenic	As	100	101.6	3.66	91-113
Barium	Ba	100	99.7	2.64	92-108
Beryllium	Be	100	105.9	4.13	88-112
Cadmium	Cd	100	100.8	2.32	94-108
Chromium	Cr	100	102.3	3.91	91-114
Cobalt	Co	100	97.7	2.66	90-106
Copper	Cu	100	100.3	2.11	94-107
Lead	Pb	100	104.0	3.42	94-114
Manganese	Mn	100	98.3	2.71	90-106
Molybdenum	Mo	100	101.0	2.21	94-108
Nickel	Ni	100	100.1	2.10	94-106
Selenium	Se	100	103.5	5.67	86-121
Silver	Ag	100	101.1	3.29	91-111
Thallium	Tl	100	98.5	2.79	90-107
Thorium	Th	100	101.4	2.60	94-109
Uranium	U	100	102.6	2.82	94-111
Vanadium	V	100	100.3	3.26	90-110
Zinc	Zn	100	105.1	4.57	91-119

21.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Because all materials utilized in this procedure are potentially radioactive sources, all samples, waste, and standards will be appropriately labeled and handled according to MCL-7718 and MCL-7715.

The waste will be minimized by using small volumes and minimizing quantities utilized for sample preparation and standards preparation. Materials for disposal will be segregated and properly labeled. Where possible, the waste will be reduced by known treatment methodologies.

Rad waste will be measured and documented and where necessary turned over to an approved commercial handling and disposal service.

ICP-MS Metals
CALIBRATION SOLUTION STANDARD #1
PREPARATION LOG

****For BeO Filters Prep - Samples & LCS are NOT diluted ****

Make 250mL of Blank solution with 10mL conc H2SO4, 10mL conc HNO3 & 20mL conc HCl

Prepared By: _____ Expiration Date: 1/13/2019 Matrix: 16% Mixed Acid

Stock Solution		Standard ID	071618-Be-filters Stock Std	071618-01 ICPMS#1	071618-02 ICPMS#2	071618-03 ICPMS#3	071618-04 ICPMS#4	071618-05 ICPMS#5
Analyte	Conc. (ug/ml)	Final Vol (ml)	50	50	50	50	50	50
ICP0472 Exp. Date 05/2020		Transfer vol.(ml)	1.0	0.010	0.030	0.125	2.5	25
Be	50	ug/L	1.0	0.20	0.60	2.50	50	500

*50mL final vol - 2mL conc H2SO4, 2mL HNO3 and 4mL conc HCl
*100mL final vol - 4mL conc H2SO4, 4mL HNO3 and 8mL conc HCl
*250mL final vol - 10mL conc H2SO4, 10mL HNO3 and 20mL conc HCl

Room# _____
Humidity _____
Temperature _____

Balance/ Weight Set	Check Mass, g	Weight #1, g	Weight #2, g	Average Wt., g	Tolarance Acceptance
					± 2%
					± 2%

Pipette s/n	Volumn, mL	Weight #1, g	Weight #2, g	Average Wt., g	Tolarance Acceptance
					± 2%
					±2%
					±2%
					±2%
					±2%
					±2%

ICP-MS Metals

CALIBRATION SOLUTION #1; 2nd SOURCE STANDARDS PREPARATION LOG

Prepared By: _____ Date Prepared: 7/17/18 Exp. Date: 1/13/2019

Matrix:16% Mixed Acid

Stock Solution		Standard ID	Be-Filter-CCV-071618-SS				
Analyte	Conc. (ug/ml)	Final Vol (ml)	50				
ICP0443 Exp Date 8/2019		Transfer vol.(ml)	0.025				
Be	100	ug/L	50.0				

*50mL final vol - 2mL conc H2SO4, 2mL HNO3 and 4mL conc HCl
*100mL final vol - 4mL conc H2SO4, 4mL HNO3 and 8mL conc HCl
*250mL final vol - 10mL conc H2SO4, 10mL HNO3 and 20mL conc HCl

	Balance/ Weight Set	Check Mass, g	Weight #1, g	Weight #2, g	Average Wt., g	Tolerance Acceptance
Room#						
Humidity						± 2%
Temperature						± 2%

Pipette s/n	Volume, mL	Weight #1, g	Weight #2, g	Average Wt., g	Tolerance Acceptance
					± 2%
					±2%
					±2%
					±2%
					±2%
					±2%

OPERATOR AID 35

OPERATION OF THE XRD

1. SAMPLE PREPARATION (POWDER DIFFRACTION)

The characteristics of the peaks (height and breadth) recorded in an x-ray diffractogram are dependent on particle size and orientation. Proper sample preparation is required if good x-ray diffraction data is to be obtained. The sample should be homogenous, very fine-grained, and with randomly oriented particles. Proper sample preparation is essential for quantitative analysis using XRD. For identification of unknowns, poor sample preparation can result in misleading peak heights (intensities) which increases the difficulty of identifying unknown samples.

Homogenization: In many cases, the material analyzed in the x-ray diffractometer is a subsample of a larger sample submitted by a client. If the analyzed sub-sample is to be representative of the bulk sample, the entire bulk sample should be homogenous and well mixed. This can be accomplished in three ways:

1) "Cone and quarter" the sample.
2) Pass the sample through a riffle splitter
3) Mix the sample in a shaker mill.

Sizing (grinding/sieving): Samples analyzed by XRD should be very-fine grained and pass through a 37 micron sieve. For most samples this can be accomplished by crushing the sample in the SPEX mixer mill for three minutes using a steel vial and hardened steel grinding ball.

Sample Mounting: For most XRD samples, the powder is mounted in specialized holders supplied by the instrument manufacturer. Glass side pack holders are preferred, and are essential for quantitative analysis. Zero background holders are used when only a very small quantity of sample is available and should not be used for quantitative analysis. Zero background holders are used only for identification of unknown materials or other purposes where quantitative information is not needed.

1) *Glass Side-Pack:* Samples are mounted in a glass slide with a rectangular 3mm deep indention as follows;
    a. Place a small amount of the sample in the well in the glass so that it fills the indentation and forms a low mound over the top of the indentation.
    b. Using a small glass slide (a petrographic slide works well) pack the powder into the well from a vertical direction. Using two fingers gently tap the edge of the slide on a hard surface so that the powder packs from the side of the slide.

c. Using the edge of the small glass slide scrape excess material from the XRD mount so that the top of the packed powder is flush with the surface of the XRD mount.
d. A gap may be present in the well containing the powder. If so, add additional sample to fill the gap and repeat a-through-c as needed.

2) *Zero-background holders:* Zero background (ZB) holders are used when the amount of available material is insufficient to fill the well in a side-packed mount. ZB holders consist of a rectangular or circular plate of elemental silicon pressed into an aluminum plate. The silicon plate is oriented in such a manner that no x-rays are diffracted from the plate surface. Two types of holders are available: 1) a ZB holder with a flat silicon plate, and 2) an indented ZB holder with a small well in the center of the plate.
   a. *Flat ZB holder:* Using a cotton applicator spread a small amount petroleum jelly onto the surface of the silicon insert. With a second clean cotton applicator, remove excess petroleum jelly so that only a thin layer remains on the silicon plate. Using a spatula, place a small amount of the powdered sample near the edge of the silicon insert. While tilting the ZB holder, lightly tap the holder with the spatula so the powder spreads evenly across the surface of the silicon insert.
   b. *Indented ZB holder:* Using a micro spatula, place a small amount of the powdered sample in the well at the center of the silicon plate. Use enough material to form a mound extending above the surface of the plate. Using the edge of a glass slide, scrape excess material from the plate so that the top of the sample is flush with the surface of the plate.

Air sensitive materials: Air sensitive materials can be analyzed using a custom chamber that is located in the XRD housing. This chamber consists of an aluminum cylinder with a window cut into one side. The window is covered with an x-ray transparent film. This assembly is connected to a nitrogen cylinder with a small diameter hose. To use, mount the sample in the diffractometer as usual. Slip the chamber over the sample with the window in the up position. Attach the chamber with the set screw. Open the nitrogen cylinder and adjust the gas flow so the nitrogen slowly bleeds through the chamber. Start the XRD run. Close the nitrogen cylinder when the analysis is complete.

Radioactive materials: Radioactive materials should only be analyzed using sample holders reserved for these samples. All appropriate radiological protocols should be followed.

2. INSTRUMENT OPERATION

Idle-mode description:

1) Check to see that no x-rays are being emitted by the instrument. The "x-rays on" indicator light should not be on.
2) Check to see that no warning lights are on. Investigate and correct any faults as necessary. See the lead operator or technical manager (microscopy) if needed.

3) Check to see that the computer is on and booted. If the computer is off, turn on and reboot. See the lead operator or technical manager (microscopy) if needed.

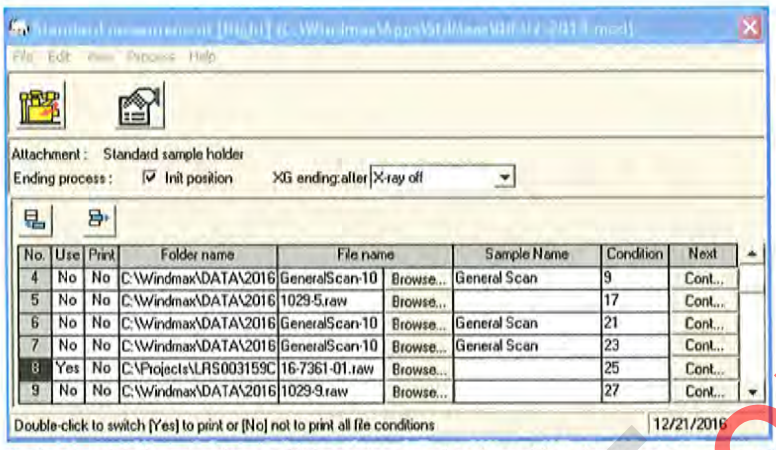
Mounting the sample in the instrument:

1) *Normal samples:* Sample holders will have a rectangular well or a silicon plate located close to one end of the holder. To mount the sample holder, insert the opposite end of the sample holder between the clips and aluminum half-cylinder in the instrument. The well/plate will be located in path of the x-ray beam. Insure that the sample holder is centered relative to the edges of the aluminum half-cylinder.
2) *Air sensitive and hygroscopic samples:* Check to see that the nitrogen cylinder contains gas. Turn on the gas supply and check to see that the LOW pressure gauge reads abut 5 psi. If not, adjust the gas pressure to suite. Leave the gas on to allow the chamber to flush (about 1 minute). Mount the sample holder to the instrument as described above. With the sample holder mounted, carefully slip the chamber over the holder onto the aluminum half cylinder. When properly positioned, the chamber set screw should be vertical. Tighten the set screw and start analysis.
3) *Tall or irregularly shaped samples:* These samples can be accommodated using a custom built holder which replaces the clip used for the usual flat sample holders. The custom holder consists of a small lab jack mounted on an L-bracket. To use, remove the two small screws which attach the clip used to hold the sample holders to the instrument. After attaching the sample to the top of the lab jack (using tape or mounting clay), use the same screws to attach the custom holder to the instrument. Use the lab jack to adjust the height of the sample surface until? it is flush with the bottom of the half cylinder.

Sample Log: **RECORD ALL SAMPLES IN THE SAMPLE LOG. THIS IS A STATE REQUIREMENT FOR OUR LICENSE.**

Using automated modes

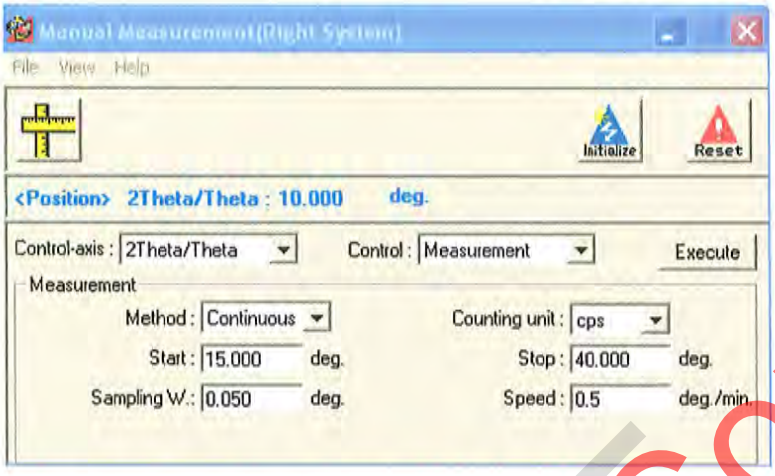
1) Select "Standard Measurements" program on the desktop. A spread sheet-like menu will open. Each row in the menu refers to a set of pre-programed instrument conditions appropriate for different types of samples. Instrument conditions are designated numerically in column 8; "conditions".



2) Select which set of conditions to use by toggling between yes and no in column 2 using the left mouse button. Only one condition should be selected so make sure all other conditions read "NO" in column 2.
3) Select "Browse" button (column 6) in the row for the selected set of conditions. This opens a window where an appropriate folder and file name can be selected or created. The XRD data from the sample run will be recorded automatically in a .RAW file using the selected file name in the selected project folder.
4) Insure that the "Init position: box is checked and that "X-ray off" is selected in the "XG ending after" option list. This turns the x-ray tube off and returns the instrument to its starting position after the analysis is complete.
5) Start the analysis by selecting the left-most, yellow icon in the tool bar. The instrument will initialize automatically, the x-ray generation will start automatically, and a window will open where the progress of the analysis can be monitored.

Using manual mode

1) Select "Manual Measurements" program on the desktop. A window will open where instrument conditions can be changed manually. Instrument control is not automated, so operations such as initialization and turning on the x-ray rube are also performed manually.



2) Select "Measurement" in the "Control" list.
3) Assign the start and stop positions in degrees 2-theta.
4) Assign the Sampling Width (also known as the step size) and speed.
5) Turn on the x-ray tube using the "X-rays on" switch in the front of the diffractometer.
6) Initialize the instrument by selecting the "initialize" icon in the Manual Measurement window.
7) Start the analysis by selecting "execute" or the yellow measurement icon in the tool bar.
8) The analysis will start and a window will open where the progress of the analysis can be observed.
9) When the analysis is complete, turn off the x-rays using the x-ray switch in the front of the diffractometer.
10) Files are not saves automatically in the manual measurement mode. Save the file by selecting "Save As" in the file menu of the monitoring window. This opens a window where an appropriate folder and file name can be selected or created.

Analysis of diffractograms: Two programs are used to analyze diffraction data.

1) *PDXL:* PDXL is the analysis software supplied by the instrument manufacturer. Refer to the PDXL user manual for instructions.
2) XPowder12: XPowder12 is a stand-alone software that offers many features not available in the PDXL program. XPowder12 is the preferred software for quantitative analysis. Refer to the XPowder12 user manual for instructions.

OPERATOR AID 36

Frisking Procedure for Leaving C-Corridor and other Radiological Areas

1. SCOPE

This procedure is to provide instructions for visitors and employees leaving the subject radiation area and can be used for leaving other radiation areas as well.

2. PROCEDURE

a) **HANDS FIRST** – Before touching anything! Hold fingers out from palm and move palm side of hand slowly over circular rad probe ¼ to ½ inch away. **REPEAT** for other hand, if no activity above background use oblong probe to check for gross alpha/beta. If no activity above background go to step 2. If activity is above background go to step 6.

b) Grab the circular probe by the handle and scan the **HOLD HERE** area on the counter. If no activity above background go to Step 3.

c) Balance yourself by holding the **HOLD HERE** area. Scan your **SHOE BOTTOMS** slowly, holding the probe about ¼ to ½ inch away.

d) Scan any other items such as **SHOE TOPS, PENS, PAPERS**.

e) **IF** the first meter in Steps 2 and 3 gives a response above background then go through steps 1-4 using second oblong probe to check for gross alpha/beta contamination. Then go back into rad area and decontaminate as described in Items 6-8. Any contaminated items leave in rad area for further evaluation. Rescan out using both meters.

f) **HAND CONTAMINATION** - proceed back into the one of the rad labs and clean your hands with soap and water and then re-scan out using both meters.

g) **SHOE CONTAMINATION** – remove your contaminated shoe(s) one at a time and put on the yellow booties found in drawers below and to the left of the meters. Ask lab personnel to assist in cleaning your shoe(s) in the rad area.

h) **CLOTHING CONTAMINATION** – seek assistance; all or part of your clothes may need to be removed and you will have to change into scrubs. Significant levels may require use of safety shower.

3. PRECAUTIONS

a) **VISITORS**: Avoid contact with items in the lab.

b) **STAFF**: Avoid touching items in Rad area that you don't need to use.

c) **DO NOT** rapid scan, which tends to happen when several people are waiting to scan out.

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE	
Low Level Mercury Determination Utilizing the Hydra llc CVAAS: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President — Date 10/4/12 Quality Assurance Officer — Date 10/4/12

1.0 PURPOSE

This document describes the procedures to determine low level Mercury concentration in samples based upon USEPA Method 7473 utilizing Teledyne/Leeman Labs Hydra llc Cold Vapor Atomic Absorption Spectrometer (CVAAS.)

2.0 SCOPE AND APPLICATION

The Hydra llc is a mercury analyzer based on the USEPA method 7473. The method eliminates the need of sample preparation. The process involves the combusting (decomposition) of a sample at high temperatures with oxygen. The gases are carried through a heated catalyst that removes halogens, nitrogen oxides, and sulfur oxides. The remaining combustion products including elemental mercury (Hg) are swept through a gold amalgamation tube. The amalgamation tube captures all of the mercury and is then heated to release as a gaseous bolus into the carrier gas toward the cold vapor atomic absorption spectrometer (CVAAS.) The transient signal is measured in series by a high sensitivity cell followed by a low sensitivity cell. The two peaks are integrated and reported against the best calibration of the two cells available. The purpose of the two cells is to provide a wider dynamic range of linearity than can be provided by a single optical cell length.

3.0 RESPONSIBILITES

3.1 MCLinc Technical Staff

The staff members utilizing this SOP are responsible for being proficient in the use of the method prior to analyzing samples. They are also responsible for the performance of all necessary maintenance and execution of appropriate quality control measures and documentation to ensure that the project's QA/QC goals are met.

3.2 Laboratory Manager

The Laboratory Manager (LM) will be responsible for ensuring that the appropriately trained operators execute all aspects of the sample preparation and analysis as defined in

this SOP. The LM is responsible for periodically reviewing and confirming that the client's QA/QC criteria are met. If criteria are not met then the analyst along with the Laboratory Manager will be responsible for the implementation of appropriate corrective actions and documentation.

3.3 QA/QC Manager

The MCLinc QA/QC Manager is responsible for ensuring that appropriate training programs are in place, periodically reviewing this SOP and its appropriateness for the actual proceedings being executed. The QA/QC Manager or designee will review all projects to evaluate the analytical data to determine the procedures precision and accuracy and to ensure that client QA/QC criteria are being met. If procedural precisions and accuracy or client criteria are not being met the QA/QC Manager will be responsible for overseeing the implementation of appropriate corrective actions and documentation.

4.0 SUMMARY OF METHOD

4.1 Balance Calibration Check

4.1.1 Prior to any analysis the balance must be checked and verified to be weighing correctly utilizing the MCLinc SOP# MCL-7754 Appendix V: Operational Aid for Balance Checks.

4.2 System Startup

4.2.1 Power up computer, monitor and printer if not already active
4.2.2 Open Oxygen supply to 15 psi (100KPa)
4.2.3 Power on the analyzer
4.2.4 Double-click ENVOY icon on the desktop
4.2.5 Check that the injector moves to outmost position (home)
4.2.6 Check furnace temperatures are set properly
Typical Temperature:

Dry default	300°C
Decomposition default	800°C
Catalyst default	600°C
Amalgam	600°C

4.2.7 Click the green Start-up icon and allow 10 minutes for furnace temperature equilibration.
4.2.8 The Hydra llc is now ready for analytical operation

4.3 System Shutdown

When the Hydra llc will be left idle overnight or longer it many make sense to power down the system completely and to stop gas flow. For complete shutdown follow the steps below.

4.3.1 Click the blue sleep icon to relax pressure on the injector seal
4.3.2 Close the ENVOY program
4.3.3 Power down the Hydra llc
4.3.4 Shut down the computer operating system (from the start button)
4.3.5 Shut down monitor, if necessary
4.3.6 Shut down the printer
4.3.7 Shut down **oxygen gas flow**

For shorter periods of time, simply click the yellow standby button on the tool bar. Standby will leave the catalyst furnace at the temperature defined on the instrument control screen but will discontinue heating to the decomposition and amalgamator furnaces. System fans will continue normally. Occasionally the injector will close and oxygen will be turned on to maintain the catalyst in a conditioned state.

4.4 Manual Operation (Single Sample)

4.4.1 Tare a boat on the balance
4.4.2 Place an amount of a standard, check (QC) standard, or unknown into the boat
4.4.3 Place the boat on the balance
4.4.4 Click either the Run Standard, Run QC, or Run Sample icon. If the injector is currently in the furnace, it will pull out for you to place the boat on the injector prongs.
4.4.5 Input the sample ID or select the appropriate Standard or QC
4.4.6 If the balance is interfaced to the computer then lick the "Get Weight" button. If not, then enter the weight in grams.
4.4.7 Place the boat on the prongs of the Injector
4.4.8 Click OK
4.4.9 The sample will move into the furnace and begin drying, the run will proceed to completion. To observe the analytical chart recording, click on the Results Tab

4.5 Autosampler Operation (Multiple Samples)

The Hydra llc has an optional autosampler. The autosampler can run 5 racks of 14 boats per rack, unattended, for a total of 70 boats. The autosampler can have samples added while it is running a sequence. This gives the capability of unlimited sample runs without stopping, provided the operator can continuously add new racks behind the existing un-run racks. Given the flexibility of adding boats and racks while the instrument is running some rules must be adhered to:

1) All racks must be placed between the left and right stem walls of the autosampler. The rack should be flat on the top of the autosampler and not resting on any features.
2) The first rack must be placed about 2" (50mm) from the front edge of the autosampler, BEHIND the stop pins.
3) Subsequent racks can be placed behind each other and should leave a gap of approximately 1/8"-1/4" (3-6mm). The rack pusher will automatically align the racks

against each other. Do not attempt placing racks on the autosampler when any mechanism is running.

4) There are two versions of the rack discharge rails. The shorter set allows collection of 2 racks with the last remaining on the autosampler for a total of 3 racks (42 boats) unattended. The longer set allows collection of 4 racks with the last remaining on the autosampler for a total of 5 racks (70 boats) unattended.

The basic sequence of operations for readying the instrument for an automated run is as follows:

4.5.1 Click on the Sequence Tab to display the autosampler sequence page
4.5.2 Click on Add Tray button
4.5.3 A spreadsheet of 14 empty locations will be added to the display
4.5.4 Each row represents a boat and its graphical representation will be updated in the lower "rack" graphic whenever the Update button is clicked
4.5.5 Enter standards, QCs, and samples with weights as needed. Add more trays as needed. When complete click the Update button to populate the navigation tree to the left with the proposed run sequence
4.5.6 Click the Run Sequence icon when ready to run

NOTES:

1) If a dialog appears on the Run Sequence icon is clicked, it is likely reporting some anomaly that may need resolving before running the sequence. Read the information carefully and decide whether to continue by clicking Yes or No.
2) A video of running a sequence can be view in HELP: Run a Sequence.

5.0 DEFINITIONS

5.1 Applicable definitions are located throughout this SOP.

6.0 SAFETY & STEPS TO AVOID LABORATORY CONTAMINATION

6.1 General laboratory protection (safety glasses, lab coat, and disposal latex/nitrile gloves) should be worn at all times when handling standards or samples.

6.2 Stock elemental mercury metal standards may pose potential health risks. Extreme care should be utilized when handling these powders or solutions.

6.3 Samples suspected to be highly contaminated with Hg should not be brought into the lab but processed or weighed into a boat outside the lab.

7.0 APPARATUS AND MATERIALS

7.1 Teledyne Leeman Labs Hydra llc Automated Direct Hg Analyzer.
7.2 Electronic Balance
7.3 Nickel Boat (p/n 302-00809-3) [Typical lifetime of a nickel boat is 30 analysis]

7.4 Micro-spatula [for loading the sample into the nickel boat]

8.0 CALIBRATION AND QUALITY CONTROL

8.1 Calibration

8.1.1 Reference the Standards Preparation Log Notebook for current concentrations for the intermediate standard and working standard solutions along with the transfer volumes for the calibration standards. (Note: per the Standards preparation methodology/calculation the instrumentation is calibrated as total µg Hg).

8.1.2 Construct a calibration curve by analyzing individually each Calibration Blank and calibration standards, increasing in Hg concentration, using the procedure given is Section 4.0 – Summary of Method. The Correlation Coefficient (R) should be >0.995, if not, re-prepare calibration standards and recalibrate.

8.2 Quality Control Samples – see Table 1: QC Sample Chart

8.2.1 Initial Calibration Verification (ICV) - Immediately after calibration, analyze an initial calibration verification standard made from an independent source (different manufacturer or lot from calibration standard) at or near mid-range. The acceptance criteria is ±10% of certified value. If the calibration curve cannot be verified within the specified limits, the cause must be determined and the instrument recalibrated before samples are analyzed.

8.2.2 Continuing Calibration Verification (CCV) - After every ten (10) samples, including QC samples such as LCS and MB and at end of analysis, a CCV must be analyzed. The CCV standard will be made from standards of the same source as the calibration standards and at or near the mid-range concentration. Acceptance criteria is ±10% of certified value.

8.2.3 Initial Calibration Blank, Continuing Calibration Blank (ICB, CCB) - An instrumental blank will be analyzed with each ICV and CCV. The ICB and CCB will not contain Hg above one-half the concentration of the lowest calibration standard.

8.2.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - If part of the project plan for samples, a selected sample will be spiked with a known concentration of Hg and analyzed in the sample analysis batch. Recovery acceptance will be defined in the project plan.

8.2.5 Method Blank (MB) - For each batch of twenty (20) or fewer samples a method blank will be prepared through the entire sample preparation procedure using all reagents for sample preparation. The MB will be acceptable if the Hg concentration is less than one-half of the lowest calibration standard concentration.

8.2.6 Laboratory Control Samples (LCS) – For each batch of twenty (20) or fewer samples at least one LCS spiked with a known concentration of Hg will be carried through the entire sample preparation procedure and analytical process using all reagents for sample preparation. The acceptance criteria is ±20% from certified value.

9.0 SAMPLE DIGESTION AND PREPARATION

The Hydra llc is a mercury analyzer based on the USEPA method 7473. The method eliminates the need of sample preparation.

10.0 WASTE MANAGEMENT

It is the laboratory's responsibility to comply with all applicable federal, state, and local regulations governing waste management. Efforts shall be made by the analyst to minimize waste generation.

11.0 PROFICIENCY TESTING

Since an AIHA-LAP, LLC-acceptable proficiency testing program for mercury is not available, MCL, Inc has set up an internal QC program to verify proficiency in the method under the management of the Quality Assurance Manager (QAM).

This program consists of:

1. Based on 20 or more QC data points for recovery, determine the mean, standard deviation and relative percent difference (rpd) for the LCS's to develop acceptance criteria. Until then +/- 20% is the default criteria.
2. The analyst, as designated by the QAM, will analyze at least twice annually, samples that were spiked independently of the analyst and represent the normal working range of the method.
3. The QAM will compare the results of Section 2 to the acceptance criteria of 3 times the standard deviation of the QC samples developed under Section 1 above.
4. If acceptance criteria are met the QAM will accept the data and notify the analyst. If one or more points fail, the QAM will investigate and determine corrective action which may require reanalysis of the whole study. Sample analyses will be suspended until acceptable results are obtained, i.e., at least 75% of samples pass with two of the last three rounds acceptable.

12.0 DOCUMENTATION

Record balance check weights, sample volumes or weights as appropriate, and calculations in a notebook or on the Instrument Data Sheet (Attachment 1). Also note any anomalies appropriately in the same document and report any quality issues to the QA Manager. Update Instrument Run Log each day of use.

11.0 REFERENCES

USEPA SW-846 Method 7473 Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry

Teledyne Leeman Labs, Hydra llc Automated Direct Hg Analyzer Operations Manual, 415-00063 Rev. H

ATTACHMENT I
Hg Decomp / Amalgamation / Cold Vapor Analysis
Hydra llc Instrument Data Sheet

MCL Project ID _____ Batch ID _____

Date _____ Operator _____

Position	MCL ID	Description	Sample Weight (g)	Hg ng/g	Hg Tng	Comments
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						

ATTACHMENT I (cont)
Hg Decomp / Amalgamation / Cold Vapor Analysis
Hydra llc Instrument Data Sheet

MCL Project ID _____ Batch ID _____

Position	MCL ID	Description	Sample Weight (g)	Hg ng/g	Hg Tng	Comments
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						
39						
40						

Balance ID		**Weight (g)**				
Calibration ID		**Actual (g)**				
Weight Set ID		**Actual (g)**				
		Avg (g)				

Table 1 – QC Sample Chart

QA/QC Type	Frequency	Acceptance Criteria	Corrective Action
Analytical /method blank (MB)	1 per daily sample groups of <20 or 1 per sample groups of 20	<1/2 of the value of the lowest reporting standard on the calibration curve	Evaluate data if there are clean samples then consider an anomaly; if consistent bias re-prepare the whole batch after consulting the work plan and the QA/QC director
Laboratory Control Sample (LCS)	1 per daily sample groups of <20 or 1 per sample groups of 20	%R=100% ± 20%	Re-prepare and run the LCS if it still fails. Review the work plan and discuss with the QA/QC Manager
Initial Calibration Verification (ICV) Second Source Standard	Immediately after calibration and before sample analysis	%R=100±10%	Reanalyze CCV; if still not acceptable, recalibrate; consult with QA Manager
Continuing Calibration Verification (CCV) Same source as calibration standards	Analyzed after every 10 real samples and after the last sample	%R= 100% ± 10%	Reanalyze CCV; if still not acceptable, then recalibrate; consult work plan and QA Manager
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Only if specified in the project work plan Spiking an aliquot of a selected sample with a known concentration of standard	Defined by the project plan	Defined by the project plan
Correlation Coefficient (R)	Upon calibration	>0.995	Re-prepare calibration standards and recalibrate

APPENDIX MM
OPERATOR AID FOR DETERMINATION OF WATER CONTENT BY MASS

1.0 Scope

This test method covers the determination of moisture content of soil, rock, and similar materials where reduction in mass by drying is due to loss of water.

2.0 Summary of Method

A test sample is dried in an oven at 110 ±2 degrees C to a constant mass. The loss of mass is considered to be water.

3.0 Equipment and Apparatus

a. Drying oven, capable of controlling temperature at 110 ± 2 degrees C.

b. Analytical balance, capable of weighing to the nearest 0.1g.

c. Specimen containers to hold samples of 25 and 200 g.

d. Dessicator

e. Hot sample handling equipment: gloves, tongs, or holder, etc.

4.0 Detailed Steps by Procedure

a. Weigh and record weight of clean specimen container (use attached bench log for recording information).

b. For soil samples, weigh 25 ± 0.1g of sample into a tared container. Weigh and record weight of sample plus container.

For rock or aggregate material, weigh 200 ± 0.5g of sample into a tared container. Weigh and record weight of sample plus container.

c. Place container with sample in oven at 110 ± 2 degrees C. Drying time may vary according to type of sample material, but drying overnight (12 to 16 hrs) is adequate.

d. After drying overnight, remove from oven and cool to room temperature in a dessicator.

e. Weigh and record the weight of the dry sample plus container.

f. Calculate weight % water of the sample as follows:

$$\% W = \frac{(Mcws - Mcs) \times 100}{Mcws - Mc}$$

Where

Mc = weight of empty sample container
Mcws = weight of container and wet sample, g
Mcs = weight of container and oven dry sample, g

5.0 Reporting of Results

These results are reported to the nearest 0.1 % as water content (wt % water) of the wet as received sample.

NOTE: FOR GEOTECHNICAL SAMPLES, RESULTS ARE REPORTED BASED ON DRY SAMPLE MASS.

6.0 Method Reference

ASTM-D2216-90 "Standard Test Method for Laboratory Determination of Water Content (% Moisture) of Soil and Rock by Mass.

BENCH LOG FOR DETERMINATION OF WATER CONTENT BY MASS

Method Reference: ASTM-D2216-90, "Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass"

Date:_____

Project#:_____

Customer Sample #:_____ MCLinc Sample #:_____

Sample Description:_____

BALANCE CALIBRATION

Balance	Date	Mass (g)	Weight (g)	Weight (g)

WEIGHTS REQUIRED FOR ANALYSIS

Mc – Mass of Empty Sample Container, g:_____

Mcws – Mass of Container and Wet Sample, g:_____

Mcs – Mass of Container and Oven Dry Sample, g:_____

Temperature:_____ Start Drying: _____ Stop Drying: _____

CALCULATION OF WATER CONTENT

$$\text{CALCUATION OF WATER CONTENT (W)} = \frac{\text{Mcws - Mcs}}{\text{Mcs - Mc}} \times 100$$

$$\text{WATER CONTENT (W)} = \frac{(\_\_\_\_\_) - (\_\_\_\_\_)}{(\_\_\_\_\_) - (\_\_\_\_\_)} \times 100$$

_____ _____
Analyst Date

QC Required: Duplicate Per Batch

BENCH SHEET FOR DETERMINATION OF WATER CONTENT BY MASS

Method Reference: ASTM-D2216-90, "Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass"

Batch #:_____ Project Number:_____

Sample Matrix:_____

	Sample Number	MCL ID#	Mc (g)	Mcws (g)	Mcs (g)	Water Content wt%
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						

BALANCE CALIBRATION

Balance	Date	Mass (g)	Weight (g)	Weight (g)

Analyst_____ Date_____

QC Required: One Duplicate per Batch

APPENDIX 38
Modified Sequential Extraction Procedure for Characterizing Source Materials from West Lake Landfill (Project Specific)

1.0 Purpose

The purpose of this operator aid is to describe a methodology for the operational assessment of the potential mobility for select potential contaminants of concern (PCOC), principally Ra-226, in submitted soil and debris samples originating from the West Lake Landfill, located in Missouri, USA. The procedure described herein is based on the selective sequential extraction methodology described by Liu and Hendry (2011), incorporating select client-recommended modifications as indicated in Appendix 1.

2.0 Sample Receipt and Preparation

Samples of radiological soil (approximately 250-g) will be shipped to MCLinc within a vacuum bag in frozen condition, in order to minimize potential constituent changes due to microbial activity. If analysis cannot be initiated on the day of receipt, core samples will be placed in a deep freeze (maintained at ~ -20 °C) until testing can be performed (see, e.g., Hlavay et al., 2004). Sample thawing will be performed within the as-received vacuum bag (e.g., by placement in a nitrogen-purged ambient-temperature storage vessel, such as a glove box), to help preserve in-situ redox conditions for anoxic sediments. It is recommended that testing be performed with aliquots of as-received sample that has experienced only minimal exposure to the ambient atmosphere prior to initiating sample conditioning and testing (Rapin et al., 1986).

Thawed samples will be dried under nitrogen to remove superficial water removed with minimal exposure to oxygen in ambient air. Analytical results are to be reported on "gas-dried" equivalent mass basis.

Sample size is dictated by the apparent sample homogeneity. If relatively large pebbles are first removed (e.g., with use of tweezers to remove particles > 2 mm diameter), a sample size of ~ 1 g (dry weight equivalent) of blended soil may be used. (Record actual mass taken). **If "container dried" soil is used, the moisture content must be estimated with use of a separate sample aliquot, to permit interpretation of results expressed on a dry-weight basis (MCL-7756, Appendix MM).**

Note: in order to yield sufficient volume of lixiviate for the requested battery of analyses, two separate 1-g aliquots of sample solids will be carried through the entire procedure, with lixiviate and water wash for each fraction combined to form a single sample for analysis.

Reagent-grade chemicals and de-ionized water (DIW) will be used for all extraction media preparations.

The lack of suitable certified reference materials for use with this procedure has precluded intra-laboratory comparability of results and hence good quality control. Quality control is thus the usual controls on analytical accuracy.

3. Fraction #1: Soluble and Exchangeable ions (Appendix 1)

Reagent #1: For 500-mL of lixiviate solution #1 (1 *M* $MgCl_2$, pH 7), add 47.6-g magnesium chloride ($MgCl_2$, FW 95.22).[5] De-aerate solution (e.g., bubble with nitrogen gas), and use without pH adjustment – **but record actual pH value**.

Note: In order to yield sufficient volume of lixiviate for the requested battery of analyses, two separate 1-g aliquots of sample solids (labeled (sample ID)-A and -B) will be processed by the procedure, with lixiviate and water wash for each fraction combined to form a single sample for analysis.

Note: Extraction Steps #1 and #2 are to be performed in a manner such as to minimize sample exposure to air. Sample aliquot will be loaded into the labeled centrifuge cone within a nitrogen-purged glove bag. De-gassed lixiviate will be added and then the centrifuge will be sealed tightly for subsequent phase contact and phase separation operations. (Providing the head space in the centrifuge cone has been purged of air, the sealed container may then be transferred out of the glove bag for external processing on a TCLP rotary extractor and for subsequent centrifugation. However, the sealed tube will only be opened within a nitrogen-purged glove bag, to minimize possible air-exposure of the contents).

For each 1.0 ± 0.1 g (dry-weight equivalent) aliquot of soil, use a labeled 50-mL polypropylene centrifuge cone. Next, add 10-mL of Reagent #1 to the sample aliquot. Seal the cone and contact the phases for 4-h at room temperature (optimum phase contact is provided by tumbling the sealed vial on a TCLP rotary extractor unit). This phase contact is to be performed with minimal potential exposure to ambient air. Slurry is subsequently centrifuged (approximately 4000 RPM for about 10-min). **The sealed centrifuge cone after centrifugation is returned to a nitrogen-purged glove bag for exchange of liquid phase.** Supernate phase is removed to a clean, labeled container with use of a transfer pipet.

The solid phase remaining in the centrifuge cone will be washed between extraction steps by phase contact with a 10-mL aliquot of de-gassed de-ionized water (DIW). Slurry in the sealed, air-free container is subsequently centrifuged (approximately 4000 RPM for about 10-min). Supernate phase is combined with the original lixiviate extract in the labeled container with use of a transfer pipet.

Extracts and wash solutions from processing two replicate 1-g sample aliquots are combined to form a single extract phase. **Record the actual pH value for the combined extract.** The combined aqueous phase will be subsequently digested (MCL-7746) and made up to an appropriate known volume (e.g., 50.0-mL, for combined extracts and wash solutions from aliquots A and B). (**Record final volume**).

[5] Bessinger (21 December 2015): Sequential Extraction Step 1: The first extraction step will be changed from 1 M $Mg(NO_3)_2$ (pH 7) to 1 M $MgCl_2$ (no pH adjustment). The objective of running the first two extractions in a glove box is to ensure the introduction of no potential oxidants. $MgCl_2$ is most-commonly used in sequential extractions and contains no potential oxidants.

4. Fraction #2: Acid Soluble and Carbonates (Appendix 1)

Note: Extraction Steps #1 and #2 are to be performed in a manner such as to minimize sample exposure to air.
Reagent #2: For 500-mL of lixiviate solution #2 (1 *M* CH_3CO_2Na, pH 5), add 68.04-g of sodium acetate hydrate ($CH_3CO_2Na•3H_2O$, FW 136.1). Adjust pH with dilute acetic acid or NaOH, as necessary to attain target pH value (5.0 ± 0.05). De-aerate solution (e.g., bubble with nitrogen gas), and record actual pH value.

To the individual solid residues from Extraction Fraction #1, add 25-mL of Reagent #2 to each centrifuge cone. Seal the cone and contact the phases for 6-h at room temperature (optimum phase contact is provided by tumbling the sealed vial on a TCLP rotary extractor unit). Slurry is subsequently centrifuged (approximately 4000 RPM for about 10-min). **The sealed centrifuge cone after centrifugation is returned to a nitrogen-purged glove bag for exchange of liquid phase**. Supernate phase is removed to a clean, labeled container with use of a transfer pipet.

The solid phase remaining in the centrifuge cone will be washed between extraction steps by phase contact with a 10-mL aliquot of de-ionized water (DIW). Slurry is subsequently centrifuged (approximately 4000 RPM for about 10-min). Supernate phase is combined with the original lixiviate extract in the labeled container with use of a transfer pipet.

Extracts and wash solutions from processing two replicate 1-g sample aliquots are combined to form a single extract phase. **Record the pH value for the combined extract**. The combined aqueous phase will be subsequently digested (MCL-7746) and made up to an appropriate known volume (e.g., 100-mL, for combined extracts and wash solutions from aliquots A and B). (**Record the final volume**).

Note: Extraction Steps #1 and #2 are to be performed in a manner such as to minimize sample exposure to air.

5. Fraction #3: Organics/Sulfides (Humic materials and iron-sulfides) (Appendix 1)

Note Extraction Step #3 and subsequent extractions may be performed without the need for air-exclusion.

Reagent #3: For 500-mL of lixiviate solution #3 (0.1 M $Na_4P_2O_7$, pH 10) add 13.3-g sodium pyrophosphate reagent (FW265.9). Adjust pH with dilute HNO_3 or NaOH, as necessary to attain target pH value (10.0 ± 0.05).

To the individual solid residues from Extraction Fraction #2, add 30-mL of Reagent #3 to each centrifuge cone. Seal the cone and contact the phases for (20 ± 1)-h at room temperature (optimum phase contact is provided by tumbling the sealed vial on a TCLP rotary extractor unit). Slurry is subsequently centrifuged (approximately 4000 RPM for about 10-min). Supernate phase is removed to a clean, labeled container with use of a transfer pipet.

The solid phase remaining in the centrifuge cone will be washed between extraction steps by phase contact with a 10-mL aliquot of de-ionized water (DIW). Slurry is subsequently centrifuged (approximately 4000 RPM for about 10-min). Supernate phase is combined with the original lixiviate extract in the labeled container with use of a transfer pipet.

Extracts and wash solutions from processing two replicate 1-g sample aliquots are combined to form a single extract phase. **Record the pH value for the combined extract**. The combined aqueous phase will be subsequently digested (MCL-7746) and made up to an appropriate known volume (e.g., 100-mL, for combined extracts and wash solutions from aliquots A and B). (**Record final volume**).

6. Fraction #4: Amorphous Oxides and Secondary Uranium Compounds (Appendix 1)

Reagent #4: For 500-mL of lixiviate solution #4 (0.2 M $(NH_4)_2C_2O_4$, pH 3), add 14.21-g of ammonium oxalate monohydrate $(NH_4)_2C_2O_4 \cdot H_2O$, FW 142.1). Adjust pH with dilute nitric acid or NaOH, as necessary to attain target pH value (3.0 ± 0.05).

To the individual solid residues from Extraction Fraction #3, add 10-mL of Reagent #4 to each centrifuge cone. Seal the cone and contact the phases for 4-h at room temperature (optimum phase contact is provided by tumbling the sealed vial on a TCLP rotary extractor unit). During the extraction step, avoid exposure of the slurry to direct light. Slurry is subsequently centrifuged (approximately 4000 RPM for about 10-min). Supernate phase is removed to a clean, labeled container with use of a transfer pipet.

Extracts and wash solutions from processing two replicate 1-g sample aliquots are combined to form a single extract phase. Record the pH value for the combined extract. The combined aqueous phase will be subsequently digested (MCL-7746) and made up to an appropriate known volume (e.g., 50-mL, for combined extracts and wash solutions from aliquots A and B). (Record final volume).

7. Fraction #5: Crystalline Oxides (Appendix 1)

Reagent #5: For 500-mL of lixiviate solution #5 (0.2 M $(NH_4)_2C_2O_4$ in 0.1 M ascorbic acid, pH 3), add 14.21-g of ammonium oxalate monohydrate $(NH_4)_2C_2O_4 \cdot H_2O$, FW 142.1) and 8.81-g L-ascorbic acid, FW 176.1). Adjust pH with dilute nitric acid or NaOH, as necessary to attain target pH value (3.0 ± 0.05).

To the individual solid residues from Extraction Fraction #4, add 25-mL of Reagent #5 to each centrifuge cone. Seal the cone and contact the phases for 0.5-h at 95 °C, with use of a thermostatic hot block. During heating, the tube lids will not be sealed, and polypropylene cover slips (e.g., environmentalexpress.com PN SC505) will be placed at the open end of the tube to minimize evaporative loss while allowing reaction gases to safely escape. Slurry is subsequently centrifuged (approximately 4000 RPM for about 10-min). Supernate phase is removed to a clean, labeled container with use of a transfer pipet. (**Record pH value of cooled extract solution**).

Extracts and wash solutions from processing two replicate 1-g sample aliquots are combined to form a single extract phase. Record the pH value for the combined extract. The combined aqueous phase will be subsequently digested (MCL-7746) and made up to an appropriate known volume (e.g., 100-mL, for combined extracts and wash solutions from aliquots A and B). (**Record final volume**).

8. Fraction #6: Alkaline-earth sulfates (Appendix 1)

Reagent #6: For a volume of 3.0-L of lixiviate solution #6 (0.11 M Na_2EDTA + 1.7 M NH_4OH) add 122.8-g disodium ethylenediamine tetraacetate (Na_2EDTA, FW 372.2) and sufficient ACS grade ammonium hydroxide solution (approximately 333-mL of stock 29 wt% reagent)[6] to yield a final concentration of 1.7 M as NH_4OH. **Caution: the ammonia reagent is toxic and noxious (use within a fume hood)**.

The solid residue from Fraction #5 is transferred by rinsing from its original 50-mL container into a 250-mL HDPE bottle with aliquots from a 200-mL aliquot of Reagent #6. The procedure (Appendix 1) requires that the solid sample residue be contacted with reagent #6 at 95 °C for a total of 4-h. Due to the physical size of the 250-mL bottle, this heating step will be performed in a thermostatic water bath placed within a fume hood. The lid to the bottle will be removed and a watch glass will be used to allow venting of ammonia vapor during the reaction.

At the end of the phase contact interval, the cooled slurry will be filtered by aspiration through a mixed cellulose ester (MCE) membrane (~ 0.7-µm pore) to recover the residual refractory solids. The solids will be rinsed with a 10-mL aliquot of DIW; filtered lixiviate and water rinse will be combined and made to a known volume (e.g., 250-mL). (Record final solution pH value).

9. Fraction #7: Residual (Appendix 1)

The MCE filter from fraction #6, with associated solids, is transferred to a suitable vessel and the filter medium and any residual organic matter is first digested with use of nitric acid and peroxide (MCL-7746). This pretreatment step is best performed with use of a hot block within a fume hood, to vent reaction gases (MCL-7746, § 5.2).

Complete digestion of refractory silicate phases requires the addition of hydrofluoric acid and may require microwave-assist (EPA Method 3052; MCL-7775, Appendix 16).

Note that failure to fume off excess HF reagent before preparing solutions for ICP analysis can cause damage to the instrument nebulizer.

10. Analysis and Data Report

Select one sample to run in duplicate (The project has designated Sample 15-5041 for the replicate). There is no accepted standard reference material identified for use in this procedure.

A filtered aliquot of the solution phase from each individual extraction step must be analyzed. Include a reagent blank (no sample) and matrix blank spike for each extraction step. Analysis by inductively coupled plasma optical emission spectroscopy (ICP-OES) may require digestion to destroy excess organic reagent (e.g., acetate) and/or substantial dilution of the sample prior to analysis (MCL-7752).

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[6] Stock ACS grade NH_4OH solution is nominally 28-30 wt% as ammonia (FW 17), or about 15.3 *M* (SG ~ 0.895). (Check lot analysis). For preparation of 3-L of 1.7 *M* dilute NH_4OH reagent, one would need approximately 333-mL (approximately 300-g) of stock ammonia reagent.

Project-specific parameters to be analyzed include U, Ra, Th, Fe, Mn, Ca, Ba, and sulfur (Appendix 1).

Report data results on the basis of the gas-dried weight of soil taken:

Metal extracted (µg/g) = (metal in extract, µg/L) * (volume of extract, L)/ (dry wt. equivalent for the original sample aliquot, g)

References

Bessinger, B. (S.S. Papadopoulos), in electronic memoranda to B. Stephenson and M. Standers (MCLinc), 9/15/2015 *et seq*.

EPA Method 3052 - Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices (1996).

Hlavay, J.; Prohaska, T.; Weisz, M.; Wenzel, W.W.; Stingeder, G.J. (2004). "Determination of Trace Elements Bound to Soils and Sediment Fractions," *Pure Appl. Chem.*, 76(2), 415-442.

Liu, D.J., and M.J. Hendry (2011), Controls on ^{226}Ra during raffinate neutralization at the Key Lake uranium mill, Saskatchewan, Canada, *Applied Geochemistry* **26** 2113–2120.

MCL-7746, Acid Digestion for Metals (EPA Method 3050B).

MCL-7752, Acid Digestion of Aqueous Samples (EPA Method 3010A).

MCL-7756, Appendix MM, Operator Aid for Determination of Water Content by Mass.

MCL-7775, Appendix 16, Operator Aid for the CEM Discover SP-D Microwave Digestion System.

Rapin, F.; Tessler, A.; Campbell, P.G.C.; Carignan, R. (1986), "Potential artifacts in the determination of metal partitioning in sediments by a sequential extraction procedure," *Environ. Sci. Technol.*; Vol/Issue: 20:8 836-840

Appendix 1

Sequential Extraction Protocols (Original sample mass is assumed to be approximately 1-g, air-dried)

Table A1. Sequential Extraction Procedure for Characterizing Source Materials

Step	Targeted Phases	Reagent
1	Soluble / Exchangeable: Exchangeable ions	10 mL of 1 M $MgCl_2$, pH 7, 4 hr, 25 °C; water wash (10 mL)
2	Acid Soluble: Carbonates	25 mL of 1 M CH_3CO_2Na, pH 5, 6 hr, 25 °C; water wash (10 mL)
3	Organics/Sulfides: Humic materials and Fe-sulfides	30 mL of 0.1 M $Na_4P_2O_7$, pH 10, 20 hr, 25 °C; water wash (10 mL)
4	Amorphous Oxides: Mn-oxides, ferrihydrite, and secondary U minerals	10 mL of 0.2 M $(NH_4)_2C_2O_4$, pH 3, 4 hr, 25 °C (dark); water wash (10 mL)
5	Crystalline Oxides: Goethite and Magnetite	25 mL of 0.2 M $(NH_4)_2C_2O_4$ in 0.1 M ascorbic acid, pH 3, 0.5 hr, 95 °C; water wash (10 mL)
6	Alkaline-earth sulfates: Barite	200 mL of 0.11 M Na_2EDTA + 1.7 M NH_4OH, 4 hr, 95 °C; water wash (10 mL)
7	Residual: Clays, primary U- and Th-oxides	HF-HNO_3 (Complete digestion)
Notes: Method based on Liu et al. (2011). All extractions use 1 gram of solid and all solutions analyzed for U, Ra, Th, pH, Fe, Mn, Ca, Ba, ~~total carbon~~ and sulfur; Procedure includes digestion/centrifugation, wash/centrifugation, and analysis steps. Finally, steps 1 and 2 will be conducted in a glove box or bag.		

SSP = Modified protocol, as recommended by B. Bessinger, S.S. Papadopoulos, in electronic memoranda to B. Stephenson, 9/15/2015 *et seq*. (Memorandum of 12/22/2015 dropped the analysis of total carbon in the extracts).
$MgCl_2$ = magnesium chloride (per B. Bessinger, S.S. Papadopoulos, in electronic memoranda to M. Sanders, 12/21/2015
CH_3CO_2Na = sodium acetate
$Na_4P_2O_7 \cdot 10H_2O$ = Sodium pyrophosphate decahydrate
$(NH_4)_2C_2O_4$ = ammonium oxalate
NH_4OH = ammonium hydroxide
$HClO_4$ = perchloric acid
HNO_3 = nitric acid
Na_2EDTA = disodium ethylenediamine tetraacetate (FW 372.2)

ATTACHMENT C-36

FERROUS IRON, SM-3500-FE-B

Pace Analytical Services, LLC
7726 Moller Road
Indianapolis, IN 46268
Phone: 317.228.3100
Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

THE DETERMINATION OF FERROUS IRON
REFERENCE METHODS: HACH METHOD 8146

SOP NUMBER:	S-IN-I-128-rev.05
EFFECTIVE DATE:	January 8, 2018
SUPERSEDES:	S-IN-I-128-rev.04

APPROVAL

General Manager — January 2, 2018 Date

Quality Manager — December 27, 2017 Date

Department Manager — December 27, 2017 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

Table of Contents

1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining Ferrous Iron in aqueous samples while meeting the requirements specified in Hach method 8146.

2. Summary of Method

2.1. The 1,10-phenanthroline indicator in the FerroVer Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The aqueous sample is filtered, reagents are added and the sample is read on a spectrophotometer at 510nm.

3. Scope and Application

3.1. Reporting limits, control limits, volumes used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.2. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ferrous iron analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1. This method is applicable for the measurement of Ferrous Iron in ground water, surface and saline waters and domestic and industrial wastewater.

5. Limits of Detection and Quantitation

5.1. The reporting limit is 0.2mg/L. Refer to the LIMS for method detection limit.

6. Interferences

6.1. Strong oxidizing agents, cyanide, nitrites, and polyphosphate can be interferences for this method. Other interferences include chromium and zinc in concentrations exceeding 10 times that of iron in a sample. Also cobalt and copper in excess of 5mg/L and nickel in excess of 2mg/L can cause interferences with this analysis.

6.2. Turbidity and color interferences may be eliminated by filtration through a 0.45um filter.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	500mL in plastic container	No preservation necessary	No temperature requirement. Avoid exposure to light.	**Sample must be analyzed as soon as possible after collection.

** Analysis can only be performed as soon as possible after receipt by the lab. A qualifier is applied to all results for Ferrous Iron analysis initiated more than 15 minutes after sample collection.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Definitions

8.1. Refer to the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

9. Equipment and Supplies

9.1. Instrumentation/Equipment

Equipment	Description / Comments
Spectrophotometer	Hach DR5000 or equivalent. For use at a wavelength of 510nm

9.2. General Supplies

Item	Description
Syringe filters	0.45um Environmental Express or equivalent
Dilu-vials	Used for mixing sample with reagents, Fisher or equivalent
Graduated cylinder	Class A, 25mL, Fisher or equivalent
Mechanical pipets	Various sizes. Eppendorf or equivalent

10. Reagents and Standards

10.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
FerroVer Iron Reagent Packets	Hach catalog # 854-99 or equivalent

10.2. Analytical Standards

10.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 10.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains all target analytes and must not be prepared from the same standards as the calibration standards.	Same solution can be used for the LCS and MS/MSD

10.2.2. Storage Conditions

Table 10.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Ferrous Iron Calibration Standard	Fisher; catalog # I77-500; solid ferrous ammonium sulfate, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Ferrous Iron Calibration Standard	Refer to Section 10.2.3.1	Must be prepared fresh daily	Not applicable
Working Ferrous Iron Calibration Standards	Refer to Section 10.2.3.2	Must be prepared fresh daily	Not applicable
Stock Ferrous Iron ICV Standard	Acros; catalog # 42372; solid ferrous ammonium sulfate, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Ferrous Iron ICV Standard	Refer to Section 10.2.3.3	Must be prepared fresh daily	Not applicable
Working Ferrous Iron ICV Standard	Refer to Section 10.2.3.4	Must be prepared fresh daily	Not applicable

10.2.3. Preparation Procedures

10.2.3.1. Intermediate Ferrous Iron Calibration Standard Preparation

Intermediate Standard 1: Dissolve 0.7022g of the Stock Ferrous Iron Calibration Standard in 500mL of reagent water to give a final concentration of 200mg/L.

10.2.3.2. Working Ferrous Iron Calibration Standard Preparation

Working calibration standards must be prepared fresh daily by diluting the Intermediate Ferrous Iron Calibration Standard (200mg/L) in reagent water. The standards below are examples only and may vary:

Standard ID	Amount of Intermediate Standard	Final Volume in Reagent Water	Final Concentration
CAL1	0.10mL	100mL	0.2mg/L
CAL2	0.0625mL	25mL	0.5mg/L
CAL3 (CCV)	0.125mL	25mL	1.0mg/L
CAL4	0.1875mL	25mL	1.5mg/L
CAL5	0.25mL	25mL	2.0mg/L

10.2.3.3. Intermediate Ferrous Iron ICV Standard Preparation

Dissolve 0.7022g of the Stock Ferrous Iron ICV Standard in 500mL of reagent water to give a final concentration of 200mg/L. This standard is also used as the LCS and MS spiking solution.

10.2.3.4. Working Ferrous Iron ICV Standard Preparation

Dilute 0.125mL of Intermediate Ferrous Iron ICV Standard (200mg/L) to 25mL with reagent water to give a standard concentration of 1.0mg/L.

11. Calibration

11.1. Initial Calibration: A minimum of 5 calibration standards is required. The lowest calibration standard must be at or below the reporting limit. A new initial calibration must be analyzed every 6 months at a minimum. Refer to the Quality Manual for more information regarding calibration curves.

11.2. Linear Calibration: After zeroing the spectrophotometer with reagent water, use the instrumentation software to prepare a standard curve by plotting absorbance versus Ferrous Iron concentration of each calibration standard. The analyst may employ a regression equation that does not pass through the origin. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.995.

11.3. Back calculate the concentration of each calibration point. Acceptable recovery range for back-calculated calibration standards is 90-110%. Acceptable recovery for the lowest calibration standard is 50-150%.

11.4. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

11.5. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. A single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

11.6. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11.7. Initial Calibration Blank (ICB): An ICB consists of reagent water. An ICB must be analyzed after each ICV. If the ICB result is above the reporting limit, another ICB may be analyzed. If the second ICB fails, then a new calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception**: If the ICB is >RL, associated samples determined to be <RL are reportable

11.8. Continuing Calibration Verification (CCV): When an ICAL is not analyzed, the calibration must be verified by analyzing a CCV at the beginning of the analytical sequence. In all cases, a CCV must also be analyzed after every 10 samples and at the end of the analytical sequence to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.

11.9. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

11.10. Continuing Calibration Blank (CCB): A CCB consists of reagent water. A CCB must be analyzed after each ICV or CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable.

12. Procedures

12.1. Filter each sample through a 0.45um filter to eliminate color and turbidity. Method Blank and LCS must also be prepared using filtered reagent water. Reagent Blank consists of unfiltered reagent water.

12.2. Measure 25mL of sample into a Dilu-vial.

12.3. Prepare a Method Blank consisting of 25mL of filtered reagent water.

12.4. Prepare an LCS: Dilute 0.125mL of Intermediate Ferrous Iron ICV Standard (200mg/L) to 25mL with filtered reagent water to give a standard concentration of 1.0mg/L.

12.5. Prepare a Matrix Spike: Add 0.125mL of Intermediate Ferrous Iron ICV Standard (200mg/L) to 25mL filtered sample to give a spike concentration of 1.0mg/L

12.6. Add the contents of a Hach FerroVer Iron Reagent Packet to each sample, standard, and blank, and mix.

12.7. Wait 3 minutes for color development. An orange color will develop in the presence of Ferrous Iron.

12.8. Adjust the wavelength control of the spectrophotometer to 510nm. Zero the spectrophotometer using reagent water. Measure and record the absorbance of the standards and samples. A typical run sequence may be as follows:

ICAL Standards
ICV
ICB
(If ICAL not run, CCV would replace the ICAL and the ICV in the sequence)
CCB
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB

12.9. To correct for background in samples, measure and record the absorbance of an aliquot of the sample without the addition of the reagent powder pillow.

12.10. Any sample with a Ferrous Iron concentration that exceeds the linear range of the calibration curve must be diluted and reanalyzed.

13. Quality Control

13.1. Batch Quality Control

Table 13.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water (filtered)	One per preparation batch of up to 20 samples.	Target analyte must be less than reporting limits	Reanalyze method blank. If target compound is still >RL in method blank and associated samples, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples.	90-110% Recovery	Reanalyze LCS. If LCS is still outside acceptance limits, re-prepare and reanalyze all associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per batch plus an additional MS if >10 samples in the batch.	90-110% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

14. Data Analysis and Calculations

14.1. From the corrected absorbance, determine the concentration of Ferrous Iron present using the calculation below.

$$X = \frac{(y - b)}{a}$$

Where: X = sample concentration
y = response or absorbance, corrected for background
a = slope
b = y-intercept

14.2. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (mg/L)} = \frac{(X)(V_f)}{(V_i)}$$

Where: X = Sample concentration in mg/L
V_f = Final sample volume in Liters
V_i = Initial sample volume in Liters

14.3. LCS equation

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

14.4. MS/MSD equation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

14.5. RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Sections 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Sections 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Sections 11 and 13.

18. Method Performance

18.1. Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

18.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

19. Method Modifications

19.1. Samples and batch QC are routinely filtered prior to analysis.

20. Instrument/Equipment Maintenance

20.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

21. Troubleshooting

21.1. Refer to instrument maintenance logs and/or manufacturer's instructions.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

22.3. **Equipment:** Portions of the preparation and analytical equipment may operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in Waste Handling, or other applicable SOP.

24. Pollution Prevention

24.1. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

24.2. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. Hach method 8146; Hach method handbook

25.2. Pace Analytical Quality Manual; latest revision.

25.3. TNI Standard; Quality Systems section; 2003 and 2009.

26. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-IN-I-128-rev.03	1. Cover: removed SM3500Fe-D reference. 2. Section 1.1: removed SM3500Fe-D reference 3. Section 2.1: summary of method was expanded per Hach method literature. 4. Section 3.2: added reference to LIMS for MDL. 5. Table 7.1: added that analysis should take place as soon as possible after sample collection. 6. Section 9.2.3.2: identified Standard 3 as the usual CCV 7. Section 11.1: clarified that batch QC must be prepared using filtered reagent water and the reagent blank is unfiltered reagent water. 8. Section 11.5: clarified that spectrophotometer is zeroed on the reagent blank. 9. Section 11.7: revised/fixed equation for concentration from curve. 10. Table 12.1: revised method blank corrective action. 11. Section 12: clarified that batch QC must be prepared using filtered reagent water and that MS is prepared using filtered sample. 12. Inserted new Method Modifications section. 13. References: removed SM3500Fe-D reference	22Oct2012
S-IN-I-128-rev.04	1. Converted to 27-section format. 2. Cover page: changed phone number, revised effective date and revised document control format. 3. Table 7.1: added to "avoid exposure to light." 4. Table 10.3: separated intermediate calibration standards into two rows. 5. Section 10.2.3: separated intermediate calibration standards into two sections and revised intermediate standard recipes to 500mL final volume. 6. Section 11: removed linear calibration equation and added ICB. 7. Section 12.8: added ICB to example sequence. 8. Table 13.1: revised MS frequency. 9. Section 14.1: updated equation for sample concentration to match prep log.	04Jan2016
S-IN-I-128-rev.05	1. Section 2.1: corrected spelling of 1,10-phenanthroline. 2. Table 7.1: removed "or within 24 hours of collection" from Hold Time and added note that H3 would be added to all results. 3. Section 9.2: added mechanical pipets. 4. Table 10.3: revised Stock ICV source and removed Int. Cal. Std. 2. 5. Section 10.2.3: removed Int. Cal. Std. 2 preparation and updated working calibration standard preparation. 6. Section 11: changed reagent blank to reagent water for zeroing instrument and for ICB and CCB with no addition of reagent pillow. Added back-calculation requirement for ICAL. 7. Section 12.8: changed reagent blank to reagent water for zeroing. 8. Table 13.1: updated corrective action for method blank and LCS. 9. Section 14.2: updated units to be in like terms with concentration from curve. 10. Section 25.3: added years 2003 and 2009 to TNI reference.	27Dec2017

ATTACHMENT D

FIELD AUDIT FORM, WEST LAKE LANDFILL, BRIDGETON, MISSOURI

Attachment D. Field Audit Form

Field Personnel

Auditor: _____ Project Manager: _____
Date: _____ Site Health & Safety Officer: _____
Location: _____ Field Staff: _____
Project: _____
Activity: _____

Health and Safety

Personal Protection Equipment: _____

Incidents: _____

Responses: _____

Noncompliance with Plan: _____

Monitoring Procedures

Sampling Procedure Noncompliance: _____

Field Parameter Measurement Noncompliance: _____

Decontamination Procedure Noncompliance: _____

Field Documentation Noncompliance: _____

Label Noncompliance: _____

Chain-of-Custody/Sample-Analysis-Request: _____

Noncompliance: _____

Preservation Noncompliance: _____

Completeness

Field Duplicate (1/10):

Field Blank (1/10):

Equipment Blank (1/10):

Trip Blank (1/cooler):

MS/MSD (1pair/20):

Noncompliances with Plan:

Auditor, Company, Date

General Notes

ATTACHMENT E

LABORATORY GUIDELINES

E-1. TIER I AND II LABORATORY PERFORMANCE GUIDELINES

E-2. TIER III AND IV LABORATORY PERFORMANCE GUIDELINES

ATTACHMENT E-1

TIER I AND II LABORATORY PERFORMANCE GUIDELINES

Attachment E-1
Tier I and II Laboratory Performance Guidelines

Communication: Trihydro requests that the laboratory notifies the project contact in the event of any non-conformances with the data. In general, all communication between the laboratory and Trihydro regarding problems or exceptions that may affect the data result for submitted samples will be directed to the specified project manager or the secondary point of contact. Notification by e-mail is acceptable and preferred, but a follow-up phone call is appreciated. In addition, the Trihydro project team should know the status and quality of the data at every stage of the process. Trihydro would appreciate prompt notification of any problems regarding the analysis of the samples or the generation of the final data report. Specifically, Trihydro requests that the point of contact be immediately notified of any of the following problems:

- Missing or incomplete chain-of-custody documents or custody seals
- Missing or damaged sample containers
- Switched, missing, or illegible sample container labels
- Sample temperatures outside of the method acceptable range of 2.0° to 6.0˚C
- Inadequate sample volume(s) to perform the required analyses and/or quality control tests
- Inadequate sample preservations
- Presence of headspace (bubbles greater than 6 millimeters in diameter) in VOA vials (holding time reduced to 7 days)
- Samples received outside of holding times or too close to the holding times for the laboratory to complete the analyses within the required timeframe
- Any other physical conditions relating to the samples which might adversely affect the final quality of the analytical results

Bottle Orders: Trihydro requests that the laboratory check bottle orders against the required analyte list prior to shipping to the project site. The field team requires a list that indicates which bottles are required for each analysis. For safety reasons, sample containers made of clear glass must be manufactured to the highest strength standard ("33 Expansion" or equivalent), and documentation must be provided for bottles that have an equivalent safety design. Additionally, adequate packing material to prevent sample breakage or scratching during shipping must be provided.

Analyses: Trihydro requests that the laboratory report only those constituents requested in the agreed upon laboratory quote. Trihydro requests that the laboratory report data using the lowest dilution feasible for each individual analyte and that the laboratory only reports the final sample result for each analyte. If the laboratory instrument is unable to meet the required reporting limit for any reason other than those previously discussed with the project manager or during the bid process, please notify the Trihydro project contact. During the analysis of the samples, the laboratory will immediately notify the project contact of problems, which may include highly buffered samples that will compromise data integrity, missed holding times (including method holding times), problems with the samples that will prevent the laboratory from meeting the requirements specified in the agreed upon laboratory quote, and significant failures of quality control checks that require re-extraction and/or reanalysis of any sample outside of holding time. The laboratory should report full spike lists equal to the requested constituents for both Laboratory Control Samples and Matrix Spikes. **Soils should be reported dry-weight for most projects. Please verify.**

Reporting: Trihydro requests that for large sampling events, the laboratory report approximately 20 samples per data set in order to minimize data validation costs. In addition, Trihydro requests that a complete and signed data set be provided by the laboratory in either a Portable Document Format (PDF) (preferred) and/or hard copy (mailed to Trihydro) within the turnaround time specified in the laboratory quote. Laboratory reports should include, at a minimum, the following elements:

a. A complete Case Narrative or laboratory notes that address major and minor exceptions relating to analysis of the data set
b. Sample data and quality control (QC) data including QC limits
c. Batch information for samples and QC data for each analysis
d. Dilution information for each sample and analyte
e. Notation of all collection, receipt, preparation/extraction, and analyses dates and times
f. All QC data as required by the method, including, but not limited to:
    i. Method Blank Results
    ii. Matrix Spike/Matrix Spike Duplicate Samples, percent recoveries, relative percent differences, QC limits, and parent samples
    iii. Laboratory Control Samples/Laboratory Control Sample Duplicates percent recoveries and RPD results
    iv. Laboratory duplicate RPD results
    v. Calibration data (if included in the standard report)
    vi. Surrogate Recoveries and QC limits for each sample
g. Signed chain-of-custody forms
h. Sample Receipt Checklist (or equivalent)
i. Definitions of laboratory qualifiers used

For electronic deliverables (EDDs), Trihydro requests that reports be issued in an electronic format acceptable to Trihydro's database (guidelines and example file are available). **Trihydro expects that the laboratory will ensure that EDDs and hard copy report values are identical and that similar QC measures are applied to the EDD and hard copy data reports.** In addition, Trihydro expects that the laboratory will be responsible for correcting any deficiencies or inaccuracies in either the electronic or hard copy versions of the data report in the time-frame specified by the contract.

ATTACHMENT E-2

TIER III AND IV LABORATORY PERFORMANCE GUIDELINES

Attachment E-2
Tier III and IV Laboratory Performance Guidelines

Communication: Trihydro requests that the laboratory notify the project contact in the event of any non-conformances with the data. In general, all communication between the laboratory and Trihydro regarding problems or exceptions that may affect the data result for submitted samples will be directed to the specified project manager or the secondary point of contact. Notification by e-mail is acceptable and preferred but a follow-up phone call is appreciated. In addition, the Trihydro project team should know the status and quality of the data at every stage of the process. Trihydro would appreciate prompt notification of any problems regarding the analysis of the samples or the generation of the final data report. Specifically, Trihydro requests that the point of contact be immediately notified of any of the following problems:

- Missing or incomplete chain-of-custody documents or custody seals
- Missing or damaged sample containers
- Switched, missing, or illegible sample container labels
- Sample temperatures outside of the method acceptable range of 2.0 to 6.0˚C
- Presence of headspace (bubbles greater than 6 millimeters in diameter) in VOA vials (holding time reduced to 7 days)
- Inadequate sample volume to perform the required analyses and/or quality control tests
- Inadequate sample preservation
- If samples are received outside of holding times or too close to the holding times for the laboratory to complete the analyses within the required timeframe
- Any other physical conditions relating to the samples which might adversely affect the final quality of the analytical results

Bottle Orders: Trihydro requests that the laboratory check bottle orders against the required analyte list prior to shipping to the project site. The field team requires a list that indicates which bottles are required for each analysis. For safety reasons, sample containers made of clear glass must be manufactured to the highest strength standard ("33 Expansion" or equivalent), and documentation must be provided for bottles that have an equivalent safety design. Additionally, adequate packing material to prevent sample breakage or scratching during shipping must be provided.

Analyses: Trihydro requests that the laboratory report only those constituents requested in the agreed upon laboratory quote. Trihydro requests that the laboratory report data using the lowest dilution feasible for each individual analyte. If the laboratory instrument is unable to meet the required reporting limit for any reason other than those previously discussed with the project manager or specified during the bid process, please notify the Trihydro project contact. During the analysis of the samples, the laboratory will immediately notify the project contact of problems which may include highly buffered samples that will compromise data integrity, missed holding times, internal standard recoveries below 50%, problems with the samples that will prevent the laboratory from meeting the requirements specified in the agreed upon laboratory quote, or significant failures of QC data that require re-extraction and/or reanalysis of any sample outside of holding time. The laboratory should report full spike lists equal to the requested constituents for both Laboratory Control Samples and Matrix Spikes. **Soils should be reported dry-weight for most projects. Please verify.**

Reporting: Trihydro requests that for large sampling events, the laboratory report approximately 20 samples per data set in order to minimize data validation costs. In addition, Trihydro requests that a complete and signed data set be provided by the laboratory in either a Portable Document Format (PDF) (preferred) and/or hard copy (mailed to Trihydro) within the turnaround time specified in the laboratory quote. Laboratory reports should include, at a minimum, the following elements:

a. A complete Case Narrative that addresses major and minor exceptions relating to analysis of the data set
b. Sample data and quality control (QC) data including QC limits
c. Batch information for samples and QC data for each analysis
d. Dilution information for each sample and analyte
e. Notation of all collection, receipt, preparation/extraction, and analyses dates and times
f. All QC data as required by the method including, but not limited to:
    i. Method Blank results
    ii. Matrix Spike/Matrix Spike Duplicate Samples, percent recoveries, relative percent differences, QC limits and parent samples
    iii. Laboratory Control Samples/Laboratory Control Sample Duplicates percent recoveries and RPD results
    iv. Serial dilution data
    v. Laboratory duplicate RPD results.
    vi. Internal standard data (where applicable)
    vii. Calibration, instrument tunes, and other instrument performance data
    viii. Raw data applicable to the samples
    ix. Surrogate recoveries and QC limits for each sample
g. Signed chain-of-custody forms
h. Sample Receipt Checklist (or equivalent)
i. Definitions of laboratory qualifiers used
j. Contract Laboratory Program (CLP) or CLP-like forms (compiled in one location within the report)

For electronic deliverables (EDDs), Trihydro requests that reports be issued in an electronic format acceptable to the Trihydro's database (guidelines and example file are available). **Trihydro expects that the laboratory will ensure that EDDs and hard copy report values are identical and that similar QC measures are applied to the EDD and hard copy data reports.** In addition, Trihydro expects that the laboratory will be responsible for correcting any deficiencies or inaccuracies in either the electronic or hard copy versions of the data report in the time-frame specified by the contract.

ATTACHMENT F

DATA VALIDATION TEMPLATE

F-1. TIER II DATA VALIDATION REPORT SUMMARY

F-2. TIER III DATA VALIDATION REPORT SUMMARY

ATTACHMENT F-1

TIER II DATA VALIDATION REPORT SUMMARY

Attachment F-1
Tier II Data Validation Report Summary

Client:	Laboratory:
Project Name:	Sample Matrix:
Project Number:	Sample Start Date:
Date Validated:	Sample End Date:
Parameters Included: ▪ ▪ ▪ ▪	
Laboratory Project ID:	
Data Validator:	
Reviewer:	

DATA EVALUATION CRITERIA SUMMARY

A Tier II Data Validation was performed by Trihydro Corporation's Chemical Data Evaluation Services Group on the analytical data report package generated by _____ evaluating samples from the _____ site located in _____.

Precision, accuracy, method compliance, and completeness of this data package were assessed during this data review. Precision was determined by evaluating the calculated relative percent difference (RPD) values from:

- Field duplicate pairs
- Laboratory duplicate pairs
- Matrix spike (MS) and matrix spike duplicate (MSD) pairs
- Laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) pairs

Laboratory accuracy was established by reviewing the demonstrated percent recoveries (%R) of the following items to verify that data are not biased.

- MS/MSD samples
- LCS/LCSD samples
- Organic system monitoring compounds (surrogates)

Field accuracy was established by collecting and analyzing the following samples to monitor for possible ambient or cross contamination during sampling and transportation.

- Trip blanks
- Field blanks
- Equipment blanks

Tier II Data Validation Report Summary

Method compliance was established by reviewing sample integrity, holding times, detection limits, surrogate recoveries, laboratory blanks, initial and continuing calibrations (where applicable), and the LCS/LCSD percent recoveries against method-specific requirements.

Completeness was evaluated by determining the overall ratio of the number of samples and analyses planned versus the number of samples with valid analyses. Determination of completeness included a review of the chain-of-custody (CoC), laboratory analytical methods, and other laboratory and field documents associated with this analytical data set.

SAMPLE NUMBERS TABLE

Client Sample ID	Laboratory Sample Number

Tier II Data Validation Report Summary

The laboratory data were reviewed to evaluate compliance with the methods and the quality of the reported data. Assessment of CoC completeness is included in Item 3 of the Data Validation Checklist. A check mark (✓) indicates that the referenced validation criteria were deemed acceptable, whereas a crossed circle (⊗) indicates validation criteria for which the data have been qualified by the data validator. An empty circle (○) indicates that the specified criterion does not apply to the reviewed data. Details are noted in the tables below.

Validation Criteria

- ✓ Data Completeness
- ✓ CoC Documentation (Item 3)
- ✓ Holding Times and Preservation (Items 6 and 7)
- ✓ Initial and Continuing Calibrations (Items 9 and 10)
- ✓ Laboratory Blanks (Items 11 and 12)
- ✓ MS/MSD (Items 13 and 14)
- ✓ LCS/LCSD (Items 15 and 16)
- ✓ System Monitoring Compounds (i.e., Surrogates) (Item 17)
- ✓ Field, Equipment, and Trip Blanks (Items 18 and 19)
- ✓ Field Duplicates (Items 20 and 21)
- ✓ Laboratory Duplicates (Item 22)
- ✓ Data Relationships (Item 23)

Guidance References

Chemical data validation was conducted in accordance with the United States Environmental Protection Agency (USEPA) Contract Laboratory Program (CLP) National Functional Guidelines for the analyses listed below, or by the appropriate method if not covered in the National Functional Guidelines.

- Data for organic analyses were evaluated according to validation criteria set forth in the USEPA CLP National Functional Guidelines for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017 with additional reference to the USEPA CLP National Functional Guidelines for Organic Data Review, document number EPA 540/R-99/008, October 1999.
- Data for inorganic analyses were evaluated according to validation criteria set forth in the USEPA CLP National Functional Guidelines for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017 with additional reference to the USEPA CLP National Functional Guidelines for Inorganic Data Review, document number EPA 540-R-04-004, October 2004.
- Review of field duplicates was conducted according to the USEPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures, EQADR-Supplement0, April 2013.
- The USEPA CLP National Functional Guidelines for High Resolution Superfund Methods Data Review, document number EPA-542-B-16-001, April 2016, was referenced for review of chlorinated dibenzodioxins (CDD) and chlorinated dibenzofurans (CDF) or chlorinated biphenyl congeners (CBC), as applicable.

Tier II Data Validation Report Summary

- Air and vapor data for samples collected in canisters and analyzed by EPA organics Method TO-15 were reviewed with reference to the USEPA Hazardous Waste Support Section, Analysis of Volatile Organic Compounds in Air Contained in Canisters by Method TO-15, SOP NO. HW-31, Revision 6, June 2014.
- Radiochemistry data were evaluated following criteria defined in USEPA Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), document number EPA 402-B-04-001A, July 2004.
- Trihydro Data Validation Variance Documentation, February 2019.
- Project-specific Quality Assurance Project Plans (QAPP) data validation requirements, as applicable.

OVERALL DATA PACKAGE ASSESSMENT

Based on a data validation review, the data are acceptable as delivered. Data qualified by the laboratory are discussed in Item 2 of the Validation Criteria Checklist.

The purpose of validating data and assigning qualifiers is to assist in proper data interpretation. Data that are not qualified meet the site data quality objectives. If values are assigned qualifiers other than an R (rejected, data not usable), the data may be used for site evaluation; however, consideration should be given to the reasons for qualification when interpreting sample concentrations. Data points that are assigned an R qualifier should not be used for site evaluation purposes.

If applicable, text was identified in **bold font** in the Validation Criteria Checklist to indicate that further action and/or qualification of the data were required. Data may have been qualified with J data flags by the laboratory if the result was greater than or equal to the method detection limit (MDL) but less than the reporting limit (RL). These laboratory-applied J flags were preserved, if present, and included in the Data Qualification Summary table at the end of this report. If applicable, data validation qualifiers were added for the items noted with crossed circles in the Validation Criteria section above. Please see the Data Qualification Summary table at the end of this report for a complete list of samples and analytes qualified.

If data would be qualified with more than one flag, one qualifier was assigned based on the severity; however, all reasons for qualification were retained. Data that would be qualified with both J+ and J- flags were evaluated based on validation criteria and assigned the appropriate flag. The hierarchy of qualifiers from the most to least severe is as follows:

- R > JB/U > NJ > J+/J- > J/UJ

Data qualifiers used during this validation are included in the following table.

Qualifier	Definition
J	Estimated concentration
J+	The result is an estimated concentration, but may be biased high
J-	The result is an estimated concentration, but may be biased low
UJ	Estimated reporting limit
U	Evaluated to be undetected at the reporting limit
JB	Estimated concentration due to blank contamination
R	Rejected, data not usable
NJ	Tentative identification and estimated concentration

Tier II Data Validation Report Summary

Data Completeness

The analyses were performed as requested on the CoC records. The associated samples were received by the laboratory and analyzed properly unless otherwise noted in the Criteria Checklist below. The complete data package consisted of _____ data points excluding blank samples. No data points were rejected. The data completeness measure for this data package is calculated to be 100% and is acceptable.

VALIDATION CRITERIA CHECKLIST	
1. Was the report free of non-conformances identified by the laboratory?	Yes
Comments:	
2. Were the data free of data qualification flags and/or notes used by the laboratory? If no, define.	Yes
Comments:	
3. Were sample CoC forms and custody procedures complete?	Yes
Comments:	
4. Were detection limits in accordance with the quality assurance project plan (QAPP), permit, or method, or indicated as acceptable?	Yes
Comments:	
5. Were the reported analytical methods and constituents in compliance with the QAPP, permit, or CoC?	Yes
Comments:	
6. Were samples received in good condition within method-specified requirements?	Yes
Comments:	
7. Were samples extracted/digested and analyzed within method-specified or technical holding times?	Yes
Comments:	
8. Were reported units appropriate for the sample matrix/matrices and analytical method(s)? Specify if wet or dry units were used for soil.	Yes
Comments:	
9. Did the laboratory provide any specific initial and/or continuing calibration results?	Yes
Comments:	
10. If initial and/or continuing calibration results were provided, were the results within acceptable limits?	Yes
Comments:	
11. Was the total number of laboratory blank samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
12. Were target analytes reported as not detected in the laboratory blanks?	Yes
Comments:	
13. Was the total number of MS samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	

VALIDATION CRITERIA CHECKLIST	
14. For MS/MSDs prepared from project samples, were percent recoveries and RPDs within data validation or laboratory quality control (QC) limits? Comments:	Yes
15. Was the total number of LCSs analyzed equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
16. Were LCS/LCSD percent recoveries and LCS/LCSD RPDs within data validation or laboratory QC limits? Comments:	Yes
17. Were surrogate recoveries within laboratory QC limits? Comments:	Yes
18. Were the number of trip blank, field blank, and/or equipment blank samples collected equal to at least 10% of the total number of samples or as required by the project guidelines, QAPP, SAP, or permit? Comments:	Yes
19. Were target analytes reported as not detected in the trip blank, field blank, and/or equipment blank samples? Comments:	Yes
20. Was the number of field duplicates collected equal to at least 10% of the total number of samples or as required by the project guidelines, QAPP, SAP, or permit? Comments:	Yes
21. Were field duplicate RPD values within data validation QC limits (soil 0-50%, water 0-30%, or air 0-25%)? Comments:	Yes
22. For laboratory duplicates prepared from project samples, were RPDs within laboratory QC limits? Comments:	Yes
23. Were the following data relationships realistic and acceptable? • Target analytes were reported by more than one method (e.g., 8260/8270, EPH/8270) and the results were in agreement? Comments: • Both total and dissolved metals analyses were performed and the total metals results were greater than or equal to the dissolved metals results? Comments:	Yes Yes

FIELD DUPLICATE SUMMARY

Client Sample ID: Field Duplicate Sample ID:				
Method	**Analyte**	**Laboratory Result (units)**	**Duplicate Result (units)**	**Relative Percent Difference (RPD)**

Field duplicate RPD control limits are not to exceed 30% for water, 50% for soil, or 25% for air or vapor as established by USEPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures, EQADR-Supplement0, April 2013.

DL – Indicates that the analyte was detected in one of the duplicate samples and was undetected in the other sample, and therefore an RPD could not be calculated. Data were not qualified since the detection was within two times the reporting limit. Non-detected results are indicated above with the applicable reporting limit as ND (RL).

+/-RL – Indicates that the detections in both of the samples were within two times the reporting limit. Qualification of data was not required.

DATA QUALIFICATION SUMMARY

Data qualifiers were not applied as a result of this validation.

Abbreviation	Reason

Analyte	Method	Field Sample ID	Lab Sample ID	Result	Limit	Units	Reviewer Qualifier	DV Flag Reasons

ATTACHMENT F-2

TIER III/IV DATA VALIDATION REPORT SUMMARY

Attachment F-2
Tier III/IV Data Validation Report Summary

Client:	Laboratory:
Project Name:	Sample Matrix:
Project Number:	Sample Start Date:
Date Validated:	Sample End Date:
Parameters Included: ▪ ▪ ▪ ▪	
Laboratory Project ID:	
Data Validator:	
Draft Reviewer:	Final Reviewer:

DATA EVALUATION CRITERIA SUMMARY

A Tier III/IV Data Validation was performed by Trihydro Corporation's Chemical Data Evaluation Services Group on the analytical data report package generated by _____ evaluating samples from the _____ site located in _____.

Precision, accuracy, method compliance, and completeness of this data package were assessed during this data review. Precision was determined by evaluating the calculated relative percent difference (RPD) values from:

- Field duplicate pairs
- Laboratory duplicate pairs
- Matrix spike (MS) and matrix spike duplicate (MSD) pairs
- Laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) pairs

Laboratory accuracy was established by reviewing the demonstrated percent recoveries (%R) of the following items to verify that data are not biased.

- MS/MSD samples
- LCS/LCSD samples
- Organic system monitoring compounds (surrogates)

Field accuracy was established by collecting and analyzing the following samples to monitor for possible ambient or cross contamination during sampling and transportation.

- Trip blanks
- Field blanks
- Equipment blanks

Tier III/IV Data Validation Report Summary

Method compliance was established by reviewing sample integrity, holding times, detection limits, surrogate recoveries, laboratory blanks, initial and continuing calibrations (where applicable), and the LCS/LCSD percent recoveries against method-specific requirements.

Completeness was evaluated by determining the overall ratio of the number of samples and analyses planned versus the number of samples with valid analyses. Determination of completeness included a review of the chain-of-custody (CoC), laboratory analytical methods, and other laboratory and field documents associated with this analytical data set.

SAMPLE NUMBERS TABLE

Client Sample ID	Laboratory Sample Number

Attachment F-2
Tier III/IV Data Validation Report Summary

The laboratory data were reviewed to evaluate compliance with the methods and the quality of the reported data. Assessment of CoC completeness is included in Item 3 of the Data Validation Checklist. A check mark (✓) indicates that the referenced validation criteria were deemed acceptable, whereas a crossed circle (⊗) indicates validation criteria for which the data have been qualified by the data validator. An empty circle (○) indicates that the specified criterion does not apply to the reviewed data. Details are noted in the tables below.

Validation Criteria

- ✓ Data Completeness
- ✓ CoC Documentation
- ✓ Holding Times and Preservation
- ✓ Field Duplicates
- ✓ Field, Equipment, and Trip Blanks
- ✓ Initial and Continuing Calibrations
- ✓ Instrument Tunes
- ✓ Internal Standards
- ✓ System Performance Checks
- ✓ Laboratory Blanks
- ✓ LCS/LCSD
- ✓ MS/MSD
- ✓ System Monitoring Compounds (i.e. Surrogates)
- ✓ Laboratory Duplicates
- ✓ Data Relationships

Guidance References

Chemical data validation was conducted in accordance with the United States Environmental Protection Agency (USEPA) Contract Laboratory Program (CLP) National Functional Guidelines for the analyses listed below, or by the appropriate method if not covered in the National Functional Guidelines.

- Data for organic analyses were evaluated according to validation criteria set forth in the USEPA CLP National Functional Guidelines for Organic Superfund Methods Data Review, document number EPA-540-R-2017-002, January 2017 with additional reference to the USEPA CLP National Functional Guidelines for Organic Data Review, document number EPA 540/R-99/008, October 1999.
- Data for inorganic analyses were evaluated according to validation criteria set forth in the USEPA CLP National Functional Guidelines for Inorganic Superfund Methods Data Review, document number EPA-540-R-2017-001, January 2017 with additional reference to the USEPA CLP National Functional Guidelines for Inorganic Data Review, document number EPA 540-R-04-004, October 2004.

- Review of field duplicates was conducted according to the USEPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures, EQADR-Supplement0, April 2013.
- The USEPA CLP National Functional Guidelines for High Resolution Superfund Methods Data Review, document number EPA-542-B-16-001, April 2016, was referenced for review of chlorinated dibenzodioxins (CDD) and chlorinated dibenzofurans (CDF) or chlorinated biphenyl congeners (CBC), as applicable.
- Air and vapor data for samples collected in canisters and analyzed by EPA organics Method TO-15 were reviewed with reference to the USEPA Hazardous Waste Support Section, Analysis of Volatile Organic Compounds in Air Contained in Canisters by Method TO-15, SOP NO. HW-31, Revision 6, June 2014.
- Radiochemistry data were evaluated following criteria defined in USEPA Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), document number EPA 402-B-04-001A, July 2004.
- Trihydro Data Validation Variance Documentation, February 2019.
- Project-specific Quality Assurance Project Plans (QAPP) data validation requirements, as applicable.

OVERALL DATA PACKAGE ASSESSMENT

Based on a data validation review, the data are acceptable as delivered. Data qualified by the laboratory are discussed in Item 2 of the Validation Criteria Checklist.

The purpose of validating data and assigning qualifiers is to assist in proper data interpretation. Data that are not qualified meet the site data quality objectives. If values are assigned qualifiers other than an R (rejected, data not usable), the data may be used for site evaluation; however, consideration should be given to the reasons for qualification when interpreting sample concentrations. Data points that are assigned an R qualifier should not be used for site evaluation purposes.

If applicable, text was identified in **bold font** in the Validation Criteria Checklist to indicate that further action and/or qualification of the data were required. Data may have been qualified with J data flags by the laboratory if the result was greater than or equal to the method detection limit (MDL) but less than the reporting limit (RL). These laboratory-applied J flags were preserved, if present, and included in the Data Qualification Summary table at the end of this report. If applicable, data validation qualifiers were added for the items noted with crossed circles in the Validation Criteria section above. Please see the Data Qualification Summary table at the end of this report for a complete list of samples and analytes qualified.

If data would be qualified with more than one flag, one qualifier was assigned based on the severity; however, all reasons for qualification were retained. Data that would be qualified with both J+ and J- flags were evaluated based on validation criteria and assigned the appropriate flag. The hierarchy of qualifiers from the most to least severe is as follows:

- R > JB/U > NJ > J+/J- > J/UJ

Attachment F-2
Tier III/IV Data Validation Report Summary

Data qualifiers used during this validation are included in the following table.

Qualifier	Definition
J	Estimated concentration
J+	The result is an estimated concentration, but may be biased high
J-	The result is an estimated concentration, but may be biased low
UJ	Estimated reporting limit
U	Evaluated to be undetected at the reporting limit
JB	Estimated concentration due to blank contamination
R	Rejected, data not usable
NJ	Tentative identification and estimated concentration

Data Completeness

The analyses were performed as requested on the CoC records. The samples were received by the laboratory and analyzed properly unless otherwise noted in the Criteria Checklist below. The complete data package consisted of _____ data points excluding blank samples. No data points were rejected. The data completeness measure for this data package is calculated to be 100% and is acceptable.

TABLE 1. GENERAL VALIDATION CRITERIA CHECKLIST	
1. Was the report free of non-conformances identified by the laboratory? Comments:	Yes
2. Were the data free of data qualification flags or other notes used by the laboratory? If no, define. Comments:	Yes
3. Were sample CoC forms and custody procedures complete? Comments:	Yes
4. Were detection limits in accordance with the quality assurance project plan (QAPP), permit, or method, or indicated as acceptable? Comments:	Yes
5. Were the reported analytical methods and constituents in compliance with the QAPP, permit, or CoC? Comments:	Yes
6. Were samples received in good condition within method-specified requirements? Comments:	Yes
7. Were samples extracted/digested and analyzed within method-specified or technical holding times? Comments:	Yes
8. Were reported units appropriate for the sample matrix/matrices and analytical method(s)? Specify if wet or dry units were used for soil. Comments:	Yes
9. Was the number of field duplicate samples collected equal to at least 10% of the total number of samples or as required by the project guidelines, QAPP, SAP, or permit? Comments:	Yes
10. Were field duplicate RPD values within data validation quality control (QC) limits (soil 0-50%, water 0-30%)? Comments:	Yes
11. Were the number of trip blank, field blank, and/or equipment blank samples collected equal to at least 10% of the total number of samples or as required by the project guidelines, QAPP, SAP, or permit? Comments:	Yes
12. Were target analytes reported as not detected in the trip blank, field blank, and/or equipment blank samples? Comments:	Yes

TABLE 1. GENERAL VALIDATION CRITERIA CHECKLIST	
13. Were the following data relationships realistic and acceptable?	Yes
• Target analytes were reported by more than one method (e.g., 8260/8270, EPH/8270) and the results were in agreement?	
Comments:	
• Both total and dissolved metals analyses were performed and the total metals results were greater than or equal to the dissolved metals results?	Yes
Comments:	

TABLE 2. VALIDATION CRITERIA CHECKLIST FOR VOC ANALYSES (8260B)	
1. Were instrument calibrations within data validation QC limits and analyzed at the appropriate frequency?	Yes
Comments:	
2. Were the instrument tunes within data validation control limits and analyzed at the appropriate frequency?	Yes
Comments:	
3. Were the internal standards within data validation control limits?	Yes
Comments:	
4. Were laboratory calculations acceptable?	Yes
Comments:	
5. Was the total number of laboratory blank samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
6. Were target analytes reported as not detected in the laboratory blank samples?	Yes
Comments:	
7. Was the total number of LCSs analyzed equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
8. Were LCS/LCSD percent recoveries and LCS/LCSD RPDs within laboratory QC limits?	Yes
Comments:	
9. Was the total number of MS samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
10. For MS/MSDs prepared from project samples, were percent recoveries and RPDs within data validation or laboratory QC limits?	Yes
Comments:	
11. Were surrogate recoveries within laboratory QC limits?	Yes
Comments:	
12. General Comments:	

TABLE 3. VALIDATION CRITERIA CHECKLIST FOR SVOC ANALYSES (8270C/8270C-SIM)	
1. Were instrument calibrations within data validation QC limits and analyzed at the appropriate frequency?	Yes
Comments:	
2. Were the instrument tunes within data validation QC limits and analyzed at the appropriate frequency?	Yes
Comments:	
3. Were the internal standards within data validation QC limits?	Yes
Comments:	
4. Were laboratory calculations acceptable?	Yes
Comments:	
5. Was the total number of laboratory blank samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
6. Were target analytes reported as not detected in the laboratory blank samples?	Yes
Comments:	
7. Was the total number of LCSs analyzed equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
8. Were LCS/LCSD percent recoveries and LCS/LCSD RPDs within laboratory QC limits?	Yes
Comments:	
9. Was the total number of MS samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
10. For MS/MSDs prepared from project samples, were percent recoveries and RPDs within data validation or laboratory QC limits?	Yes
Comments:	
11. Were surrogate recoveries within laboratory QC limits?	Yes
Comments:	
12. General Comments:	

TABLE 4. VALIDATION CRITERIA CHECKLIST FOR HPLC ANALYSES (8310)	
1. Were instrument calibrations within data validation QC limits and analyzed at the appropriate frequency?	Yes
Comments:	
2. Were laboratory calculations acceptable?	Yes
Comments:	
3. Was the total number of laboratory blank samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
4. Were target analytes reported as not detected in the laboratory blank samples?	Yes
Comments:	
5. Was the total number of LCSs analyzed equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
6. Were LCS/LCSD percent recoveries and LCS/LCSD RPDs within laboratory QC limits?	Yes
Comments:	
7. Was the total number of MS samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method?	Yes
Comments:	
8. For MS/MSDs prepared from project samples, were percent recoveries and RPDs within data validation or laboratory QC limits?	Yes
Comments:	
9. Were surrogate recoveries within laboratory QC limits?	Yes
Comments:	
10. General Comments:	

TABLE 5. VALIDATION CRITERIA CHECKLIST FOR SVOC ANALYSES (8015)	
1. Were instrument calibrations within data validation QC limits and analyzed at the appropriate frequency? Comments:	Yes
2. Were laboratory calculations acceptable? Comments:	Yes
3. Was the total number of laboratory blank samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
4. Were target analytes reported as not detected in the laboratory blank samples? Comments:	Yes
5. Was the total number of LCSs analyzed equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
6. Were LCS/LCSD percent recoveries and LCS/LCSD RPDs within laboratory QC limits? Comments:	Yes
7. Was the total number of MS samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
8. For MS/MSDs prepared from project samples, were percent recoveries and RPDs within data validation or laboratory QC limits? Comments:	Yes
9. Were surrogate recoveries within laboratory QC limits? Comments:	Yes
10. Were laboratory duplicates prepared? Comments:	Yes
11. For laboratory duplicates prepared from project samples, were RPDs within laboratory QC limits? Comments:	Yes
12. General Comments:	

TABLE 6. VALIDATION CRITERIA CHECKLIST FOR METAL ANALYSES (6010B/6020/7470A/7471)	
1. Were instrument calibrations within data validation QC limits and analyzed at the appropriate frequency? Comments:	Yes
2. Were the instrument tunes within method control limits and analyzed at the appropriate frequency? Comments:	Yes
3. Were system performance criteria met, including internal standards, interference check samples, and serial dilutions? Comments:	Yes
13. Were laboratory calculations acceptable? Comments:	Yes
4. Was the total number of laboratory blank samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
5. Were target analytes reported as not detected in the laboratory blank samples? Comments:	Yes
6. Was the total number of LCSs analyzed equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
7. Were LCS/LCSD percent recoveries and LCS/LCSD RPDs within data validation QC limits? Comments:	Yes
8. Was the total number of MS samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
9. For MS/MSDs prepared from project samples, were percent recoveries and RPDs within data validation or laboratory QC limits? Comments:	Yes
10. Were laboratory duplicates prepared? Comments:	Yes
11. For laboratory duplicates prepared from project samples, were RPDs within laboratory QC limits? Comments:	Yes
12. General Comments:	

TABLE 7. VALIDATION CRITERIA CHECKLIST FOR GENERAL CHEMISTRY PARAMETERS	
1. Were instrument calibrations within method or data validation QC limits and analyzed at the appropriate frequency? Comments:	Yes
2. Were laboratory calculations acceptable? Comments:	Yes
3. Was the total number of laboratory blank samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
4. Were target analytes reported as not detected in the laboratory blank samples? Comments:	Yes
5. Was the total number of LCSs analyzed equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
6. Were LCS/LCSD percent recoveries and LCS/LCSD RPDs within data validation or laboratory QC limits? Comments:	Yes
7. Was the total number of MS samples prepared equal to at least 5% of the total number of samples or analyzed as required by the method? Comments:	Yes
8. For MS/MSDs prepared from project samples, were percent recoveries and RPDs within data validation or laboratory QC limits? Comments:	Yes
9. Were laboratory duplicates prepared? Comments:	Yes
10. For laboratory duplicates prepared from project samples, were RPDs within laboratory QC limits? Comments:	Yes
11. General Comments:	

TABLE 8. FIELD DUPLICATE SUMMARY

Client Sample ID: Field Duplicate Sample ID:				
Method	**Analyte**	**Laboratory Result (units)**	**Duplicate Result (units)**	**Relative Percent Difference (RPD)**

Field duplicate RPD control limits are not to exceed 30% for water, 50% for soil, or 25% for air or vapor as established by USEPA New England Environmental Data Review Supplement for Regional Data Review Elements and Superfund Specific Guidance/Procedures, EQADR-Supplement0, April 2013.

DL – Indicates that the analyte was detected in one of the duplicate samples and was undetected in the other sample, and therefore an RPD could not be calculated. Data were not qualified since the detection was within two times the reporting limit. Non-detected results are indicated above with the applicable reporting limit as ND (RL).

+/-RL – Indicates that the detections in both of the samples were within two times the reporting limit. Qualification of data was not required.

ATTACHMENT A

BATCH MATCH TABLE

BATCH MATCH TABLE

Field Sample ID	Lab Sample ID	Dataset	SM20 2540 G	SW 6010 B

ATTACHMENT B

DATA QUALIFICATION SUMMARY

DATA QUALIFICATION SUMMARY

Data qualifiers were not applied as a result of this validation

Abbreviation	Reason

Analyte	Method	Field Sample ID	Lab Sample ID	Result	Limit	Units	Reviewer Qualifier	DV Flag Reasons