

LETTER OF TRANSMITTAL

Environmental Resources Management

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To: Mr. David Lederer
US EPA Region I
1 Congress Street, Suite 1100
Boston, MA 02114

Date: 5/17/2002
Project #: 215.19
Subject: Shpack Report

Enclosed please find:

- Copy of Report
- Copy of Letter
- Request For Information
- Proposal
- Graphics

Site: SHACK
Break: 3.2
Other: Professional Profiles

Other

Copies	Date of Information	Description
2	9-May-2002	Cabrera Services- Radiological Environmental Remediation on CD

SDMS DocID 000200473



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Comments:

Mr. Lederer,

Here are two CD's that go in the back of the Shpack Reports in the slipcover of three ring binders. That you received this morning by Federal Express package. I apologize for my mistake. If you have any questions, please call me at (617) 267-8377.

Copy To: _____

Signed: Alice Delano

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Appendix B, Standard Operating Procedures \ Cabrera Administrative Procedures

FILE NAMES:

AP-001, Record Retention, rev 0.pdf
AP-003, RCA Report, rev 0.pdf
AP-004, Radiological Compliance Audits, rev 0.pdf
AP-005, ALARA, rev 0.pdf
AP-006, Respiratory Protection Program, rev 0.pdf
AP-007, Bioassay, rev 0.pdf
AP-008, Dosimetry Program, rev 0.pdf
AP-009, Training, rev 0.pdf
AP-010, Personnel Protective Equipment, rev 0.pdf
AP-011, Emergency Response, rev 0.pdf
AP-012, Radiation Work Permits, rev 0.pdf
AP-013, Packaging Radioactive Material, rev 0.pdf
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FILE NAMES:

EOP-002, IDW Management, rev 0.pdf

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FILE NAMES:

OP-001, Radiological Surveys, rev 0.pdf

OP-002, Air Sampling and Analysis, rev 0.pdf

OP-004, Unconditional Release of Materials, rev 0.pdf

OP-005, Volumetric and Material Sampling, rev 0.pdf

OP-008, Chain of Custody, rev 0.pdf

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FILE NAMES:

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Laboratory Quality Assurance Plan

Revision 4

**Paragon Analytics, Inc.
225 Commerce Drive
Fort Collins, CO 80524
(970)490-1511**

Approved By:

Debra Henderer

Debra Henderer
Quality Assurance Manager

02-28-99

Date

Donald Gipple

Donald Gipple
Laboratory Manager

02/28/99

Date

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TERMS AND DEFINITIONS

Accuracy: The degree of agreement between a measured value and the true or expected value. The equation for accuracy is:

$$\text{Accuracy}(\% \text{Recovery}) = \frac{\text{Amount Found}(100)}{\text{Amount Added}}$$

Aliquot: A measured portion of a sample taken for analysis.

Analyte: The specific entity an analysis seeks to determine.

Background: Ambient signal response recorded by measuring instruments, that is independent of radioactivity contributed by the radionuclides being measured in the sample.

Batch: A grouping of no more than twenty samples of similar matrix which are prepared and analyzed together with the same method and the same lots of reagents within the same time frame. A sample may be analyzed in a different analytical batch than the one with which it was prepared.

Bias: The deviation of an expected value from a corresponding known or expected value.

Blank: A blank is an artificial sample designed to detect and/or monitor the contribution of analyte and non-analyte contamination, instrumental background and sample processing to the measurement system.

Blind Sample: A sample submitted for analysis whose composition is known to the submitter but unknown to the analyst.

Calibration: The process of establishing the relationship between instrument response and known, traceable quantities of analytes of interest.

Chemical Carrier (Yield): Stable (non-radioactive) nuclides or chemical analogs of the element added to each sample and QC sample. Comparison of the quantity of carrier added to that recovered following processing provides the basis for calculating chemical yield.

Chemical Yield (Recovery): The fraction of target analyte carried through a radiochemical separation or purification process. This value is used to correct radiochemical results for acceptable losses occurring during the preparation process.

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Comparability:	Comparability is a qualitative parameter expressing 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:	Measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. The equation for completeness is: $\% \text{ Completeness} = \frac{\# \text{ of valid data points obtained}}{\# \text{ of data points expected}} \times 100$
Continuing Calibration:	The process of analyzing standards periodically to verify the maintenance of calibration of the analytical system
Control Chart:	A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control
Control Limit:	A range within which specified measurement results must fall to signify compliance. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that nonconforming data be investigated and flagged.
Counting Uncertainty (Poissonian):	A statistical estimate of uncertainty in a radiochemical measurement due to the random nature of decay. Every radiochemical result is reported with an associated counting uncertainty, usually at the 95% confidence interval.
Daily Reliability Check:	A periodic check of the Continuing Calibration of an instrument used for radiochemical measurements.
Data Quality Objectives:	The qualitative of quantitative statements that specify the quality of data required to support decision for any process requiring chemical or physical analysis.
Detection Limit:	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.
Dry Weight:	The weight of a sample based on percent solids. The weight after drying in an oven at 105 °C.

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Duplicate Analysis:	A second measurement made on the same sample extract or digestion to assist in the evaluation of precision of analysis.
Duplicate (Replicate) Error Ratio (DER/RER):	A measure of precision used to assess agreement between radiochemical duplicates (replicates) which compares the discrepancy between two measurements to the associated uncertainties.
Duplicate Sample:	A second aliquot of the same sample that is treated the same as the original sample in order to determine the precision of the method
Equipment Blank:	Special type of field blank used primarily as a check on equipment decontamination procedures. Laboratory deionized water is passed over sampling equipment after decontamination.
Field Blank:	A quality control sample used to assess the contamination effects on accuracy due to the combined activities of sampling and analysis. Typically, it is composed of a reagent and analyte free matrix (deionized water) provided by the laboratory.
Field Sample:	A portion of material received by the laboratory to be analyzed, that is contained in single or multiple containers and identified by a unique field ID number.
Holding Time:	The elapsed time expressed in days from the date of sample collection by the field personnel until the date of its processing/analysis. For the Contract Laboratory Program, holding times start at the Verified Time of Sample Receipt (VTSR) by the laboratory. Holding time requirements are dictated by the method or QAPP.
Homogeneity:	The degree to which a property or substance is evenly distributed throughout a material.
Instrument Detection Limit:	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The instrument detection limit is generally lower than the method detection limit.
Initial Calibration:	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response, linearity and dynamic range of the

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instrument to the analytes of interest. Initial calibration is performed whenever the results of a continuing calibration do not conform to the requirements of the method in use or at a frequency specified in the method.

Internal Standards:	Analytes added to every standard, blank, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis. The function of an internal standard is to adjust the response factor used in quantitating target analytes. Internal standards are used as the basis for quantitation of the target compounds, and are generally applicable to organic analyses.
Laboratory Control Sample (LCS):	A control sample of known composition spiked with a known concentration of analytes of interest. Aqueous and solid laboratory control samples are analyzed using the same preparation, reagents, and analytical methods employed for field samples received.
LIMS	Laboratory Information Management System (LIMS) that is used to schedule and track work orders and report hardcopy and electronic data.
Lot:	A quantity of bulk material of similar composition processed or manufactured at the same time.
Matrix:	The predominant material of which the sample to be analyzed is composed.
Matrix Spike:	Aliquot of sample fortified (spiked) with known quantities of specified compounds and subjected to the entire procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.
Matrix Spike Duplicate:	A second aliquot of the sample that is treated the same as the original matrix spike sample. The relative percent difference between the matrix spike and matrix spike duplicate is calculated and used to assess analytical precision.
Method Blank:	An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory contamination and demonstrate that this level does not exceed acceptance limits. Acceptable levels of contamination are defined by project specific data quality objectives.
Method Detection	The Method Detection Limit (MDL) is defined as the minimum

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Limit:	concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. It may be determined using replicate spike samples prepared by the lab and taken through all steps of the method. The detection limit is calculated using the appropriate student's t-parameter times the standard deviation of a series of spiked samples. (Ref. 40 CFR Part 136, Appx. B)
Minimum Detectable Activity (Lower Limit of Detection):	The amount of radionuclide, which if present in a sample, would be detected with a 5% probability of non-detection while accepting a probability of 5% of erroneously detecting that radionuclide in a blank sample. Often used interchangeably with Minimum Detectable Concentration.
Minimum Detectable Concentration:	The minimum detectable activity expressed in concentration units.
Performance Audit or Evaluation:	A process to evaluate the proficiency of an analyst or laboratory by evaluation of the results obtained on known test materials.
Precision:	The measurement of agreement of a set of replicate results among themselves without any prior information as to the true result. Precision is assessed by means of duplicate/replicate sample analysis
Protocol:	A stated plan that clearly defines the objectives, methods and procedures for accomplishing a task.
QAPjP:	A Quality Assurance Project Plan or QAPjP is a project specific document that describes the policies, organization, objectives, functional activities, and specific QA and QC activities designed to achieve the data quality goals of a specific project.
Quality Assurance:	A system of policies and procedures whose purpose is to ensure, confirm and document that the product or service rendered fulfills the requirements of Paragon and its client. Quality Assurance includes quality planning, quality control, quality assessment (auditing), quality reporting and corrective action.
Quality Control:	A system of checks and corrective measures, integrated with the activities that directly generate the product or service, that serves to monitor and adjust the process to maintain conformance to predetermined requirements.
Radionuclide Tracer:	A traceable internal standard, usually a unique isotope of the element being determined, added to each sample in known amount which enables

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quantitation of analytes of interest independent of external means of calibration.

Replicate:	Multiple aliquots of a sample.
Relative Percent Difference:	A measure of precision between two duplicate (replicate) results expressed as the percent difference between the results relative to the average of the results.
Replicate Samples:	A second, separate sample collected at the same time, from the same place, for the same analysis, as the original sample in order to determine overall precision.
Reporting Limit:	The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the statistically determined MDL, but may be higher based on any of the above considerations. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.
Rounding Rules:	If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded to 11.44. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded to 11.45. If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded to 11.44, while 11.425 is rounded to 11.42. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.
Sensitivity:	Capability of methodology or instrumentation to discriminate between samples having differing concentrations or containing differing amounts of an analyte.
Split Sample:	A portion or subsample of a total sample obtained in such a manner that is not believed to differ significantly from other portions of the same sample.
Standard:	A substance or material the properties of which are believed to be known with sufficient accuracy to permit its use to evaluate the same property in a sample.

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Standard Blank:	An 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 Operating Procedure:	A procedure adopted for repetitive use when performing specific measurement or sampling operation. It may be an industry accepted standard method or one developed by the user.
Standard Reference Material:	Material characterized by the National Institute of Standards, or equivalent recognized body, for the activity of radionuclides and issued with a certificate that gives the results of the characterization.
Standard (spike) Addition	In radiochemistry, the addition of a known quantity of a radiotracer to a sample and to a split or splits of a sample. Both the sample and split(s) are then processed through the method and the difference in response between the samples used to correct for overall bias resulting measurement bias and from losses during preparation. This method of internal calibration is used in radiochemical determinations where isotopic differentiation between target analyte and tracer is not possible.
Surrogates:	When employed, these are compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard prior to any processing or preparation. These compounds are used to evaluate analytical efficiency by measuring recovery. Surrogate compounds are not expected to be detected in environmental media, but are similar to the analytes of interest. Surrogates are generally utilized for organic analyses.
Systems Audit:	An on-site inspection or assessment of a laboratories' quality control system.
Traceability:	The ability to trace the source and accuracy of a material (i.e. standard) to a recognized primary reference source such as the National Institute of Standards and Technology (NIST) or USEPA. This concept also includes the ability to independently reconstruct and review all aspects of the measurement system through available laboratory notebooks and documentation and reach the same results.
Total Propagated Uncertainty (TPU)	An estimate of the total error associated with a single radiochemical measurement for a single sample.
Trip Blank:	This blank is used to detect sample contamination from the container and

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preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory pure water; any preservative used in the sample is added; and then the blank is stored, shipped, and analyzed with its group of samples

Validation: The process by which a sample, measurement, method, or piece of data is deemed useful for a specified purpose.

Warning Limits The limits (typically 2 standard deviations either side of the mean) shown on a control chart within which most results are expected to lie (within a 95% probability) while the system remains in a state of statistical control.

LIST OF ACRONYMS

AA	atomic absorption
AFCEE	Air Force Center for Environmental Excellence
AFIID	Air Force installation identification
A2LA	American Association for Laboratory Accreditation
ARAR	applicable or relevant and appropriate requirement
ASCII	American Standard Code Information Interchange
ASTM	American Society for Testing and Materials
BFB	bromofluorobenzene
Bq	Becquerels
Br-	bromide
BTEX	benzene, toluene, ethylbenzene, xylene
°C	degrees Celsius
CCC	calibration check compound
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
Ci	Curies
CF	calibration factor
CFR	Code of Federal Regulation
Cl	chloride
CL	control limit
CLP	Contract Laboratory Program
COC	chain of custody
2,4-D	2,4 dichlorophenoxy acetic acid
2,4-DB	2,4 dichlorophenoxy butyric acid
DCA	dichloroethane
DCB	dichlorobenzene
DCBP	decachlorobiphenyl
DCE	dichloroethene
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethene
DDT	dichlorodiphenyltrichloroethane
DEQPPM	Defense Environmental Quality Program Policy Memorandum
DFTPP	decafluorotriphenylphosphine
DNB	dinitrobenzene
DNT	dinitrotoluene
DOD	Department of Defense
DQO	data quality objective
DRO	diesel range organics

LIST OF ACRONYMS

EDB	ethylene dibromide
EICP	extracted ion current profile
EPA	Environmental Protection Agency
F	fluoride
FID	flame ionization detector
FLAA	flame atomic absorption
FS	feasibility study
FSP	field sampling plan
g	gram
G	glass
GC	gas chromatography
GC/MS	gas chromatography/mass spectroscopy
GFAA	graphite furnace atomic absorption
GRO	gasoline range organics
Handbook	Handbook for the Installation Restoration Program (IRP) Remedial Investigation and Feasibility Studies (RI/FS), September 1993
HCl	hydrochloric acid
HECD	(Hall) electrolytic conductivity detector
HpCDD	heptachlorodibenzo-p-dioxin
HpCDF	heptaclorordibenzofuran
HxCDD	hexachlorodibenzo-p-dioxin
HxCDF	hexachlorodibenzofuran
HMX	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HNO ₃	nitric acid
HPLC	high-performance liquid chromatography
H ₂ SO ₄	sulfuric acid
IAW	in accordance with
ICP	inductively coupled plasma
ICPES	inductively coupled plasma emission spectroscopy
ICP-MS	inductively coupled plasma - mass spectroscopy
ICS	interference check standard
ID	identification
IRP	Installation Restoration Program
IRPIMS	Installation Restoration Program Information Management System
IS	internal standard
LCL	lower control limit
LCS	laboratory control sample

LIST OF ACRONYMS

MCPA	(4-chloro-2-methylphenoxy) acetic acid
MCPP	2-(4-chloro-2-methylphenoxy) propionic acid
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliter
mm	millimeter
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable
Na ₂ S ₂ O ₃	sodium thiosulfate
NCP	National Contingency Plan
ng/L	nanograms per liter
ng/mL	nanograms per milliliter
NIST	National Institute of Standards and Technology
nm	nanometer
NO ₂	nitrite
NO ₃	nitrate
NTU	nephelometric turbidity unit
OCDD	octachlorodibenzo-p-dioxin
ORP	oxidation-reduction potential
OVA	organic vapor analyzer
P	polyethylene
PAH	polynuclear aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo-p-dioxin
PCDF	polychlorinated dibenzofuran
PE	performance evaluation
PeCDD	pentachlorodibenzo-p-dioxin
PeCDF	pentachlorodibenzofuran
PID	photoionization detector
PO ₄	phosphate
ppb	parts per billion
ppm	parts per million
ppmv	parts per million volume
PQL	practical quantitation limit
QA	quality assurance
QAPjP	quality assurance project plan
QC	quality control

LIST OF ACRONYMS

R	recovery
RCA	recommendations for corrective action
RCRA	Resource Conservation and Recovery Act
RDX	hexahydro-1,3,5-trinitro-1,3,5-triazine
RF	response factor
RI	remedial investigation
RI/FS	remedial investigation/feasibility study
RPD	relative percent difference
RSD	relative standard deviation
S	soil
SAP	sampling and analysis plan
SARA	Superfund Amendments and Reauthorization Act
SO ₄	sulfate
SOP	standard operating procedure
SOW	statement of work
SPCC	system performance check compound
SVOC	semivolatile organic compound
2,4,5-T	2,4,5-trichlorophenoxy acetic acid
T	California brass
TCA	trichloroethane
TCDD	tetrachlorodibenzo-p-dioxin
TCDF	tetrachlorodibenzofuran
TCE	trichloroethene
TCLP	toxicity characteristic leaching procedure
TCMX	tetrachlorometaxylene
TIC	tentatively identified compound
TNB	trinitrobenzene
TNT	trinitrotoluene
2,4,5-TP	2,4,5-trichlorophenoxy acetic acid (silvex)
TPH	total petroleum hydrocarbon
UCL	upper control limit
VOC	volatile organic compound
v/v	volume to volume
W	water

SYMBOLS

Symbol	Definition
ug/kg	microgram per kilogram
ug/L	microgram per liter
ug/mL	microgram per milliliter
mg/kg	milligram per kilogram
mg/L	milligram per liter
uL	microliter
ug/m ³	microgram per cubic meter
um	micrometer
nm	nanometer

1. INTRODUCTION

1.1 STATEMENT OF POLICY

Paragon Analytics, Inc. (Paragon) is a full service, client-oriented environmental and radiochemistry testing laboratory. Paragon offers a wide range of analyses for radioactive, hazardous, and mixed waste samples. The management team's integrated approach to quality assurance, client service, and operations enables Paragon to provide compliant data that meet all technical and service requirements as prescribed by our clients. We perform analyses according to various federal and state QA/QC programs and analyze samples in strict accordance with promulgated methodologies, including: US EPA SW-846, US EPA CLP, Methods for Chemical Analysis of Water and Wastes (MCAWW), 40CFR, and DOE/HASL-300. Paragon specializes in serving the Department of Defense (DOD), the Department of Energy (DOE), and engineering/consulting companies. We routinely provide validatable data packages, custom reports, and custom electronic deliverables. Our strong commitment to producing high quality, compliant data and reporting data on time make Paragon a reliable team member.

1.2 CODE OF ETHICS

Paragon Analytics, Inc. is responsible for creating a work environment that enables all employees to perform their duties in an ethical manner. Paragon requires all employees to conduct themselves with integrity and honesty at all times. It is Paragon's expectation that all employees will exhibit professionalism and respect for clients and each other in all interactions and tasks. It is Paragon's expectation that each employee abide by the following responsibilities.

- Each Paragon employee is responsible for the propriety and consequences of his or her actions.
- Each Paragon employee is required to conduct themselves in a professional manner toward all clients, regulators, vendors, and other employees. Professional conduct relates to honesty, integrity, and tolerance for cultural diversity.

- Each Paragon employee will perform all assigned duties in accordance with Paragon's established policies and procedures that have been developed in substantial conformity with contractual and regulatory requirements. Every employee must disclose any instance of noncompliance. Further, Paragon may be obligated to report certain noncompliance issues to the client.
- It is the responsibility of each Paragon employee to report any suspicion of unethical conduct to a Manager.

Strict adherence to Paragon's Code of Ethics is essential to the reputation and continued health of the business. All Paragon employees are required to acknowledge their responsibility in acting in an ethical manner by attesting (in writing) to their intent to adhere to the requirements described above.

1.3 PURPOSE AND SCOPE

This document will describes Paragon's minimum QC procedures for sample analysis, as well as the administration of the laboratory QA program and the general QA program requirements. In the absence of client or project specific requirements, the processes and procedures described in this document will be followed by the laboratory for all programs in which the laboratory participates. Where client or project specific requirements exist, they will modify/supersede the requirements discussed in this LQAP.

Paragon operates a mobile laboratory that can perform on-site analytical services that may be required by a client. Because of the specific and limited nature of requirements that apply to the mobile laboratory during its operation, this LQAP will not discuss the activities that will be performed while engaged in project support. Before the mobile laboratory is placed in service at any project site, a site-specific Quality Assurance Project Plan will be developed by Paragon. This document will pertain only to the mobile laboratory's operations during its field support activities for the duration of a project.

1.4 QUALITY ASSURANCE DOCUMENTS

To efficiently manage and document its quality assurance program, Paragon generates several documents that support this effort. This Laboratory Quality Assurance Plan (LQAP), Standard Operating Procedures (SOPs) and Quality Assurance Project Plans (QAPjPs) are the key documents to support the overall quality assurance program. The sections below will describe in more detail the scope and intended use of these documents.

1.5 QUALITY ASSURANCE DOCUMENT HIERARCHY

There exists a hierarchy of documents in the quality assurance program implemented by Paragon that allows for comprehensive and flexible coverage of all project QA requirements. The basic document is the LQAP, which lists the practices followed by Paragon in the absence of any other project specific requirements.

Superseding this document are the individual SOPs detailing specific practices in the laboratory. Because the SOPs can be updated more frequently than the LQAP and they are more detailed in their discussion of common laboratory practices, where they differ from concepts discussed in the LQAP, the requirements of the SOPs will supersede the requirements of the LQAP.

The last (and highest) documents in this hierarchy are the QAPjP or “Program Specifications” used by Paragon for specific projects. The QAPjP is written either by the client or Paragon, and describes in detail the specific practices that must be used by Paragon for a given project. Program Specifications are generated by Paragon Project Managers and summarize project-specific requirements that are different from Paragon’s normal procedures. Because of the short-term nature of these documents and their finite scope, Program Specifications are often extremely detailed in their requirements. As a result, when these documents differ in their requirements and those that are present in the SOPs and LQAP, Program Specifications will supersede the other two documents.

1.5.1 QUALITY ASSURANCE PLAN

The LQAP is intended to be the basic document that describes the minimum requirements for all processes in the laboratory and presents an overview of how the laboratory functions in its technical operations. The LQAP is the main guidance document for laboratory operations when there exists no other project or program-specific requirements to which the laboratory must conform. When appropriate, this LQAP will be filed with a client and/or regulatory agency and after approval, may be referenced in lieu of repetitive submissions of plans in which only a portion of the information is changed.

This document will be reviewed and updated at a minimum frequency of once every two years, or more frequently if there are significant changes in procedures or capabilities in the laboratory. Updates to this document must be reviewed and approved by the Laboratory Manager and the Quality Assurance Manager before they are released for implementation by Paragon.

1.5.2 STANDARD OPERATING PROCEDURES

Standard Operating Procedures (SOPs) are documents that describe in detail how laboratory procedures will be performed by the staff. SOPs will be reviewed and updated at a minimum frequency of once every two years, or more frequently if there are significant changes in procedures (e.g., SW-846 update). A summary of available SOPs may be found in Appendix E. The SOPs may be used to describe general procedures that apply to all areas of the laboratory (i.e., Chain-of-Custody requirements, documentation procedures, health and safety procedures), or may apply only to specific processes and analytical procedures (i.e., analysis of pesticides by gas chromatography, digestion of soil samples for metals analysis, calibration of analytical balances). In all cases, SOPs provide detailed instructions to laboratory personnel that describe how to perform specific procedures within the laboratory.

Because of the technically detailed nature of the SOPs, laboratory staff performing these processes are the primary authors of these documents. When new SOPs are created or

existing SOPs are revised, the laboratory staff will initiate the creation/changes to the procedures. After the changes have been completed, the document will be reviewed and signed-off by the following personnel: a group leader or other staff member knowledgeable in the technical processes described by the SOP, the laboratory QA Manager, and the Laboratory Manager. Only after approval of the document by these staff members is the document is released for implementation in the laboratory.

1.5.3 PROJECT QUALITY ASSURANCE MANUALS

Project Quality Assurance Manuals (also called Quality Assurance Project Plans - QAPjPs) are similar to the LQAP except that they are limited in scope to cover only those processes and procedures that pertain to a specific project. These documents are written by the client or Paragon's Quality Assurance Department and are written to meet specific project requirements. After completion, these documents will be signed by the QA Manager and the Laboratory Manager, and delivered to the client or agency who required the QAPjP. After their approval, the document will be released for implementation in the laboratory.

For the duration of a project for which the QAPjP applies, the requirements in this document will take precedence over the LQAP (when there are deviations from the LQAP) for that project's samples, and for processes applied to those samples. With the completion of the project, the LQAP will be removed from service in the laboratory. If the duration of the project is long enough, the QAPjP may require periodic updating (as specified by the client), and revisions to this document will follow the same authorization procedure as was performed for its initial issuance.

1.5.4 DOCUMENT CONTROL, DISTRIBUTION AND REVISION

This LQAP will be revised periodically by the Quality Assurance Department as changes in procedures become necessary. Changes will be documented by the date and revision number of each section. When a section is revised, the revision date will replace the original date in the heading in the affected sections and the table of contents will be updated. Records of the distribution of the LQAP will be maintained by the QA

Department to ensure that all copies in the laboratory will be properly updated. If clients request copies of this document for review and approval, they will be generally issued as uncontrolled documents.

Project-specific QAPjPs will be distributed to laboratory operations managers when final approval from the client has been received. Only those managers whose groups will be involved in the analysis of project samples will receive copies of the QAPjP.

Additionally, the laboratory's Project Manager for that project will receive a copy of this plan. The QA Manager will retain the original document, and will make additional copies available to the laboratory staff as necessary.

Standard Operating Procedures are controlled documents, and the QA department will maintain records of their distribution to the laboratory. The originals will be kept in the QA Department's offices, and two full copies of all laboratory SOPs will be placed in designated locations in the laboratory. The laboratory copies will be printed on specially marked paper that indicates the controlled nature of these documents. Laboratory staff are allowed to make additional copies of the SOPs for ready reference in the laboratory, but will be required to destroy these extra copies when new revisions are made available.

The laboratory staff also has access to write-protected electronic copies of the SOPs. These electronic documents are marked with special headers to indicate (when printed) that these copies are for internal use only. As new SOP revisions are released, the QA Department will update the electronic files of all SOPs.

2. LABORATORY ORGANIZATION AND RESPONSIBILITIES

This section gives an overview of the Paragon Analytics, Inc. quality assurance organization and states key personnel, their responsibilities, and the lines of communication between these persons. In the event of a temporary absence, key personnel shall notify all employees of their absence and reassign their duties to another employee (e.g., a Project Manager may assign another Project Manager to cover his/her duties; an Operations Manager may assign a senior analyst to cover his/her duties; the Laboratory Manager may assign the Operations Manager to cover his duties).

An organizational/chain of command chart is attached as Appendix A. Personnel resumes are available upon request.

2.1 KEY PERSONNEL

2.1.1 LABORATORY MANAGER

This person is in charge of all laboratory operations, including business functions such as marketing and financial issues, and technical/administrative functions such as sample control, inorganic analysis, organic analysis, data management, and QA/QC. The Laboratory Manager directs the activity of the personnel in each operational section through the supervisors, who ensure that QC procedures are being performed and any out of control situations or discrepancies are remedied properly and promptly.

2.1.2 PROJECT MANAGER

The Project Manager serves as the primary point of contact between clients and Paragon. This person reports directly to the Laboratory Manager. Specific duties for this function are as follows:

1. Manage and coordinate the laboratory's participation from contract award, project initiation, sample preparation, sample analysis, report generation and shipment of required deliverables to a client.

2. Review all final reports for completeness, compliance with project requirements, clerical accuracy and reasonableness.
3. Ensure that all pertinent project information is disseminated to the appropriate laboratory personnel so that Data Quality Objectives are achieved.
4. Monitor holding times (if appropriate) and deliverable deadlines for all project sample analyses.
5. Review any QC deficiencies reported by the Section Managers, and coordinate any data changes resulting from review the QA Manager.
6. Oversee the set-up of the project by opening work order numbers, organizing the preparation of sample kits, and collecting all pertinent information to complete the project successfully.
7. Prepare and authorize for transmission of invoices to client for payment of project effort.
8. Maintains continuous contact with clients, and transmits any anomalous situations to them for resolution.

2.1.3 QUALITY ASSURANCE MANAGER

The Laboratory Quality Assurance Manager (LQAM) reports to the Laboratory Manager, and has responsibility for monitoring the quality of the laboratory's work, taking appropriate actions to ensure that quality standards are being met, to ensure that proper notification and data qualifiers are being used, and for stopping the release of data which are of suspect quality. The LQAM has a high degree of independence and authority in the laboratory's organization. The LQAM may be assisted in carrying out their responsibilities by a Laboratory Quality Assurance Specialist (LQAS). The LQAS may carry out any of the responsibilities of the LQAM. The LQAS reports to the Laboratory Manager and reviews the work of groups and individuals, and is generally independent of production pressures. The LQAM has the authority to generate a stop work order when systems are sufficiently out of control to compromise the integrity of the data generated by the laboratory.

The LQAM carries out the following activities within the laboratory's operations:

1. Coordinating the laboratory's certification program participation for all state and federal agencies.
2. Coordinating any on-site QA/QC inspections.
3. Revising or overseeing the revision of the laboratory QA/QC plan.
4. Monitoring compliance with the laboratory's QA/QC plan.
5. Coordinating the laboratory's participation in all relevant Performance Evaluation Programs (EPA WP, WS, EML, etc.).
6. Serving as laboratory point-of-contact for the exchange of QA/QC information, and preparing and approving, along with the laboratory manager, release of QA/QC information.
7. Scheduling the review and maintaining distribution records of controlled documents.

2.1.4 INFORMATION SERVICES MANAGER

This individual reports to the Laboratory Manager, and sets department goals, establishes priorities for specific projects, and evaluates performance. The IS manager will plan and execute the duties of the position and will keep the laboratory manager informed of all important events relating to information services activities. They are responsible for the implementation and support of the laboratory information systems to serve the needs of the technical, business and management functions of the laboratory. Specific duties include:

1. Supervise Information Services staff and management of the laboratory computer system. This includes establishing network server structure, security, maintenance, and backup procedures. Document operating procedures through SOPs or manuals. Serve as a technical resource on computer related issues.
2. Analyze information flow in the laboratory and suggest the most effective hardware, application software, and/or programming changes as solutions to meet long term customer requirements. Implement those changes in data by purchase of hardware

and applications software; or by software development, using the appropriate tools and methodology.

3. Determine specific customer requirements for electronic data format, and then meet the requirements for data submission.

2.1.5 INORGANIC DEPARTMENT MANAGER

This staff member reports to the Laboratory Manager, is responsible for all laboratory activities relating to the analysis and reporting of inorganic parameters, and is responsible for all data generated by the inorganics department. His specific responsibilities include:

1. Perform and/or supervise inorganic environmental analyses according to appropriate current laboratory protocols; including EPA-600, SW-846, and CLP SOW procedures.
2. Assign daily and longer range tasks to analysts and technicians assigned to the section; including QC requirements, deadlines, and assignment of personnel to maximize staff utilization.
3. Supervise training of subordinate personnel for new tasks and newly implemented methodologies.
4. Provide input to the overall laboratory coordination effort; including current workload deadlines, and requirements for anticipated or prospective work loads.
5. Control/monitor inventory requirements and evaluate expenditures.
6. Provide data and methodology input to efforts surrounding new method development and/or certifications.
7. Act as a technical resource for all inorganic staff for the development/performance of new or existing analysis methods.

2.1.6 RADIOCHEMISTRY DEPARTMENT MANAGER

The Radiochemistry Department Manager reports to the Laboratory Manager, is responsible for all laboratory activities relating to the analysis and reporting of radiochemistry parameters, and is responsible for all data generated by the radiochemistry department. His specific responsibilities are as follows:

1. Understand all radiochemical analysis procedures and act as a technical resource for the various analysis protocols performed by the laboratory. If new procedures are required for a project, they are responsible for the purchase of equipment, training of personnel and full implementation of all method requirements.
2. Supervise and train all radiochemistry analytical staff for all analytical procedures they will perform.
3. Assign job tasks and schedule project effort for all radiochemical analyses, keeping track of the following:
 - a. Project quality control;
 - b. Completion of work assignments to meet project turnaround times;
 - c. Assignment of workloads to balance and maximize staff utilization.
4. Coordinate and control the purchase of reagents, standards, glassware, equipment and other material used in the analytical methods, and ensure that they are of adequate quality and quantity to meet project goals.
5. Ensure that all equipment is adequately maintained, properly calibrated and correctly used for all project analyses.

2.1.7 GC/HPLC DEPARTMENT MANAGER

The GC/HPLC Department Manager reports to the Laboratory Manager, is responsible for all laboratory activities relating to the analysis and reporting of organic parameters (generated by GC/HPLC instruments), and is responsible for all data generated for the GC/HPLC department. His specific responsibilities are as follows:

1. Understand GC/HPLC procedures and act as a technical resource for the various analytical protocols, available instrumentation and methods performed by Paragon. This includes methodologies from EPA 600 series, SW-846 and CLP SOW procedures.
2. For new procedure development, responsibilities include the set-up and implementation of the new GC/HPLC method, including developing the QA/QC requirements that will apply to the new method.

3. Supervise and train GC and HPLC operators in the technical procedures performed at Paragon.
4. Assign job tasks and schedule projects for the GC and HPLC personnel, keeping track of the following: project quality control, completion of tasks within the required deadlines, maximizing staff utilization, and ensuring that all program/project requirements are met by the department's staff.
5. Meet with the Project Management staff and other Department Managers on a weekly basis to discuss quality issues, project deadline status, utilization and future workloads.
6. Control material inventory and usage. Anticipate the standards and materials needed for projects in advance to avoid "Rush Charges". Evaluate all materials as to background problems, prior to routine usage in the laboratory.
7. Maintain a routine preventive maintenance program on all instruments used in GC and HPLC analysis.

2.1.8 GC/MS DEPARTMENT MANAGER

The GC/MS Department Manager reports to the Laboratory Manager, is responsible for all laboratory activities relating to the analysis and reporting of organic parameters (generated by GC/MS instruments), and is responsible for all data generated for the GC/MS department. His specific responsibilities are as follows:

1. Understand GC/MS procedures and act as a technical resource for the various analytical protocols, equipment used and methods performed by Paragon. This includes EPA 600 series, SW-846 and CLP SOW procedures.
2. Assign daily and longer range tasks to analysts and technicians assigned to the section; including QC requirements, deadlines, and assignment of personnel to maximize staff utilization.
3. Train GC/MS operators in the technical procedures performed at Paragon.
4. Meet with the Project Management staff and other Department Managers on a weekly basis to discuss quality issues, project deadline status, utilization and future workloads.

5. Maintain a routine preventive maintenance program on all instruments used in GC/MS analysis.
6. Coordinate and control the purchase of reagents, standards, glassware, equipment and other material used in the analytical methods, and ensure that they are of adequate quality and quantity to meet laboratory requirements.

2.1.9 INORGANIC ANALYST

An Inorganic Analyst reports to the Inorganics Section Manager, and their specific responsibilities are as follows:

1. Perform routine inorganic environmental analyses using established methodologies. Independently set-up, adjust, calibrate, and operate common laboratory instrumentation included in areas of assigned responsibilities.
2. Perform and/or supervise appropriate sample preparations or extractions prior to analysis. Properly prepare and utilize standard reference solutions used in routine analyses.
3. Provide routine maintenance on assigned instrumentation or equipment. Take responsibility for the procurement of reagents and expendable supplies pertinent to areas of assigned responsibilities.
4. Generate laboratory-level reports from generated lab data, including preliminary QC sample performance evaluations.

2.1.10 RADIOCHEMISTRY ANALYST

This individual receives direction from the Radiochemistry Department Manager who sets goals, establishes priorities for workload, and evaluates performance. Each individual confers with their supervisor for clarification and discusses developments of on-going projects. Work is reviewed in terms of effectiveness, timeliness and compliance with company policies.

This position is responsible for several aspects of radiochemical analysis including report generation, sample analysis, compliance with standard methods, and meeting all QA/QC requirements for legal data quality. Specific responsibilities are as follows:

1. Perform routine radiochemical environmental analyses using established methodologies. Independently set-up, adjust, calibrate, and operate common laboratory instrumentation included in areas of assigned responsibilities.
2. Verifies that all documents are properly maintained and radiochemistry SOPs are followed including: Filling out all logbook pages, documenting all standards that are prepared, maintaining records for data archival, etc.
3. Generates complete reports according to the protocols required. Takes responsibility for the quality and accuracy of the report generated.
4. Assists other members of the radiochemistry group and other members of the laboratory as needed and as assigned by the Radiochemistry Department Manager.
5. Operates and repairs the radiochemistry analysis instrumentation and checks the associated QA/QC documentation including: logbooks, calibration, instrument drift, QC criterion, blank contamination, etc. Takes corrective action when necessary.

2.1.11 GC/HPLC ANALYST

This individual receives direction from the GC/HPLC Department Manager who sets goals, establishes priorities and evaluates performance. They plan and execute the duties of the position by conferring with their manager for clarification and to keep them apprised of developments and the status of on-going work. Work is reviewed in terms of effectiveness and timeliness and compliance with guidelines and company policies.

This position is responsible for several aspects of GC/HPLC analysis including report generation, sample analysis, compliance with standard methods, and meeting all QA/QC requirements for legal data quality. Specific responsibilities are as follows:

1. Generates complete reports according to the protocols required. Takes responsibility for the quality and accuracy of the reports generated.
2. Operates and repairs the GC/HPLC systems and checks the associated QA/QC documentation including: logbooks, calibration, instrument drift, QC criterion, blank contamination, etc. Takes corrective action when necessary.

3. Verifies that all documents are properly maintained and the SOPs are followed including: filling out all logbook pages, documenting all standards that are prepared, maintaining records for data archival, etc.
4. Verifies standard solutions prior to using them for analysis Tracks the traceability of all working standards to independently prepared, or EPA, reference standards.
5. Checks solvent lots for purity and performs other purity checks as the organic extraction group provides material for verification.
6. Assists other members of the GC/HPLC group and other members of the laboratory as needed and as time allows.

2.1.12 GC/MS ANALYST

This individual receives direction from the GC/MS Department Manager who sets goals, establishes priorities and evaluates performance. Plans and executes the duties of the position by conferring with their manager for clarification and to keep them apprised of developments and the status of on-going work. Work is reviewed in terms of effectiveness and timeliness and compliance with guidelines and company policies.

This position is responsible for several aspects of GC/MS analysis including report generation, sample analysis, compliance with standard methods, and meeting all QA/QC requirements for legally defensible data. Specific responsibilities are as follows:

1. Generates complete reports according to the protocols required. Takes responsibility for the quality and accuracy of the reports generated.
2. Performs mass spectral interpretation/evaluation of the output generated by the GC/MS data system. Is expected to exercise sound analytical judgment in determining the validity of the mass spectral match proposed by the MS system, and is expected to override and correct the output of the instrument when inappropriate output from the data system is detected.
3. Operates and repairs the GC/MS systems and checks the associated QA/QC documentation including: logbooks, calibration, instrument drift, QC criterion, blank contamination, etc. Takes corrective action when necessary.

4. Verifies that all documents are properly maintained and the SOPs are followed including: filling out all logbook pages, documenting all standards that are prepared, maintaining records for data archival, etc.
5. Verifies standard solutions prior to using them for analysis Tracks the traceability of all working standards to independently prepared, or EPA, reference standards.
6. Checks solvent lots for purity and performs other purity checks as the organic extraction group provides material for verification.
7. Assists other members of the GC/MS group and other members of the laboratory as needed and as time allows.

2.1.13 SAMPLE CUSTODIAN

This staff member must maintain the integrity and validity of all samples as they are received from the field. This person reports to the Sample Control Leader, who shares Program Management responsibilities with oversight of the Sample Custodian's activities. Specific duties for the Sample Custodian include:

1. Inspect all incoming samples and records to verify that they are received in good condition and with the proper containers/preservatives (as applicable). Perform the necessary screening procedures to verify the activity of the received field samples.
2. Complete field chain-of-custody forms and verify that no discrepancies exist between COC documentation and the actual samples received. Fill out and complete Paragon's sample receipt documentation. Any discrepancies discovered during login process are documented by the Sample Custodian, who will notify the appropriate Project Manager of any problems that requires client contact for resolution.
3. Log in samples into the laboratory's LIMS and initiate the notification of receipt of samples to all affected analytical sections. Notifies Project Manager of the receipt of samples and of requested analyses and required turnaround times for analyses.
4. Prepare sample kits for shipment to the field as required by project.

5. Maintain the inventory of sample containers to ensure adequate supplies are on hand to meet project needs.
6. Distribute samples to sample coolers in the laboratory for storage prior to analysis.

2.2 LABORATORY FACILITIES

Appendix C contains a diagram of the Paragon laboratory facility. The following paragraphs highlight the areas of the laboratory that are involved with sample receipt, handling and preparation of field samples.

2.2.1 SAMPLE RECEIPT AREA

Paragon's sample receiving area consists of a large dedicated room of more than 500 ft². It contains two fume hoods and radiation survey equipment to safely handle incoming radiochemistry and mixed waste samples. There is a direct outside access door to facilitate sample delivery, shipping, and sample kit preparation.

2.2.2 SAMPLE STORAGE AREA

The laboratory has a large walk-in cooler in the sample receiving area used for centralized storage of general project samples. Additionally, there are several sample storage locations in various work areas of the laboratory reserved exclusively for the storage of samples scheduled for specific analysis groups. Segregated storage is provided for the following areas: organic extractions, volatiles analyses, inorganic/metals analyses, fuels analyses, and radiochemistry analyses.

2.2.3 SAMPLE PREPARATION AREAS

The laboratory has eight sample preparation/extraction/digestion areas divided into six radiochemistry preparation areas, one organics extraction lab and one inorganic preparation/digestion lab in a total of approximately 4500 ft² of floor space. Laboratory preparation procedures are segregated as much as possible to minimize the potential for contamination, maximize processing efficiency, and maintain analytical integrity. Rigorous washing/cleaning of glassware and apparatus ensure that cross-contamination is kept to a minimum. Each laboratory area is on a dedicated or locally shared HVAC

system that continuously exchanges the laboratory air with filtered and conditioned outside air. Each sample preparation area has at least one hood that is capable of maintaining an average face velocity of 100 feet per minute, and there are a total of 34 hoods in the eight sample preparation areas.

2.2.4 DEIONIZED WATER SYSTEM

Within the laboratory, there are three deionized (DI) water distribution systems available for glassware cleaning, bulk reagent preparation (acid and base solutions and other aqueous reagents), and general use. DI water is defined as municipal tap water which has been treated by passing through a standard resin column and an activated carbon unit, with a final “polishing” cartridge as the last unit in the processing chain. This water contains no detectable (Paragon’s routine reporting limits) heavy metals or inorganic compounds of analytical interest, is relatively free of organic compounds, and meets the requirements specified for ASTM Type II water (10 uohm resistivity or greater).

Ultra-pure water, used for equipment blanks and standards preparation is defined as DI water that has been additionally treated through a Milli-Q (or equivalent) treatment system and contains no organic compounds of analytical interest above Paragon’s routine reporting limits. One Milli-Q system is available at the laboratory, and it is capable of continuously delivering water that meets the requirements specified for ASTM Type I water (18 uohm resistivity or greater). If clients request that Paragon supply field sampling teams with reagent blank water (excluding volatile trip blanks), ultra-pure water will be collected and sent to the client.

Organic-free water, used for volatile trip blanks, GC, and GC/MS volatile instrument blanks is prepared by purging ultra-pure water for 24 hours with high-purity nitrogen.

2.3 ANALYTICAL INSTRUMENTATION

Appendix B of this LQAP lists all major equipment currently available at this laboratory.

3. QUALITY ASSURANCE OBJECTIVES

The purpose of this Laboratory Quality Assurance Plan (LQAP) is to define procedures for the documentation, evaluation, validation, and reporting of data. The objective is to provide a uniform basis for sampling, sample handling, instrument maintenance and calibration, methods control, performance evaluation, and analytical data generation and reporting. Specific, detailed procedures to be used for sampling, chain of custody, calibration of instruments, laboratory analysis, reporting, internal quality control, audits, preventive maintenance, and corrective actions are described in various sections of this plan.

As stated, Paragon's objective is to provide data of known quality. To accomplish this task, Paragon will:

- Maintain an effective, on-going QA/QC program that measures and verifies laboratory performance.
- Provide a Quality Assurance Department independent of the pressures of production schedules that has the responsibility and authority to audit and develop corrective action procedures.
- Provide sufficient flexibility to allow controlled changes in routine methodology in order to achieve client-specific data requirements as prescribed in project-specific quality plans.
- Recognize as soon as possible and correct any factors that adversely affect data quality.
- Monitor operational performance of the laboratory on a routine basis and provide corrective action as needed. Maintain complete records of sample submittal, raw data, laboratory performance, and completed analyses to support reported data.

3.1 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) are qualitative and quantitative statements developed by data users that specify the quality of data from field and laboratory data collection activities in order to support specific decisions or regulatory actions. The DQOs describe what data are needed, why the data are needed, and how the data will be used to address the problem being investigated. DQOs also establish numeric limits for the data to allow the data user or reviewer determine whether the data are of sufficient quality for their intended application.

A means to measure the attainment of stated DQOs is through the use of five characteristics: precision, accuracy, representativeness, completeness, and comparability (PARCCs parameters). The sections that follow discuss the definition and application of these parameters. The QA protocols used for the majority of analyses performed are taken from the following sources: EPA Contract Laboratory Program's Statement of Work (Organics and Inorganics), 40 CFR 136 Methodologies, and SW 846 methodologies that contain detailed descriptions of the quality control measures routinely employed.

3.2 LEVEL OF QA EFFORT

The reliability of data generated in the laboratory will be evaluated at the 99% confidence level (mean \pm 3 standard deviations) for control and at the 95% confidence level (mean \pm 2 standard deviations) for warning. Precision of analyses will be evaluated using sample duplicates and/or matrix spike duplicates. Accuracy will be monitored by calculating the recovery of analytes from surrogate spikes, matrix spikes, tracers, EPA reference check standards, and Proficiency Testing (PT) standards.

3.3 PRECISION

Precision is a statistical measurement of the reproducibility of repetitive measurements. It is strictly defined as the degree of mutual agreement among independent measurements

as the result of repeated application of the same process under similar conditions.

Analytical precision is a measurement of the variability associated with duplicate (two) or replicate (more than two) analyses of the same sample in the laboratory and is determined by analysis of laboratory duplicates. Total precision is a measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples and incorporates variability introduced by both the laboratory and field operations.

Precision data must be interpreted by taking into consideration the possible sources of variability. Duplicate (two) samples or spiked samples are analyzed to assess field and analytical precision and the results are assessed using the relative percent difference (RPD) between duplicate measurements. Precision objectives are presented for each analytical method.

Analytical precision shall be evaluated by using matrix spike/matrix spike duplicates (MS/MSD), by LCS pairs, or by using duplicate samples. Precision is independent of the bias (accuracy) of the analyses and reflects only the degree to which the measurements agree with one another, not the degree to which they agree with the "true" value of the parameter measured.

Precision is calculated in terms of Relative Percent Deviation (RPD), which is calculated as follows:

$$RPD(\%) = \frac{X_1 - X_2}{(X_1 + X_2) / 2} (100)$$

Where:

RPD = relative percent different

X₁, X₂ = value of sample 1 and sample 2

RPDs are compared to the laboratory-established RPD control limits for the analysis. Sample homogeneity/non-homogeneity is an important factor that influences the precision of duplicate samples for stable chemistry analyses.

For radiochemical analyses, precision is measured in terms of Duplicate Error Ratio (DER), which is calculated as follows:

$$DER = \frac{|S - D|}{2 * \sqrt{\sigma_s^2 + \sigma_d^2}}$$

Where:

DER = Duplicate Error Ratio

S, D = values of (S)ample and (D)uplicate

σ = One sigma error value associated with sample result

DERs are compared to the laboratory established control values for the analysis. Sample homogeneity/non-homogeneity is an important factor that influences the precision of duplicate samples for radiological analyses.

The analyst, department manager, and/or technical manager must investigate the cause of values outside stated acceptance limits. Follow-up action may include: recalibration, reanalysis of QC samples, sample reanalysis, or flagging and qualification of the data.

3.4 ACCURACY

Accuracy is a statistical measurement of agreement between the measured and known or correct value. Accuracy is influenced by random error (variability due to imprecision) and systematic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Accuracy is typically measured by determining the percent recovery of known target analytes that are spiked into a field

sample (a surrogate or matrix spike) or reagent water (laboratory control sample). Surrogate compound recovery is reported and is used to assess method performance for each sample analyzed for volatile and semivolatile organic compounds. For organic and inorganic parameters, the stated accuracy objectives apply to spiking levels at approximately five times the method detection limits (MDLs) or at the midpoint of the calibration curve. For radiochemical analyses, the spiking levels for the control spikes may vary from five to fifty times the method reporting limits.

Results for blanks, matrix spikes, LCSs, and surrogates will be the primary indicators of accuracy. These results will be used to control accuracy by requiring that they meet specific criteria. As spiked samples are analyzed, spike recoveries will be calculated and compared to pre-established acceptance limits.

The calculation formula for percent recovery is:

$$R(\%) = \frac{(C_1 - C_2)(100)}{C_3}$$

Where:

R% = Spike amount recovered

C₁ = Concentration of analyte in spiked sample

C₂ = Concentration of analyte in unspiked sample

C₃ = Concentration of spike added

Acceptance limits will be based upon previously established laboratory performance for similar samples. In this approach, the control limits reflect the minimum and maximum recoveries expected for individual measurements for an in-control system. Recoveries outside the established limits indicate some assignable cause, other than normal measurement error, and possible need for corrective action. This includes recalibration of the instrument, reanalysis of the QC sample, reanalysis of the samples in the batch,

repreparation of samples in the batch, or flagging and qualifying the data as suspect if the problems cannot be resolved. For contaminated samples, recovery of matrix spikes may depend on sample homogeneity, matrix interference, and dilution requirements for quantitation.

Both accuracy and precision are calculated for analytical batches, and the associated sample results must be interpreted by considering these specific measures. The QA objectives for precision and accuracy are to achieve the QC acceptance criteria specified in the proposed analytical procedures. For the organic and inorganic procedures, the precision and accuracy guideline requirements are specified in the individual methods.

Field blanks and duplicates are collected and analyzed to assess field sampling activities. The results check procedural contamination and/or ambient conditions at the site.

Due to the extensive number of organic parameters and potential matrices, the development of precision and accuracy objectives and control limits for every matrix is difficult. This is typically done with (1) matrix spike and matrix spike duplicate compounds which are added to selected samples before extraction and analysis, and/or (2) surrogate spike compounds which are added to every sample, before extraction and analysis. Although the surrogate and matrix spike analyses do not provide statistically valid statements about precision and accuracy for every compound in a sample, they do give the data reviewer enough information to make judgments about precision and accuracy on a sample-by-sample basis.

Inorganic precision and accuracy data are determined by using duplicate samples or matrix spike duplicate samples (precision), matrix spike and laboratory control samples (accuracy). The following procedure is used:

For a duplicate sample analysis, at least one duplicate sample is analyzed per sample matrix type (e.g. water, soil) and concentration (e.g. low, medium) per batch of samples or for each

20 samples received, whichever is more frequent, or as specified by state/project requirements. Samples identified as field blanks can NOT be used for duplicate samples analyses. If two analytical methods are used to obtain the reported values for the same element for a batch of samples (i.e., ICAP, GFAA), duplicate samples will be run by each method. The relative percent difference (RPD) for each component is calculated for later use during data assessment.

Radiochemical precision and accuracy data are generated from the results of duplicate samples or matrix spike duplicate samples (precision), laboratory control sample duplicates (precision) and laboratory control samples (accuracy). For a duplicate sample analysis, at least one duplicate sample is analyzed per sample matrix type (e.g. water, soil) and concentration (e.g. low medium) per batch of samples or for each 20 samples received, whichever is more frequent, or as specified by project specific requirements. Samples identified as field blanks can NOT be used for duplicate samples analyses. Percent accuracy is the most commonly used measure of accuracy in radiochemistry. The most commonly used measure of precision in radiochemistry is duplicate error ratio (DER).

Generally, established QC limits are laboratory specific, having been statistically derived from an individual laboratory's data. QC objectives for a specific project will be included in a project specific QAPP or for general information as a facility specific addendum to this document. QC limits are used to judge acceptability of data generated by the laboratories. Where acceptability criteria do not exist for a given method being utilized for the first time, the laboratories will establish control limits derived from a minimum of four data points. Until verified by a statistically significant data population, the control limits will be considered as advisory limits only and will not automatically initiate a rerun or reanalysis criteria if they are not met.

The QC limits for accuracy and precision are developed based upon laboratory derived data. When applicable, control limits established by the EPA CLP are used to judge acceptability

of data generated by the laboratories. Where EPA acceptability criteria does not exist for a given method being utilized for the first time, the laboratory will establish control limits derived from a minimum of four data points. Until verified by a statistically significant data population, the control limits will be considered as advisory limits only and will not automatically initiate a rerun or reanalysis criteria if they are not met.

3.5 REPRESENTATIVENESS

Representativeness is a qualitative element that is related to the ability to collect a sample that accurately reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. It is specifically addressed in the work plan.

Representativeness is a measure of how closely the measured results reflect the actual concentration or distribution of the chemical compounds in the sample. Sample handling protocols (e.g., storage, preservation and transportation) have been developed to preserve the representativeness of the samples. Proper documentation will establish that protocols have been followed and sample identification and integrity ensured. Every attempt will be made to ensure that the aliquots taken for analysis are homogeneous and representative of the samples received.

3.6 COMPARABILITY

Comparability qualitatively expresses the confidence with which one data set can be compared to another data set. Comparability is ensured through the use of established, standardized, and approved sample collection techniques and analytical methods, consistency in the basis of analysis (wet or dry weight, volume, etc.), consistency in reporting units, and analysis of standard reference materials. Organic analysis results will be reported in $\mu\text{g/L}$ for water samples and $\mu\text{g/kg}$ for soil samples. Water and soil samples analyzed for trace metals and cyanide will be reported in $\mu\text{g/L}$ and mg/kg , respectively. Water samples analyzed for miscellaneous water quality parameters will be reported in

mg/L. Results for soil samples will usually be reported on a dry-weight basis, except for analysis results going to those agencies (State or Federal) that accept wet-weight analysis results. Additionally, for those methods in which the performance of the procedure obviates the need for moisture percent correction (such as explosives in soil by 8330, or tritium in soils), the laboratory will report the appropriate units mandated by the method. When moisture determinations have been performed, percent moisture results will be presented on all forms listing analytical results.

Radiochemical parameters are generally reported in units of picocuries per liter (pCi/L) or picocuries per gram (pCi/g). Kinetic Phosphorescence Analysis of Uranium is usually measured and reported in units of $\mu\text{g/g}$ or $\mu\text{g/L}$.

Comparability is also considered during preparation of a site-specific work plan. The objective of comparability is to ensure that results of similar activities conducted by different parties are comparable. Paragon uses EPA-approved or other methods and procedures to ensure comparability with data from previous or following studies (interlaboratory comparability). Paragon participates in external and interlaboratory proficiency testing (PT) studies to demonstrate our ability to produce accurate and comparable data.

3.7 COMPLETENESS

Completeness is a measure of all information necessary for a valid scientific study. For completeness, it is expected that the methodology proposed for chemical characterization of the samples collected will provide data meeting QC acceptance criteria following standard laboratory data review and validation for at least 90-95% of all samples collected.

Completeness may also be defined as a comparison of the number of tests successfully completed (with acceptable QC) to the number of tests requested. Discrepancy reports are completed to provide explanation when QC criteria are not met.

Every attempt will be made to generate completely valid data. However, it is recognized that some samples will exhibit highly contaminated matrices necessitating multiple analyses and/or extensive dilutions. As a result of these atypical applications, recoveries and MDLs/MDCs may be deemed questionable based on internal QC results by the external data validation process. The objective will be to have 90-95% completeness on samples unaffected by matrix interferences. For uncontaminated background samples and first time samples not showing interferences, completeness should be 100% with a requirement for reanalysis of these critical samples mandatory if the objective is not met.

The equation used to calculate this number is shown below:

$$C\% = \frac{S}{R} (100)$$

Where:

C = completeness

S = number of successful analyses

R = number of requested analyses

Successful analyses are defined as those where the samples arrived at the laboratory intact, properly preserved, in sufficient quantity to perform the requested analyses, and accompanied by a completed chain of custody. Furthermore, the sample must be analyzed within the specified holding time (if applicable) and in such a manner that analytical QC described in this document are met.

Factors that adversely affect completeness include:

- Receipt of samples in broken containers.
- Receipt of samples in which chain of custody or sample integrity is compromised in some way.

- Samples received with insufficient volume to perform initial analyses or repeat analyses if initial efforts do not meet QC acceptance criteria.
- Improperly preserved samples.
- Samples held in the field or laboratory longer than expected, thereby jeopardizing holding time requirements. This is defined as samples that arrive at the laboratory after 50 percent of the regulatory holding time has expired.
- Samples containing high levels of contamination that can exhibit persistent effects on instrumentation designed for trace-level analyses.

Completeness for the entire project also involves completeness of field and laboratory documentation, whether all samples and analyses specified in the work plans have been processed, and the procedures specified in supporting documents (e.g., the QAPjP) have been implemented.

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 as a result of random error (preamble to 40 CFR Part 136, Vol. 49, No. 209, October 26, 1984). As the number of compounds measured increases in a given sample, the probability for realizing statistical error also increases.

The number of compounds present in numerous EPA methods (e.g. GC/MS methods 8260B and 8270C, metals included in ICAP method 6010B, and gamma spectroscopy parameters measured by method 901.1) increases the probability that one or more analytes will not meet acceptance criteria to significantly more than the 5% per analyte frequency. The number of target analytes included in these tests can be used to show that a minimum of four to seven target analytes will exceed the control limits established for these methods as a result of the statistical probability for random error. The establishment of QC criteria that are not consistent with the measurement of the quality objectives for which they are intended should be discouraged.

3.8 METHOD DETECTION LIMITS

The Method Detection Limit (MDL) is 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. The MDL is defined as follows in 40 CFR Part 136 Appendix B:

$$\text{MDL} = t_{(n-1, 1-\alpha, = 0.99)} \times S$$

Where:

- MDL = method detection limit;
- S = Standard deviation of the replicate analyses;
- $t_{(n-1, 1-\alpha, = 0.99)}$ = Student's t-value appropriate to a 99-percent confidence level.

Paragon performs annual MDL studies for each determinative method, matrix, instrument, and analytical column. Paragon performs its MDL studies according to 40CFR Part 136 Appendix B guidelines. In summary, a minimum of eight (8) replicates are spiked with the same concentration of the analytes of interest such that the spike concentration is between one (1) and 10 times the calculated MDL (“10x rule”). Paragon requires that the calculated MDL value is at least three times lower than the reporting limit (“3x rule”). Paragon will accept marginal sporadic failures for the rules cited.

MDL values are established for the determinative methods used by Paragon. For some methods (e.g., pH, total dissolved solids, total suspended solids, hardness, alkalinity, soil permeability, grain size, etc.), the determination of an MDL is not relevant. Reporting limits (where applicable) for these parameters are established based on the laboratory's knowledge of extraction efficiency, instrument sensitivity, and experience with the procedure.

3.9 METHOD REPORTING LIMITS

Paragon defines a method Reporting Limit (RL) as the analyte concentration, at or above which, the laboratory's precision and accuracy requirements can be routinely demonstrated and achieved. The RL values for most analytes reported by Paragon are numbers that are between 3 to 10 times the value of the MDL for those analytes. For analyte concentrations that fall between the MDL and the RL, the laboratory is not able to maintain the precision and accuracy specified for an analysis technique, and sample concentrations in this range are flagged as being estimated.

3.10 MINIMUM DETECTABLE CONCENTRATION

The Minimum Detectable Concentration (MDC) is used for radiochemical procedures. It is defined as the concentration at which there is a 95 % confidence that an analyte signal will be detected, and a 95 % confidence of avoiding reporting a false positive.

The general formula for calculating the MDC is based on work done by Currie (Currie, L.A., "Limits for Qualitative Detection and Quantitative Determination", Analytical Chemistry 40(3); pp. 586-693; 1968) and is generally calculated as follows:

$$MDC = \frac{(4.65 X \sigma_b) + 2.73}{T * K}$$

Where:

- MDC = Minimum Detectable Concentration
- σ_b = Standard deviation of the measurement background;
- T = Sample count time;
- K = Factor incorporating efficiency, abundance, aliquot yield, ingrowth and decay, and activity conversion factors.

3.11 TOTAL PROPAGATED UNCERTAINTY

Total Propagated Uncertainty (TPU) is an estimated measure of “total uncertainty” in a radiochemical result. Various sources of uncertainty associated with the preparation (aliquot yield) and measurement process (efficiency, counting uncertainty) are propagated using the law of propagation of uncertainty. The TPU is an integral part of every radiochemical result and is reported as \pm TPU.

3.12 TRACEABILITY

Traceability is the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards and that which explicitly describes the history of each sample from collection to analysis. Refer to the sections on sample custody and records management for more information about Paragon’s procedures.

3.13 QUALITY ASSURANCE PROJECT PLAN EXCEPTIONS

Due to the unknown nature of environmental samples prior to analysis, Paragon has minimal control over analytical and quality control complications that arise from unique sample matrix conditions. These conditions may include such items as highly concentrated samples containing target compounds of interest and/or non-target components; extremes in sample pH, viscosity, and solubility; and high organic content (both natural and synthetic). Each of these conditions presents a variety of challenges to the laboratory.

Most often these extremes in sample matrix require the laboratory to employ dilution techniques in order to change the sample state into one that can be analyzed by the desired protocol. Unfortunately, dilution techniques raise reporting limits and often adversely impact the surrogate standard and matrix spiking acceptance criteria.

The laboratory has the responsibility to clearly identify cases where matrix interferences preclude the generation of "compliant" data. This determination may be made by

demonstrating reproducibility (i.e., reanalysis of the affected sample) -- that the quality control measurement failure resulted from unique sample matrix conditions beyond the control of laboratory -- and not as a result of laboratory error. For example, in situations which the surrogate standard recoveries fall outside of control limits, samples are re-extracted and/or re-analyzed. Similar "non-compliant" results in the reanalysis indicate that it is something inherent to the sample that prevented the laboratory from reporting results deemed method compliant under data validation criteria.

Analytical projects containing particularly "dirty" samples (i.e., highly contaminated with target compounds and/or matrix co-extractives) will often fail to meet pre-established QA completeness goals (set forth in the QAPjP) when prior site history does not reveal the potential for excessive values. Again, while the laboratory performs all analytical testing and clean up procedures by the prescribed protocols, the results obtained may not meet validation criteria as a result of elevated reporting limits or the frequency at which surrogate and matrix spikes failed to meet acceptance limits. In cases where the laboratory is unable to meet QC criteria because of sample matrix complications beyond their control, results which are flagged or "qualified" by data validation guidelines are often still "useable" by the end user of the data.

Paragon is committed to adhering to method requirements and program quality control applications as established by our client and will work rigorously to provide data of the highest quality possible. Because of the uncertainties associated with environmental samples, Paragon will not assume responsibility for conditions beyond our reasonable control which directly impact the "validity" versus the usability of the associated analytical data generated.

3.14 SUMMARY OF METHOD OBJECTIVES

The following tables present Paragon's statements of typical performance for all methods routinely performed at this laboratory. Historical Paragon data have been used as the

basis for developing acceptance criteria for assessing the precision and accuracy for most of the methods presented. In the event that a significant historical collection of data generated by Paragon is lacking for an analysis method, default values such as EPA precision and accuracy data are presented for the individual methods. ***Paragon notes that the values presented in the following tables are intended to be representative and are subject to change. Consult the QA Department and LIMS database for current values.***

Representative criteria to be used for assessment of method performance are given in Table 3.1 - 3.27. Table 3.28 at the end of this section lists the sample preparation methods routinely used by Paragon.

3.15 DEFINITION OF TERMS

The following is a brief explanation of the terms that appear in the following tables.

Items in the tables that are not applicable are denoted by "NA." Note that if both water and soil analyses are listed in the tables and only one set of precision and accuracy criteria is listed, then the criteria applies to both matrices.

Reference: The reference to the standard analytical methodology used for each procedure.

Reporting Limit: Paragon's standard value reportable for a given method. Actual reporting limits may vary from reporting limits shown in the following tables, depending on sample aliquot, cleanup procedures performed, preparatory and/or analytical dilutions performed, and current method detection limit (MDL) values.

Table 3.1. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR WET CHEMISTRY (INORGANIC) ANALYSES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Ammonia	Water	EPA 350.3	15	85-115	0.50 mg/L
Acidity	Water	EPA 305.1	15	85-115	5.0 mg/L
Alkalinity (Total)	Water	EPA 310.1	15	85-115	5.0 mg/L
Bicarbonate	Water	EPA 310.1 mod	15	85-115	5.0 mg/L
Carbonate	Water	EPA 310.1 mod	15	85-115	5.0 mg/L
Hydroxide	Water	EPA 310.1 mod	15	85-115	5.0 mg/L
Chloride	Water	EPA 325.3	15	85-115	5.0 mg/L
Total Cyanide	Water	EPA 9010B, 335.3	30	63-131	0.010 mg/L
	Solid	EPA 9010B	30	63-131	0.50 mg/kg
Fluoride	Water	EPA 340.2	15	85-115	0.50 mg/L
Hardness (Calculation)	Water	SM 2340B	NA	NA	2.50 mg/L
Hexavalent Chromium	Water	EPA 7196A	20	80-120	0.01 mg/L
	Solid	EPA 7196A (aqueous leachate)	25	75-125	0.50 mg/kg
	Solid	EPA 7196A (alkaline digestion)	25	75-125	10 mg/kg
NO ₂ + NO ₃ as N	Water	EPA 353.3	20	80-120	0.050 mg/L
NO ₃ as N	Water	EPA 353.3	20	80-120	0.050 mg/L
NO ₂ as N	Water	EPA 354.1	20	80-120	0.010 mg/L

Table 3.1. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR WET CHEMISTRY (INORGANIC) ANALYSES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
pH	Water	EPA 9040B, 9045C	±0.1 units	N/A	N/A
		EPA 150.1	±0.1 units	N/A	N/A
Ortho Phosphate as P	Water	EPA 365.2	20	80-120	0.050 mg/L
Total Phosphorous as P	Water	EPA 365.2	20	80-120	0.050 mg/L
Specific Conductivity	Water	EPA 9050A	10	75-125	1.0 µmho/cm
		EPA 120.1	10	75-125	1.0 µmho/cm
Sulfate	Water	EPA 375.4	25	75-125	5.0 mg/L
Sulfide	Water	EPA 376.1	20	80-120	5.0 mg/L
TDS	Water	EPA 160.1	15	85-115	20 mg/L
TSS	Water	EPA 160.2	15	85-115	20 mg/L
Fluoride	Water	EPA 300.0, EPA 9056	20	80-120	0.10 mg/L
Chloride	Water	EPA 300.0, EPA 9056	20	75-115	0.20 mg/L
Bromide	Water	EPA 300.0, EPA 9056	20	85-115	0.20 mg/L
Nitrate as N	Water	EPA 300.0, EPA 9056	20	85-115	0.20 mg/L
Nitrite as N	Water	EPA 300.0, EPA 9056	20	85-115	0.10 mg/L
Ortho Phosphate as P	Water	EPA 300.0, EPA 9056	20	85-115	0.2 mg/L
Sulfate	Water	EPA 300.0, EPA 9056	20	85-115	1.0 mg/L
Reactive Cyanide	Solid	EPA SW 846 (7)	NA	N/A	0.10 mg/kg
Reactive Sulfide	Solid	EPA SW 846 (7)	NA	N/A	50 mg/kg

Table 3.1. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR WET CHEMISTRY (INORGANIC) ANALYSES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Total Organic Carbon	Water	EPA 415.1, EPA 9060	20	80-120	1.0 mg/L
Total Organic Halides	Water	EPA 9020B	20	80-120	10 µg/L
Adsorbable Organic Halides (AOX)	Water	EPA 1650	20	71-116	20 µg/L
Extractable Organic Halides (EOX)	Solid	EPA 9023	20	75-125	10 mg/kg
Total Halides	Oil	EPA 9076	20	80-120	25 mg/kg
Oil & Grease	Water	EPA 413.2, EPA 418.1	20	70-130	1.0 mg/L
	Solid	EPA 413.2 mod, EPA 418.1 mod	20	70-130	30 mg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.2. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR METALS ANALYSES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Aluminum	Water	EPA 6010B, 200.7	20	80-120	0.20 mg/L
	Solid	EPA 6010B			20 mg/kg
Antimony	Water	EPA 6010B, 200.7 (Radial)	20	80-120	0.06 mg/L
	Water	EPA 6010B, 200.7 (Axial)			0.02 mg/L
	Solid	EPA 6010B (Radial)			6.0 mg/kg
	Solid	EPA 6010B (Axial)			2.0 mg/kg
Arsenic	Water	EPA 6010B, 200.7 (Radial)	20	80-120	0.06 mg/L
	Water	EPA 6010B, 200.7 (Axial)			0.01 mg/L
	Solid	EPA 6010B (Radial)			6.0 mg/kg
	Solid	EPA 6010B (Axial)			1.0 mg/kg
Barium	Water	EPA 6010B, 200.7	20	80-120	0.10 mg/L
	Solid	EPA 6010B			10 mg/kg
Beryllium	Water	EPA 6010B, 200.7	20	80-120	0.005 mg/L
	Solid	EPA 6010B			0.50 mg/kg
Boron	Water	EPA 6010B, 200.7	20	80-120	0.10 mg/L
	Solid	EPA 6010B			10 mg/kg
Calcium	Water	EPA 6010B, 200.7	20	80-120	1.0 mg/L
	Solid	EPA 6010B			100 mg/kg

Table 3.2. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR METALS ANALYSES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Cadmium	Water	EPA 6010B, 200.7	20	80-120	0.005 mg/L
	Solid	EPA 6010B			0.50 mg/kg
Chromium	Water	EPA 6010B, 200.7	20	80-120	0.01 mg/L
	Solid	EPA 6010B			1.0 mg/kg
Cobalt	Water	EPA 6010B, 200.7	20	80-120	0.01 mg/L
	Solid	EPA 6010B			1.0 mg/kg
Copper	Water	EPA 6010B, 200.7	20	80-120	0.01 mg/L
	Solid	EPA 6010B			1.0 mg/kg
Iron	Water	EPA 6010B, 200.7	20	80-120	0.10 mg/L
	Solid	EPA 6010B			10 mg/kg
Lead	Water	EPA 6010B, 200.7 (Radial)	20	80-120	0.05 mg/L
	Water	EPA 6010B, 200.7 (Axial)			0.003 mg/L
	Solid	EPA 6010B (Radial)			5.0 mg/kg
	Solid	EPA 6010B (Axial)			0.30 mg/kg
Lithium	Water	EPA 6010B, 200.7	20	80-120	0.01 mg/L
	Solid	EPA 6010B			1.0 mg/kg
Magnesium	Water	EPA 6010B, 200.7	20	80-120	1.0 mg/L
	Solid	EPA 6010B			100 mg/kg

Table 3.2. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR METALS ANALYSES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Manganese	Water	EPA 6010B, 200.7	20	80-120	0.010 mg/L
	Solid	EPA 6010B			1.0 mg/kg
Mercury	Water	EPA 245.1, 7470	20	80-120	0.0002 mg/L
	Solid	EPA 7471, 245.5			0.10 mg/kg
Molybdenum	Water	EPA 6010B, 200.7	20	80-120	0.010 mg/L
	Solid	EPA 6010B			1.0 mg/kg
Nickel	Water	EPA 6010B, 200.7	20	80-120	0.020 mg/L
	Solid	EPA 6010B			2.0 mg/kg
Potassium	Water	EPA 6010B, 200.7	20	80-120	1.0 mg/L
	Solid	EPA 6010B			100 mg/kg
Sodium	Water	EPA 6010B, 200.7	20	80-120	1.0 mg/L
	Solid	EPA 6010B			100 mg/kg
Selenium	Water	EPA 6010B, 200.7 (Radial)	20	80-120	0.10 mg/L
	Water	EPA 6010B, 200.7 (Axial)			0.0050 mg/L
	Solid	EPA 6010B (Radial)			10 mg/kg
	Solid	EPA 6010B (Axial)			0.50 mg/kg
Silicon	Water	EPA 6010B, 200.7	20	80-120	0.050 mg/L
	Solid	EPA 6010B			5.0 mg/kg

Table 3.2. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR METALS ANALYSES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Silver	Water	EPA 6010B, 200.7	20	80-120	0.010 mg/L
	Solid	EPA 6010B			1.0 mg/kg
Thallium	Water	EPA 6010B, 200.7 (Radial)	20	80-120	0.20 mg/L
	Water	EPA 6010B, 200.7 (Axial)			0.010 mg/L
	Solid	EPA 6010B (Radial)			20 mg/kg
	Solid	EPA 6010B (Axial)			1.0 mg/kg
Titanium	Water	EPA 6010B, 200.7	20	80-120	0.020 mg/L
	Solid	EPA 6010B			2.0 mg/kg
Tin	Water	EPA 6010B, 200.7	20	80-120	0.050 mg/L
	Solid	EPA 6010B			5.0 mg/kg
Vanadium	Water	EPA 6010B, 200.7	20	80-120	0.010 mg/L
	Solid	EPA 6010B			1.0 mg/kg
Zinc	Water	EPA 6010B, 200.7	20	80-120	0.020 mg/L
	Solid	EPA 6010B			2.0 mg/kg

Note: Reporting limits listed above are from radially-viewed instrumentation unless otherwise noted. Axially-viewed instrumentation can achieve lower reporting limits for selected metals (e.g., arsenic, lead, selenium, thallium).

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.3. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR **TCLP** METALS ANALYSES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Arsenic	Leachate	EPA 1311/6010B (Axial)	20	80–120	0.10 mg/L
Barium	Leachate	EPA 1311/6010B (Axial)	20	80–120	1.0 mg/L
Cadmium	Leachate	EPA 1311/6010B (Axial)	20	80–120	0.050 mg/L
Chromium	Leachate	EPA 1311/6010B (Axial)	20	80–120	0.10 mg/L
Lead	Leachate	EPA 1311/6010B (Axial)	20	80–120	0.030 mg/L
Mercury	Leachate	EPA 1311/7470	20	80–120	0.0020 mg/L
Selenium	Leachate	EPA 1311/6010B (Axial)	20	80–120	0.050 mg/L
Silver	Leachate	EPA 1311/6010B (Axial)	20	80–120	0.10 mg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.4. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
MTBE	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg
Benzene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg
Toluene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg
Chlorobenzene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg
Ethylbenzene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg
Ortho-xylene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg
Meta, para-xylene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg
1,3-Dichlorobenzene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg

¹ Precision and accuracy criteria are the same for water and solid matrices.

Table 3.4. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
1,4-Dichlorobenzene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg
1,2-Dichlorobenzene	Water	EPA 602, 8021B	20	85-115	0.50 ug/L
	Solid	EPA 8021B			0.50 ug/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.5. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR TOTAL PETROLEUM HYDROCARBONS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
TPH's - Gasoline	Water	SW 8015 mod., CAL LUFT	20	85-115	0.050 mg/L
	Solid	SW 8015 mod., CAL LUFT	20	84-115	0.10 mg/kg
TPH's - Diesel	Water	SW 8015 mod. CAL LUFT	20	76-137	1.0 mg/L 0.10 mg/L
	Solid	SW 8015 mod., CAL LUFT	20	74-150	5.0 mg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.6. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
5 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Dichlorodifluoromethane	Water	EPA 8260B			10 ug/L
	Solid	EPA 8260B			10 ug/kg
Chloromethane	Water	EPA 8260B			10 ug/L
	Solid	EPA 8260B			10 ug/kg
Vinyl chloride	Water	EPA 8260B			10 ug/L
	Solid	EPA 8260B			10 ug/kg
Bromomethane	Water	EPA 8260B			10 ug/L
	Solid	EPA 8260B			10 ug/kg
Chloroethane	Water	EPA 8260B			10 ug/L
	Solid	EPA 8260B			10 ug/kg
Trichlorofluoromethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,1-Dichloroethene	Water	EPA 8260B	15	73 - 127	5.0 ug/L
	Solid	EPA 8260B	15	59 - 136	5.0 ug/kg
Trichlorotrifluoroethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Acetone	Water	EPA 8260B			20 ug/L

Table 3.6. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
5 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
	Solid	EPA 8260B			20 ug/kg
Iodomethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Carbon Disulfide	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Methylene chloride	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
trans-1,2-Dichloroethene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,1-Dichloroethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Methyl-t-butyl-ether	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Vinyl acetate	Water	EPA 8260B			20 ug/L
	Solid	EPA 8260B			20 ug/kg
cis-1,2-Dichloroethene (MEK)	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
2-Butanone	Water	EPA 8260B			20 ug/L

Table 3.6. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
5 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
	Solid	EPA 8260B			20 ug/kg
Bromochloromethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Chloroform	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,1,1-Trichloroethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
2,2-Dichloropropane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Carbon tetrachloride	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,1-Dichloropropene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,2-Dichloroethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Benzene	Water	EPA 8260B	15	84 - 119	5.0 ug/L
	Solid	EPA 8260B	15	76 - 123	5.0 ug/kg
Trichloroethene	Water	EPA 8260B	15	85 - 121	5.0 ug/L

Table 3.6. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
5 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
1,2-Dichloropropane	Solid	EPA 8260B	15	74 - 127	5.0 ug/kg
	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Dibromomethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Bromodichloromethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
2-Chloroethyl vinyl ether	Water	EPA 8260B			10 ug/L
	Solid	EPA 8260B			10 ug/kg
cis-1,3-Dichloropropene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
4-Methyl-2-Pentanone (MIBK)	Water	EPA 8260B			20 ug/L
	Solid	EPA 8260B			20 ug/kg
Toluene	Water	EPA 8260B	15	83 - 123	5.0 ug/L
	Solid	EPA 8260B	15	75 - 124	5.0 ug/kg
trans-1,3-Dichloropropene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,1,2-Trichloroethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg

Table 3.6. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
5 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
2-Hexanone	Water	EPA 8260B			20 ug/L
	Solid	EPA 8260B			20 ug/kg
Tetrachloroethene (Perchloroethylene)	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,3-Dichloropropane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Dibromochloromethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,2-Dibromoethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Chlorobenzene	Water	EPA 8260B	15	85 - 119	5.0 ug/L
	Solid	EPA 8260B	15	75 - 124	5.0 ug/kg
1,1,1,2-Tetrachloroethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Ethylbenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
m,p-Xylene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg

Table 3.6. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
5 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
o-Xylene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Styrene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Bromoform	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Isopropylbenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,2,3-Trichloropropane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,1,2,2-Tetrachloroethane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Bromobenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
n-Propylbenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
2-Chlorotoluene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg

Table 3.6. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
5 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
1,3,5-Trimethylbenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
4-Chlorotoluene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
tert-Butylbenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,2,4-Trimethylbenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
sec-Butylbenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,3-Dichlorobenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
p-Isopropyltoluene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,4-Dichlorobenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
n-Butylbenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg

Table 3.6. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
5 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
1,2-Dichlorobenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,2-Dibromo-3-chloropropane	Water	EPA 8260B			10 ug/L
	Solid	EPA 8260B			10 ug/kg
1,2,4-Trichlorobenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Hexachlorobutadiene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Naphthalene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1,2,3-Trichlorobenzene	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
1-Chlorohexane	Water	EPA 8260B			5.0 ug/L
	Solid	EPA 8260B			5.0 ug/kg
Acrolein	Water	EPA 8260B			50 ug/L
	Solid	EPA 8260B			50 ug/kg
Acrylonitrile	Water	EPA 8260B			50 ug/L
	Solid	EPA 8260B			50 ug/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.7. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
25 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit (25 mL purge volume)
Dichlorodifluoromethane	Water	EPA 8260B			1.0 ug/L
Chloromethane	Water	EPA 8260B			1.0 ug/L
Vinyl chloride	Water	EPA 8260B			1.0 ug/L
Bromomethane	Water	EPA 8260B			1.0 ug/L
Chloroethane	Water	EPA 8260B			1.0 ug/L
Trichlorofluoromethane	Water	EPA 8260B			1.0 ug/L
1,1-Dichloroethene	Water	EPA 8260B	15	73 - 127	1.0 ug/L
Trichlorotrifluoroethane	Water	EPA 8260B			1.0 ug/L
Acetone	Water	EPA 8260B			10 ug/L
Iodomethane	Water	EPA 8260B			1.0 ug/L
Carbon Disulfide	Water	EPA 8260B			1.0 ug/L
Methylene chloride	Water	EPA 8260B			1.0 ug/L

Table 3.7. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
25 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit (25 mL purge volume)
trans-1,2-Dichloroethene	Water	EPA 8260B			1.0 ug/L
1,1-Dichloroethane	Water	EPA 8260B			1.0 ug/L
Methyl-t-butyl-ether	Water	EPA 8260B			1.0 ug/L
Vinyl acetate	Water	EPA 8260B			10 ug/L
cis-1,2-Dichloroethene	Water	EPA 8260B			1.0 ug/L
2-Butanone	Water	EPA 8260B			10 ug/L
Bromochloromethane	Water	EPA 8260B			1.0 ug/L
Chloroform	Water	EPA 8260B			1.0 ug/L
1,1,1-Trichloroethane	Water	EPA 8260B			1.0 ug/L
2,2-Dichloropropane	Water	EPA 8260B			1.0 ug/L
Carbon tetrachloride	Water	EPA 8260B			1.0 ug/L
1,1-Dichloropropene	Water	EPA 8260B			1.0 ug/L
1,2-Dichloroethane	Water	EPA 8260B			1.0 ug/L

Table 3.7. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
25 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit (25 mL purge volume)
Benzene	Water	EPA 8260B	15	84 - 119	1.0 ug/L
Trichloroethene	Water	EPA 8260B	15	85 - 121	1.0 ug/L
1,2-Dichloropropane	Water	EPA 8260B			1.0 ug/L
Dibromomethane	Water	EPA 8260B			1.0 ug/L
Bromodichloromethane	Water	EPA 8260B			1.0 ug/L
2-Chloroethyl vinyl ether	Water	EPA 8260B			1.0 ug/L
cis-1,3-Dichloropropene	Water	EPA 8260B			1.0 ug/L
4-Methyl-2-Pentanone	Water	EPA 8260B			10 ug/L
Toluene	Water	EPA 8260B	15	83 - 123	1.0 ug/L
trans-1,3-	Water	EPA 8260B			1.0 ug/L
1,1,2-Trichloroethane	Water	EPA 8260B			1.0 ug/L
2-Hexanone	Water	EPA 8260B			10 ug/L

Table 3.7. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
25 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit (25 mL purge volume)
Tetrachloroethene	Water	EPA 8260B			1.0 ug/L
1,3-Dichloropropane	Water	EPA 8260B			1.0 ug/L
Dibromochloromethane	Water	EPA 8260B			1.0 ug/L
1,2-Dibromoethane	Water	EPA 8260B			1.0 ug/L
Chlorobenzene	Water	EPA 8260B	15	85 - 119	1.0 ug/L
1,1,1,2-	Water	EPA 8260B			1.0 ug/L
Ethylbenzene	Water	EPA 8260B			1.0 ug/L
m,p-Xylene	Water	EPA 8260B			1.0 ug/L
o-Xylene	Water	EPA 8260B			1.0 ug/L
Styrene	Water	EPA 8260B			1.0 ug/L
Bromoform	Water	EPA 8260B			1.0 ug/L
Isopropylbenzene	Water	EPA 8260B			1.0 ug/L
1,2,3-Trichloropropane	Water	EPA 8260B			1.0 ug/L

Table 3.7. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
25 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit (25 mL purge volume)
1,1,2,2-	Water	EPA 8260B			1.0 ug/L
Bromobenzene	Water	EPA 8260B			1.0 ug/L
n-Propylbenzene	Water	EPA 8260B			1.0 ug/L
2-Chlorotoluene	Water	EPA 8260B			1.0 ug/L
1,3,5-Trimethylbenzene	Water	EPA 8260B			1.0 ug/L
4-Chlorotoluene	Water	EPA 8260B			1.0 ug/L
tert-Butylbenzene	Water	EPA 8260B			1.0 ug/L
1,2,4-Trimethylbenzene	Water	EPA 8260B			1.0 ug/L
sec-Butylbenzene	Water	EPA 8260B			1.0 ug/L
1,3-Dichlorobenzene	Water	EPA 8260B			1.0 ug/L
p-Isopropyltoluene	Water	EPA 8260B			1.0 ug/L
1,4-Dichlorobenzene	Water	EPA 8260B			1.0 ug/L
n-Butylbenzene	Water	EPA 8260B			1.0 ug/L

Table 3.7. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)
25 mL PURGE VOLUME FOR WATER MATRIX

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit (25 mL purge volume)
1,2-Dichlorobenzene	Water	EPA 8260B			1.0 ug/L
1,2-Dibromo-3-chloropropane	Water	EPA 8260B			2.0 ug/L
1,2,4-Trichlorobenzene	Water	EPA 8260B			1.0 ug/L
Hexachlorobutadiene	Water	EPA 8260B			1.0 ug/L
Naphthalene	Water	EPA 8260B			1.0 ug/L
1,2,3-Trichlorobenzene	Water	EPA 8260B			1.0 ug/L
1-Chlorohexane	Water	EPA 8260B			1.0 ug/L
Acrolein	Water	EPA 8260B			50 ug/L
Acrylonitrile	Water	EPA 8260B			50 ug/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.8. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR **TCLP** GC/MS VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Vinyl chloride	Leachate	EPA 1311/8260B	20		0.050 mg/L
1,1-Dichloroethene	Leachate	EPA 1311/8260B	20	73 - 127	0.025 mg/L
2-Butanone	Leachate	EPA 1311/8260B	20		0.10 mg/L
Chloroform	Leachate	EPA 1311/8260B	20		0.025 mg/L
Carbon tetrachloride	Leachate	EPA 1311/8260B	20		0.025 mg/L
1,2-Dichloroethane	Leachate	EPA 1311/8260B	20		0.025 mg/L
Benzene	Leachate	EPA 1311/8260B	20	84 - 119	0.025 mg/L
Tetrachloroethene	Leachate	EPA 1311/8260B	20	76 - 123	0.025 mg/L
Trichloroethene	Leachate	EPA 1311/8260B	20	85 - 121	0.025 mg/L
Chlorobenzene	Leachate	EPA 1311/8260B	20	85 - 119	0.025 mg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.9. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS (METHOD 624) -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy ¹ (% Recovery)	Reporting Limit
Chloromethane	Water	EPA 624	20	D-273	10 µg/L
Vinyl chloride	Water	EPA 624	20	D-251	10 µg/L
Bromomethane	Water	EPA 624	20	D-242	10 µg/L
Chloroethane	Water	EPA 624	20	14-230	10 µg/L
Trichlorofluoromethane	Water	EPA 624	20	17-181	5.0 µg/L
1,1-Dichloroethene	Water	EPA 624	20	D-234	5.0 µg/L
Methylene chloride	Water	EPA 624	20	D-221	5.0 µg/L
trans-1,2-Dichloroethene	Water	EPA 624	20	54-156	5.0 µg/L
1,1-Dichloroethane	Water	EPA 624	20	59-155	5.0 µg/L
Chloroform	Water	EPA 624	20	51-138	5.0 µg/L
1,1,1-Trichloroethane	Water	EPA 624	20	52-162	5.0 µg/L
Carbon tetrachloride	Water	EPA 624	20	70-140	5.0 µg/L
1,2-Dichloroethane	Water	EPA 624	20	49-155	5.0 µg/L
Benzene	Water	EPA 624	20	37-151	5.0 µg/L
Trichloroethene	Water	EPA 624	20	71-157	5.0 µg/L
1,2-Dichloropropane	Water	EPA 624	20	D-210	5.0 µg/L
Bromodichloromethane	Water	EPA 624	20	35-155	5.0 µg/L
2-Chloroethyl vinyl ether	Water	EPA 624	20	D-305	10 µg/L
cis-1,3-Dichloropropene	Water	EPA 624	20	D-227	5.0 µg/L
Toluene	Water	EPA 624	20	47-150	5.0 µg/L
trans-1,3-Dichloropropene	Water	EPA 624	20	17-183	5.0 µg/L
1,1,2-Trichloroethane	Water	EPA 624	20	52-150	5.0 µg/L
Tetrachloroethene	Water	EPA 624	20	64-148	5.0 µg/L
Dibromochloromethane	Water	EPA 624	20	53-149	5.0 µg/L

¹ Accuracy Ranges Source: 40 CFR Part 136, Appendix A, Method 624, Table 5.

Table 3.9. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS (METHOD 624) -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy ¹ (% Recovery)	Reporting Limit
Chlorobenzene	Water	EPA 624	20	37-160	5.0 µg/L
Ethylbenzene	Water	EPA 624	20	37-162	5.0 µg/L
Bromoform	Water	EPA 624	20	45-169	5.0 µg/L
1,1,2,2-Tetrachloroethane	Water	EPA 624	20	46-157	5.0 µg/L
1,3-Dichlorobenzene	Water	EPA 624	20	59-156	5.0 µg/L
1,4-Dichlorobenzene	Water	EPA 624	20	18-190	5.0 µg/L
1,2-Dichlorobenzene	Water	EPA 624	20	18-190	5.0 µg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.10. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS (METHOD 524.2) -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Dichlorodifluoromethane	Water	EPA 524.2	20	70-130	0.50 µg/L
Chloromethane	Water	EPA 524.2	20	70-130	0.50 µg/L
Vinyl Chloride	Water	EPA 524.2	20	70-130	0.50 µg/L
Bromomethane	Water	EPA 524.2	20	70-130	0.50 µg/L
Chloroethane	Water	EPA 524.2	20	70-130	0.50 µg/L
Trichlorofluoromethane	Water	EPA 524.2	20	70-130	0.50 µg/L
1,1-Dichloroethene	Water	EPA 524.2	20	70-130	0.50 µg/L
Methylene Chloride	Water	EPA 524.2	20	70-130	0.50 µg/L
Trans-1,2-dichloroethene	Water	EPA 524.2	20	70-130	0.50 µg/L
1,1-Dichloroethane	Water	EPA 524.2	20	70-130	0.50 µg/L
Cis-1,2-dichloroethene	Water	EPA 524.2	20	70-130	0.50 µg/L
Bromochloromethane	Water	EPA 524.2	20	70-130	0.50 µg/L
Chloroform	Water	EPA 524.2	20	70-130	0.50 µg/L
1,1,1-Trichloroethane	Water	EPA 524.2	20	70-130	0.50 µg/L
2,2-Dichloropropane	Water	EPA 524.2	20	70-130	0.50 µg/L
Carbon Tetrachloride	Water	EPA 524.2	20	70-130	0.50 µg/L
1,1-Dichloropropene	Water	EPA 524.2	20	70-130	0.50 µg/L
1,2-Dichloroethane	Water	EPA 524.2	20	70-130	
Benzene	Water	EPA 524.2	20	70-130	0.50 µg/L
Trichloroethene	Water	EPA 524.2	20	70-130	0.50 µg/L
1,2-Dichloropropane	Water	EPA 524.2	20	70-130	0.50 µg/L
Dibromomethane	Water	EPA 524.2	20	70-130	0.50 µg/L
Bromodichloromethane	Water	EPA 524.2	20	70-130	0.50 µg/L
Cis-1,3-dichloropropene	Water	EPA 524.2	20	70-130	0.50 µg/L
Toluene	Water	EPA 524.2	20	70-130	0.50 µg/L
Trans-1,3-dichloropropene	Water	EPA 524.2	20	70-130	0.50 µg/L

Table 3.10. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS (METHOD 524.2) -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
1,1,2-Trichloroethane	Water	EPA 524.2	20	70-130	0.5 µg/L
Tetrachloroethene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,3-Dichloropropane	Water	EPA 524.2	20	70-130	0.5 µg/L
Dibromochloromethane	Water	EPA 524.2	20	70-130	0.5 µg/L
1,2-Dibromoethane (EDB)	Water	EPA 524.2	20	70-130	0.5 µg/L
Chlorobenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,1,1,2-Tetrachloroethane	Water	EPA 524.2	20	70-130	0.5 µg/L
Ethylbenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
M+P-xylene	Water	EPA 524.2	20	70-130	0.5 µg/L
O-xylene	Water	EPA 524.2	20	70-130	0.5 µg/L
Styrene	Water	EPA 524.2	20	70-130	0.5 µg/L
Bromoform	Water	EPA 524.2	20	70-130	0.5 µg/L
Isopropylbenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,2,3-Trichloropropane	Water	EPA 524.2	20	70-130	0.5 µg/L
1,1,2,2-Tetrachloroethane	Water	EPA 524.2	20	70-130	0.5 µg/L
Bromobenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
N-propylbenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
2-Chlorotoluene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,3,5-Trimethylbenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
4-Chlorotoluene	Water	EPA 524.2	20	70-130	0.5 µg/L
Tert-butylbenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,2,4-Trimethylbenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
Sec-butylbenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,3-Dichlorobenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
P-Isopropyltoluene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,4-Dichlorobenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
N-Butylbenzene	Water	EPA 524.2	20	70-130	0.5 µg/L

Table 3.10. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS VOLATILE ORGANICS (METHOD 524.2) -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
1,2-Dichlorobenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,2-Dibromo-3-chloropropane (DBCP)	Water	EPA 524.2	20	70-130	5.0 µg/L
1,2,4-Trichlorobenzene	Water	EPA 524.2	20	70-130	0.5 µg/L
Hexachlorobutadiene	Water	EPA 524.2	20	70-130	0.5 µg/L
Naphthalene	Water	EPA 524.2	20	70-130	0.5 µg/L
1,2,3-Trichlorobenzene	Water	EPA 524.2	20	70-130	0.5 µg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.11. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Pyridine	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
n-Nitrosodimethylamine	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Aniline	Water	EPA 8270C			25 µg/L
	Solid	EPA 8270C			830 µg/kg
Phenol	Water	EPA 8270C	42	25 - 105	10 µg/L
	Solid	EPA 8270C	35	25 - 112	330 µg/kg
bis (2-Chloroethyl) ether	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2-Chloropheno	Water	EPA 8270C	40	23 - 106	10 µg/L
	Solid	EPA 8270C	50	28 - 110	330 µg/kg
1,3-Dichlorobenzene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
1,4-Dichlorobenzene	Water	EPA 8270C	28	13 - 113	10 µg/L
	Solid	EPA 8270C	27	27 - 104	330 µg/kg

¹ Source: EPA Contract Laboratory Program, Statement of Work, OLM03.1.

Table 3.11. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
1,2-Dichlorobenzene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Benzyl alcohol	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Bis(2-chloroisopropyl) ether (2,2'-Oxybis[1-chloropropane])	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2-Methylphenol	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
N-Nitroso-di-n-propylamine	Water	EPA 8270C	38	25 - 113	10 µg/L
	Solid	EPA 8270C	38	24 - 116	330 µg/kg
4-Methylphenol	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Hexachloroethane	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Nitrobenzene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg

Table 3.11. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Isophorone	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2-Nitrophenol	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2,4-Dimethylphenol	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
bis (2-Chloroethoxy) methane	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2,4-Dichlorophenol	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Benzoic acid	Water	EPA 8270C			50 µg/L
	Solid	EPA 8270C			1700 µg/kg
1,2,4-Trichlorobenzene	Water	EPA 8270C	28	22 - 106	10 µg/L
	Solid	EPA 8270C	23	33 - 105	330 µg/kg
Naphthalene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
4-Chloroaniline	Water	EPA 8270C			25 µg/L
	Solid	EPA 8270C			830 µg/kg

Table 3.11. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Hexachlorobutadiene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
4-Chloro-3-methylphenol	Water	EPA 8270C	42	34 - 104	10 µg/L
	Solid	EPA 8270C	33	31 - 108	330 µg/kg
2-Methylnaphthalene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Hexachlorocyclopentadiene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2,4,6-Trichlorophenol	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2,4,5-Trichlorophenol	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2-Chloronaphthalene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2-Nitroaniline	Water	EPA 8270C			50 µg/L
	Solid	EPA 8270C			1700 µg/kg
Dimethyl phthalate	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg

Table 3.11. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
2,6-Dinitrotoluene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Acenaphthylene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
3-Nitroaniline	Water	EPA 8270C			50 µg/L
	Solid	EPA 8270C			1700 µg/kg
Acenaphthene	Water	EPA 8270C	31	24 - 110	10 µg/L
	Solid	EPA 8270C	19	33 - 102	330 µg/kg
2,4-Dinitrophenol	Water	EPA 8270C			50 µg/L
	Solid	EPA 8270C			1700 µg/kg
4-Nitrophenol	Water	EPA 8270C	50	18 - 114	50 µg/L
	Solid	EPA 8270C	50	21 - 133	1700 µg/kg
Dibenzofuran ²	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
2,4-Dinitrotoluene	Water	EPA 8270C	38	28 - 109	10 µg/L
	Solid	EPA 8270C	47	23 - 121	330 µg/kg
Diethyl phthalate	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg

Table 3.11. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Fluorene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
4-Chlorophenyl phenyl ether	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
4-Nitroaniline	Water	EPA 8270C			50 µg/L
	Solid	EPA 8270C			1700 µg/kg
Azobenzene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
4,6-Dinitro-2-methylphenol	Water	EPA 8270C			50 µg/L
	Solid	EPA 8270C			1700 µg/kg
N-Nitrosodiphenylamine	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
4-Bromophenyl phenyl ether	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Hexachlorobenzene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Pentachlorophenol	Water	EPA 8270C	50	23 - 112	50 µg/L
	Solid	EPA 8270C	47	11 - 120	1700 µg/kg

Table 3.11. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Phenanthrene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Anthracene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Carbazole	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Di-n-butyl phthalate	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Fluoranthene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Benzidine	Water	EPA 8270C			50 µg/L
	Solid	EPA 8270C			1700 µg/kg
Pyrene	Water	EPA 8270C	31	23 - 119	10 µg/L
	Solid	EPA 8270C	36	29 - 114	330 µg/kg
Butyl benzyl phthalate	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Benzo(a)anthracene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg

Table 3.11. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision ¹ (RPD - %)	Accuracy (% Recovery)	Reporting Limit
3,3'-Dichlorobenzidine	Water	EPA 8270C			50 µg/L
	Solid	EPA 8270C			1700 µg/kg
Chrysene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Bis(2-ethylhexyl) phthalate	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Di-n-octyl phthalate	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Benzo(b&k)fluoranthene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Benzo(a)pyrene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Indeno(1,2,3-cd)pyrene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Dibenz(a,h)anthracene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg
Benzo(g,h,i)perylene	Water	EPA 8270C			10 µg/L
	Solid	EPA 8270C			330 µg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.12. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS (METHOD 625) -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy ¹ (% Recovery)	Reporting Limit
Phenol	Water	EPA 625	40	5-112	10 µg/L
bis (2-Chloroethyl) ether	Water	EPA 625	40	12-158	10 µg/L
2-Chlorophenol	Water	EPA 625	40	23-134	10 µg/L
1,3-Dichlorobenzene	Water	EPA 625	40	D-172	10 µg/L
1,4-Dichlorobenzene	Water	EPA 625	40	20-124	10 µg/L
1,2-Dichlorobenzene	Water	EPA 625	40	32-129	10 µg/L
bis (2-Chloroisopropyl) ether	Water	EPA 625	40	36-166	10 µg/L
N-Nitroso-di-n-propylamine	Water	EPA 625	40	D-230	10 µg/L
Hexachloroethane	Water	EPA 625	40	40-113	10 µg/L
Nitrobenzene	Water	EPA 625	40	35-180	10 µg/L
Isophorone	Water	EPA 625	40	21-196	10 µg/L
2-Nitrophenol	Water	EPA 625	40	29-182	10 µg/L
2,4-Dimethylphenol	Water	EPA 625	40	32-119	10 µg/L
bis (2-Chloroethoxy) methane	Water	EPA 625	40	33-184	10 µg/L
2,4-Dichlorophenol	Water	EPA 625	40	39-135	10 µg/L
1,2,4-Trichlorobenzene	Water	EPA 625	40	44-142	10 µg/L
Naphthalene	Water	EPA 625	40	21-133	10 µg/L
Hexachlorobutadiene	Water	EPA 625	40	24-116	10 µg/L
4-Chloro-3-methylphenol	Water	EPA 625	40	22-147	10 µg/L
2,4,6-Trichlorophenol	Water	EPA 625	40	37-144	10 µg/L

¹ Accuracy Ranges Source: 40 CFR Part 136, Appendix A, Method 625, Table 6.

Table 3.12. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS (METHOD 625) -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy ¹ (% Recovery)	Reporting Limit
2-Chloronaphthalene	Water	EPA 625	40	60-118	10 µg/L
Dimethyl phthalate	Water	EPA 625	40	D-112	10 µg/L
2,6-Dinitrotoluene	Water	EPA 625	40	50-158	10 µg/L
Acenaphthylene	Water	EPA 625	40	33-145	10 µg/L
Acenaphthene	Water	EPA 625	40	47-145	10 µg/L
2,4-Dinitrophenol	Water	EPA 625	40	D-191	50 µg/L
4-Nitrophenol	Water	EPA 625	40	D-132	50 µg/L
2,4-Dinitrotoluene	Water	EPA 625	40	39-139	10 µg/L
Diethyl phthalate	Water	EPA 625	40	D-114	10 µg/L
Fluorene	Water	EPA 625	40	59-121	10 µg/L
4-Chlorophenyl phenyl ether	Water	EPA 625	40	25-158	10 µg/L
4,6-Dinitro-2- methylphenol	Water	EPA 625	40	D-181	50 µg/L
4-Bromophenyl phenyl ether	Water	EPA 625	40	53-127	10 µg/L
Hexachlorobenzene	Water	EPA 625	40	D-152	10 µg/L
Pentachlorophenol	Water	EPA 625	40	14-176	50 µg/L
Phenanthrene	Water	EPA 625	40	54-120	10 µg/L
Anthracene	Water	EPA 625	40	27-133	10 µg/L
Di-n-butyl phthalate	Water	EPA 625	40	1-118	10 µg/L
Fluoranthene	Water	EPA 625	40	26-137	10 µg/L
Pyrene	Water	EPA 625	40	52-115	10 µg/L
Butyl benzyl phthalate	Water	EPA 625	40	D-152	10 µg/L
Benzo(a)anthracene	Water	EPA 625	40	33-143	10 µg/L
3,3'-Dichlorobenzidine	Water	EPA 625	40	D-262	50 µg/L
Chrysene	Water	EPA 625	40	17-169	10 µg/L

Table 3.12. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR GC/MS SEMI-VOLATILE ORGANICS (METHOD 625) -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy ¹ (% Recovery)	Reporting Limit
Bis(2-ethylhexyl) phthalate	Water	EPA 625	40	8-158	10 µg/L
Di-n-octyl phthalate	Water	EPA 625	40	4-146	10 µg/L
Benzo(b&k)fluoranthene	Water	EPA 625	40	24-159	10 µg/L
Benzo(a)pyrene	Water	EPA 625	40	17-163	10 µg/L
Indeno(1,2,3-cd)pyrene	Water	EPA 625	40	D-171	10 µg/L
Dibenz(a,h)anthracene	Water	EPA 625	40	D-227	10 µg/L
Benzo(g,h,i)perylene	Water	EPA 625	40	D-219	10 µg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.13. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR **TCLP** GC/MS SEMI-VOLATILE ORGANICS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
1,4-dichlorobenzene	Leachate	EPA 1311/8270C			0.10 mg/L
2-Methylphenol (o-cresol)	Leachate	EPA 1311/8270C			0.10 mg/L
3-Methylphenol (m-cresol)	Leachate	EPA 1311/8270C			0.10 mg/L
4-Methylphenol (p-cresol)	Leachate	EPA 1311/8270C			0.10 mg/L
Pyridine	Leachate	EPA 1311/8270C			0.10 mg/L
Hexachloroethane	Leachate	EPA 1311/8270C			0.10 mg/L
Nitrobenzene	Leachate	EPA 1311/8270C			0.10 mg/L
Hexachlorobutadiene	Leachate	EPA 1311/8270C			0.10 mg/L
2,4,6-Trichlorophenol	Leachate	EPA 1311/8270C			0.10 mg/L
2,4,5-Trichlorophenol	Leachate	EPA 1311/8270C			0.50 mg/L
2,4-Dinitrotoluene	Leachate	EPA 1311/8270C	38	28 - 109	0.10 mg/L
Hexachlorobenzene	Leachate	EPA 1311/8270C			0.10 mg/L
Pentachlorophenol	Leachate	EPA 1311/8270C	50	23 - 112	0.50 mg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.14. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR PESTICIDES/PCB'S -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
alpha - BHC	Water	EPA 608, 8081A			0.050 µg/L
	Solid	EPA 8081A			1.7 µg/kg
gamma - BHC (Lindane)	Water	EPA 608, 8081A	20	77 - 120	0.050 µg/L
	Solid	EPA 8081A	20	674 - 127	1.7 µg/kg
Heptachlor	Water	EPA 608, 8081A	20	66 - 135	0.050 µg/L
	Solid	EPA 8081A	20	71 - 140	1.7 µg/kg
Aldrin	Water	EPA 608, 8081A	20	74 - 122	0.050 µg/L
	Solid	EPA 8081A	20	70 - 127	1.7 µg/kg
beta - BHC	Water	EPA 608, 8081A			0.050 µg/L
	Solid	EPA 8081A			1.7 µg/kg
delta - BHC	Water	EPA 608, 8081A			0.050µg/L
	Solid	EPA 8081A			1.7 µg/kg
Heptachlor Epoxide	Water	EPA 608, 8081A			0.050 µg/L
	Solid	EPA 8081A			1.7 µg/kg
Endosulfan I	Water	EPA 608, 8081A			0.050 µg/L
	Solid	EPA 8081A			1.7 µg/kg
Gamma Chlordane	Water	EPA 608, 8081A			0.050 µg/L
	Solid	EPA 8081A			1.7 µg/kg

Table 3.14. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR PESTICIDES/PCB'S -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Alpha Chlordane	Water	EPA 608, 8081A			0.050 µg/L
	Solid	EPA 8081A			1.7 µg/kg
4, 4' - DDE	Water	EPA 608, 8081A			0.10 µg/L
	Solid	EPA 8081A			3.3 µg/kg
Dieldrin	Water	EPA 608, 8081A	20	79 - 137	0.10 µg/L
	Solid	EPA 8081A	20	80 - 134	3.3 µg/kg
Endrin	Water	EPA 608, 8081A	20	75 - 136	0.10 µg/L
	Solid	EPA 8081A	20	76 - 136	3.3 µg/kg
4, 4' - DDD	Water	EPA 608, 8081A			0.10 µg/L
	Solid	EPA 8081A			3.3 µg/kg
Endosulfan II	Water	EPA 608, 8081A			0.10 µg/L
	Solid	EPA 8081A			3.3 µg/kg
4, 4' - DDT	Water	EPA 608, 8081A	20	83 - 127	0.10 µg/L
	Solid	EPA 8081A	20	73 - 136	3.3 µg/kg
Endrin Aldehyde	Water	EPA 608, 8081A			0.10 µg/L
	Solid	EPA 8081A			3.3 µg/kg
Methoxychlor	Water	EPA 608, 8081A			0.50 µg/L
	Solid	EPA 8081A			17 µg/kg

Table 3.14. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR PESTICIDES/PCB'S -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Endosulfan Sulfate	Water	EPA 608, 8081A			0.10 µg/L
	Solid	EPA 8081A			3.3 µg/kg
Endrin Ketone	Water	EPA 608, 8081A			0.10 µg/L
	Solid	EPA 8081A			3.3 µg/kg
Toxaphene	Water	EPA 608, 8081A			5.0 µg/L
	Solid	EPA 8081A			170 µg/kg
Aroclor 1016	Water	EPA 608, 8082			1.0 µg/L
	Solid	EPA 8082			33 µg/kg
Aroclor 1221	Water	EPA 608, 8082			2.0 µg/L
	Solid	EPA 8082			67 µg/kg
Aroclor 1232	Water	EPA 608, 8081A			1.0 µg/L
	Solid	EPA 8081A			33 µg/kg
Aroclor 1242	Water	EPA 608, 8082			1.0 µg/L
	Solid	EPA 8082			33 µg/kg
Aroclor 1248	Water	EPA 608, 8082			1.0 µg/L
	Solid	EPA 8082			33 µg/kg
Aroclor 1254	Water	EPA 608, 8082			1.0 µg/L
	Solid	EPA 8082			33 µg/kg

Table 3.14. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR PESTICIDES/PCB'S -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Aroclor 1260 ¹	Water	EPA 608, 8082	20	73 - 116	1.0 µg/L
	Solid	EPA 8082	20	70 - 118	33 µg/kg
Aroclor 1268	Water	EPA 608, 8082			1.0 µg/L
	Solid	EPA 8082			33 µg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

¹ Method Control Analyte when only PCBs are to be reported.

Table 3.15. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR **TCLP** PESTICIDES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
gamma – BHC (Lindane)	Leachate	EPA 1311/8031A	20	77 - 120	0.050 mg/L
Heptachlor	Leachate	EPA 1311/8081A	20	66 - 135	0.050 mg/L
Heptachlor Epoxide	Leachate	EPA 1311/8081A			0.050 mg/L
Technical Chlordane	Leachate	EPA 1311/8081A			1.0 mg/L
Endrin	Leachate	EPA 1311/8081A	20	75 - 136	0.10 mg/L
Methoxychlor	Leachate	EPA 1311/8081A			0.50 mg/L
Toxaphene	Leachate	EPA 1311/8081A			5.0 mg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.16. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR EDB & DBCP -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Ethylene dibromide	Water	EPA 504.1, EPA 8011	40	60 - 140	0.020 µg/L
1,2-Dibromo-3-chloropropane	Water	EPA 504.1, EPA 8011	40	60 - 140	0.020 µg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.17. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR CHLORINATED HERBICIDES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Dalapon	Water	EPA 8151A			4.0 µg/L
	Solid	EPA 8151A			130 µg/kg
Dicamba	Water	EPA 8151A			0.20 µg/L
	Solid	EPA 8151A			6.6 µg/kg
MCPP	Water	EPA 8151A			100 µg/L
	Solid	EPA 8151A			3300 µg/kg
MCPA	Water	EPA 8151A			100µg/L
	Solid	EPA 8151A			3300 µg/kg
Dichloroprop	Water	EPA 8151A			1.0 µg/L
	Solid	EPA 8151A			33 µg/kg
2,4-D ¹	Water	EPA 8151A	20	55-140	1.0 µg/L
	Solid	EPA 8151A	20	50-150	33 µg/kg
2,4,5-TP (Silvex)	Water	EPA 8151A	20	50-120	0.10 µg/L
	Solid	EPA 8151A	20	50-150	3.3 µg/kg
2,4,5-T ¹	Water	EPA 8151A	20	65-120	0.10 µg/L
	Solid	EPA 8151A	20	50-150	3.3 µg/kg

¹ Method Control Analyte.

Table 3.17. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR CHLORINATED HERBICIDES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
2,4-DB	Water	EPA 8151A			1.0 µg/L
	Solid	EPA 8151A			33 µg/kg
Dinoseb	Water	EPA 8151A			1.0 µg/L
	Solid	EPA 8151A			33 µg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.18. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR **TCLP** CHLORINATED HERBICIDES -- REPRESENTATIVE VALUES *(SUBJECT TO UPDATE AS NECESSARY)*

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
2,4-D	Leachate	EPA 1311/8151A	20	55-140	0.10 mg/L
2,4,5-TP (Silvex)	Leachate	EPA 1311/8151A	20	50-120	0.010 mg/L

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.19. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR POLYNUCLEAR AROMATIC HYDROCARBONS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Naphthalene	Water	EPA 8310			0.50 µg/L
	Solid	EPA 8310			15 µg/kg
Acenaphthylene	Water	EPA 8310	20	36 - 93	1.0 µg/L
	Solid	EPA 8310	20	27 - 90	30 µg/kg
1-Methylnaphthalene	Water	EPA 8310			1.0 ug/L
	Solid	EPA 8310			30 ug/kg
2-Methylnaphthalene	Water	EPA 8310			1.0 ug/L
	Solid	EPA 8310			30 ug/kg
Acenaphthene	Water	EPA 8310			1.0 µg/L
	Solid	EPA 8310			20 µg/kg
Fluorene	Water	EPA 8310			0.10 µg/L
	Solid	EPA 8310			3.0 µg/kg
Phenanthrene	Water	EPA 8310	20	45 - 107	0.050 µg/L
	Solid	EPA 8310	20	46 - 96	2.0 µg/kg
Anthracene	Water	EPA 8310			0.10 µg/L
	Solid	EPA 8310			2.0 µg/kg

Table 3.19. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR POLYNUCLEAR AROMATIC HYDROCARBONS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Fluoranthrene	Water	EPA 8310			0.10 µg/L
	Solid	EPA 8310			3.0 µg/kg
Pyrene	Water	EPA 8310	20	40 - 104	0.050 µg/L
	Solid	EPA 8310	20	43 - 96	2.0 µg/kg
Benzo(a)anthracene	Water	EPA 8310			0.050 µg/L
	Solid	EPA 8310			3.0 µg/kg
Chrysene	Water	EPA 8310			0.050 µg/L
	Solid	EPA 8310			2.0 µg/kg
Benzo(b)fluoranthrene	Water	EPA 8310			0.10 µg/L
	Solid	EPA 8310			3.0 µg/kg
Benzo(k)fluoranthrene	Water	EPA 8310	20	61 - 126	0.050 µg/L
	Solid	EPA 8310	20	66 - 115	2.0 µg/kg
Benzo(a)pyrene	Water	EPA 8310			0.10 µg/L
	Solid	EPA 8310			3.0 µg/kg
Dibenzo(a,h)anthracene	Water	EPA 8310	20	55 - 113	0.10 µg/L
	Solid	EPA 8310	20	20 - 133	3.0 µg/kg

Table 3.19. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR POLYNUCLEAR AROMATIC HYDROCARBONS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Benzo(g,h,i)perylene	Water	EPA 8310			0.10 µg/L
	Solid	EPA 8310			2.0 µg/kg
Indeno(1,2,3-c,d)pyrene	Water	EPA 8310			0.10 µg/L
	Solid	EPA 8310			2.0 µg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.20. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR NITROAROMATICS/EXPLOSIVES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	Water	EPA 8330	30	33 - 113	1.0 µg/L
	Solid	EPA 8330	30	61 - 137	2.2 mg/kg
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	Water	EPA 8330	30	35 - 113	0.84 µg/L
	Solid	EPA 8330	30	74 - 127	1.0 mg/kg
1,3,5-Trinitrobenzene (1,3,5-TNB)	Water	EPA 8330	30	27 - 104	0.26 µg/L
	Solid	EPA 8330	30	79 - 117	0.25 mg/kg
1,3-Dinitrobenzene (1,3-DNB)	Water	EPA 8330	30	35 - 106	0.25 µg/L
	Solid	EPA 8330	30	80 - 132	0.25 mg/kg
Tetryl (Methyl-2,4,6-trinitrophenylnitramine)	Water	EPA 8330	30		1.0 µg/L
	Solid	EPA 8330	30		0.65 mg/kg
Nitrobenzene (NB)	Water	EPA 8330	30	34 - 91	1.0 µg/L
	Solid	EPA 8330	30	78 - 129	0.26 mg/kg
2,4,6-Trinitrotoluene (2,4,6-TNT)	Water	EPA 8330	30	41 - 107	0.25 µg/L
	Solid	EPA 8330	30	82 - 126	0.25 mg/kg
4-Amino-2,6-DNT	Water	EPA 8330	30		0.25 µg/L
	Solid	EPA 8330	30		0.25 mg/kg
2-Amino-4,6-DNT	Water	EPA 8330	30	41 - 111	0.25 µg/L
	Solid	EPA 8330	30	81 - 125	0.25 mg/kg

Table 3.20. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR NITROAROMATICS/EXPLOSIVES -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
2,6-Dinitrotoluene (2,6-DNT)	Water	EPA 8330	30		0.25 µg/L
	Solid	EPA 8330	30		0.26 mg/kg
2,4-Dinitrotoluene (2,4-DNT)	Water	EPA 8330	30	38 - 100	0.25 µg/L
	Solid	EPA 8330	30	75 – 130	0.25 mg/kg
o-Nitrotoluene (2-NT)	Water	EPA 8330	30		1.0 µg/L
	Solid	EPA 8330	30		0.25 mg/kg
p-Nitrotoluene (4-NT)	Water	EPA 8330	30		1.0 µg/L
	Solid	EPA 8330	30		0.25 mg/kg
m-Nitrotoluene (3-NT)	Water	EPA 8330	30		1.0 µg/L
	Solid	EPA 8330	30		0.25 mg/kg
Nitroglycerin	Water	EPA 8330 mod			5.0 ug/L
	Solid	EPA 8330 mod			1.8 mg/kg
PETN	Water	EPA 8330 mod			5.0 ug/L
	Solid	EPA 8330 mod			1.8 mg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.21. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR NITROGUANIDINE -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Nitroguanidine	Water	PAI SOP 439	30		100 ug/L
	Solid	PAI SOP 439	30		6.0 mg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.22. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR ORGANONITROGEN/ORGANOPHOSPHOROUS PESTICIDES -- REPRESENTATIVE VALUES
(SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Coumaphos	Water	EPA 8141A			2.0 µg/L
	Solid	EPA 8141A			67 µg/ kg
Demeton O & S	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/ kg
Diazinon	Water	EPA 8141A	25	56-140	1.0 µg/L
	Solid	EPA 8141A	25	51-123	33 µg/ kg
Dichlorovos	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/ kg
Disulfoton	Water	EPA 8141A			4.0 µg/L
	Solid	EPA 8141A			130 µg/ kg
Ethoprop	Water	EPA 8141A	25	50-123	1.0 µg/L
	Solid	EPA 8141A	25	49-115	33 µg/ kg
Fensulfothion	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/ kg
Fenthion	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/ kg

Table 3.22. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR ORGANONITROGEN/ORGANOPHOSPHOROUS PESTICIDES -- REPRESENTATIVE VALUES
 (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Merphos A & B	Water	EPA 8141A			2.0 µg/L
	Solid	EPA 8141A			67 µg/ kg
Methyl Azinophos	Water	EPA 8141A			2.0 µg/L
	Solid	EPA 8141A			67 µg/ kg
Methyl Parathion	Water	EPA 8141A	25	54-115	1.0 µg/L
	Solid	EPA 8141A	25	62-112	33 µg/kg
Mevinphos	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/kg
Naled	Water	EPA 8141A			3.0 µg/L
	Solid	EPA 8141A			100 µg/kg
Phorate	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/kg
Ronnel	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/kg
Sulprofos	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/kg
Tetrachlorvinphos	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/kg

Table 3.22. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR ORGANONITROGEN/ORGANOPHOSPHOROUS PESTICIDES -- REPRESENTATIVE VALUES
 (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit
Tokuthion	Water	EPA 8141A			1.0 µg/L
	Solid	EPA 8141A			33 µg/kg
Trichloronate	Water	EPA 8141A	25	50-114	1.0 µg/L
	Solid	EPA 8141A	25	54-115	33 µg/kg

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.23. SUMMARY OF ACCURACY CRITERIA & REPORTING LIMITS FOR ALPHA SPECTROMETRY PARAMETERS
-- REPRESENTATIVE VALUES

Parameter	Matrix	Reference	Accuracy (% Recovery)	Reporting Limit (Default MDC ¹)
Isotopic Americium (241)	Water	SOP 776/778/780/714	79 – 118	0.10 pCi/L / isotope
	Solid	SOP 721/773/778/780/714	79 – 118	0.05 pCi/g / isotope
	Other	SOP 721/773/778/780/714	79 – 118	0.10 pCi / aliq. / isotope
Isotopic Curium (244)	Water	SOP 776/778/780/714	79 – 118	0.10 pCi/L / isotope
	Solid	SOP 721/773/778/780/714	79 – 118	0.05 pCi/g / isotope
	Other	SOP 721/773/778/780/714	79 – 118	0.10 pCi / aliq. / isotope
Isotopic Plutonium (238, 239, 240)	Water	SOP 776/777/778/714	82 – 118	0.10 pCi/L / isotope
	Solid	SOP 721/773/778/714	82 – 118	0.05 pCi/g / isotope
	Other	SOP 721/773/778/714	82 – 118	0.10 pCi / aliq. / isotope
Isotopic Uranium (234, 235, 238)	Water	SOP 776/778/714	82 – 122	0.20 pCi/L / isotope
	Solid	SOP 721/773/778/714	82 – 122	0.10 pCi/g / isotope
	Other	SOP 721/773/778/714	82 – 122	0.20 pCi / aliq. / isotope
Total Uranium (from Alpha Isotopic)	Water	ASTM D3972-mod; SOP 776/778/714	75 – 125	0.50 pCi/L
	Solid	ASTM D3972-mod SOP 721/773/778/714	75 – 125	0.25 pCi/g
	Other	ASTM D3972-mod SOP 721/773/778/714	75 – 125	0.50 pCi / aliq.
Polonium – 210	Water	EERF 00-03-mod; SOP 719/714	83 - 117	0.50 pCi/L / isotope
	Solid	EERF 00-03-mod; SOP 721/719/714	83 - 117	0.25 pCi/g / isotope

¹ MDC = Minimum Detectable Concentration.

Table 3.23. SUMMARY OF ACCURACY CRITERIA & REPORTING LIMITS FOR ALPHA SPECTROMETRY PARAMETERS
 -- REPRESENTATIVE VALUES

Parameter	Matrix	Reference	Accuracy (% Recovery)	Reporting Limit (Default MDC ¹)
	Other	EERF 00-03-mod; SOP 721/719/714	83 - 117	0.50 pCi / aliq. / isotope
Neptunium - 237	Water	SOP 765/714	75 - 125	0.20 pCi/L / isotope
	Solid	SOP 765/714	75 - 125	0.10 pCi/g / isotope
	Other	SOP 765/714	75 - 125	0.20 pCi / aliq. / isotope
Isotopic Thorium (228, 230, 232)	Water	SOP 776/777/782/714	88 - 127	0.20 pCi/L / isotope
	Solid	SOP 773/777/714	88 - 127	0.10 pCi/g / isotope
	Other	SOP 773/777/714	88 - 127	0.20 pCi / aliq. / isotope

Table 3.24. SUMMARY OF ACCURACY CRITERIA & REPORTING LIMITS FOR GAMMA SPECTROMETRY PARAMETERS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Accuracy (% Recovery)	Reporting Limit (Default MDC)
Gamma Emitting Materials	Water	EPA 901.1-mod; SOP 739/713	85 - 115	10 pCi/L (ref. Cs-137)
	Solid	EPA 901.1-mod; SOP 739/713	85 - 115	0.50 pCi/g (ref. Cs-137)
	Other	EPA 901.1-mod; SOP 739/713	85 - 115	10 pCi / aliq. (ref. Cs-137)
Radium – 226	Water	EPA 901.1(mod Kahn) SOP 706	75 - 125	1.0 pCi/L / isotope
Radium – 228	Water	EPA 901.1(mod Kahn) SOP 706	75 - 125	1.0 pCi/L / isotope
Radium-226/228 (Screening)	Solid	EPA 901.1-mod; SOP 739/713	85 - 115	0.50 pCi/L / isotope
Iron – 55	Water	RESL ¹ Fe-01-mod	70 - 130	50 pCi/L
	Solid	RESL Fe-01-mod	70 - 130	20 pCi/g
	Other	RESL Fe-01-mod	70 - 130	50 pCi / aliq.
Nickel – 59	Water	RESL Ni-01-mod; SOP 752/713	65 - 135	500 pCi/L
	Solid	RESL Ni-01-mod; SOP 752/713	65 - 135	100 pCi/g
	Other	RESL Ni-01-mod; SOP 752/713	65 - 135	500 pCi / aliq.

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

¹ RESL = Analytical Chemistry Branch Procedures Manual, Radiological and Environmental Sciences Laboratory, Department of Energy, Idaho Operations Office

Table 3.25. SUMMARY OF ACCURACY CRITERIA & REPORTING LIMITS FOR LIQUID SCINTILLATION COUNTING PARAMETERS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Accuracy (% Recovery)	Reporting Limit (Default MDC)
Carbon – 14	Water	EERF C-01; SOP 772/704	63 – 117	500 pCi/L
	Solid	EERF C-01-mod; SOP 772/704	63 – 117	100 pCi/g
	Other	EERF C-01-mod; SOP 772/704	63 – 117	100 pCi / aliquot
Tritium	Water	906.0; SOP 700/704	85 – 115	400 pCi/L
	Solid	EERF H-01-03; SOP 754/704	85 – 115	1.0 pCi/g
	Other	EERF H-01-03; SOP 754/704	75 – 125	4.0 pCi / aliquot
Nickel – 63	Water	RESL Ni-01; SOP 737/704	70 – 130	10 pCi/L
	Solid	RESL Ni-01; SOP 737/704	70 – 130	10 pCi/g
	Other	RESL Ni-01; SOP 737/704	70 – 130	10 pCi / aliquot
Plutonium – 241	Water	SOP 776/777/782/714/704	65 – 135	20 pCi/L
	Solid	SOP 721/773/777/780/704	65 – 135	20 pCi/g
	Other	SOP 721/773/777/780/704	65 – 135	20 pCi / aliquot
Other beta emitters	Other	SOP 704	65 – 135	Various

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.26. SUMMARY OF ACCURACY CRITERIA & REPORTING LIMITS FOR GAS FLOW PROPORTIONAL COUNTING PARAMETERS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Accuracy (% Recovery)	Reporting Limit (Default MDC)
Gross Alpha	Water	EPA 900.0/9010; SOP 702/724	70 – 130	3.0pCi/L
	Solid	EPA 900.0-mod/9010-mod; SOP 702/724	70 – 130	3.0 pCi/g
	Other	EPA 900.0-mod/9010-mod; SOP 702/724	70 – 130	3.0 pCi / aliquot
Gross Beta	Water	EPA 900.0/9010; SOP 702/724	70 – 130	4.0pCi/L
	Solid	EPA 900.0-mod/9010-mod; SOP 702/724	70 – 130	4.0 pCi/g
	Other	EPA 900.0-mod/9010-mod; SOP 702/724	70 – 130	4.0 pCi / aliquot
Gross Alpha Coprecipitate	Water	SM 7110C; SOP 702/724	70 – 130	1.0pCi/L
	Solid	SM7110C; SOP 702/724	70 – 130	1.0 pCi/g
	Other	SM 7110C; SOP 702/724	70 – 130	1.0 pCi / aliquot
Radium – 228	Water	EPA 904.0/9320; SOP 746/724	65 – 135	1.0 pCi /L / isotope
	Solid	EPA 904.0-mod/9320; SOP 746/724	65 – 135	20 pCi /g / isotope
Total Alpha Emitting Radium	Water	EPA 903.0/9315; SOP 712/724	75 – 125	1.0pCi/L
	Solid	EPA 903.0-mod/9315; SOP 712/724	75 – 125	20 pCi/g
	Other	EPA 903.0-mod/9315; SOP 712/724	75 – 125	1.0 pCi / aliquot
Iodine – 129	Water	EPA 902.0; SOP 753/724	65 – 135	10pCi/L
	Solid	EPA 902.0-mod; SOP 753/724	65 – 135	10 pCi/g
	Other	EPA 902.0-mod; SOP 753/724	65 – 135	10 pCi / aliquot
Lead – 210	Water	ASTM D5811-95-mod; SOP 726/724	70 – 130	1.0pCi/L
	Solid	ASTM D5811-95-mod; SOP 726/724	70 – 130	1.0 pCi/g
	Other	ASTM D5811-95-mod; SOP 726/724	70 – 130	1.0 pCi / aliquot
Strontium – 89/90	Water	ASTM D5811-95-mod; SOP 707/724	75 – 125	1.0pCi/L
	Solid	ASTM D5811-95-mod; SOP 707/724	75 – 125	0.50 pCi/g
	Other	ASTM D5811-95-mod; SOP 707/724	75 – 125	1.0 pCi / aliquot

Table 3.26. SUMMARY OF ACCURACY CRITERIA & REPORTING LIMITS FOR GAS FLOW PROPORTIONAL COUNTING PARAMETERS -- REPRESENTATIVE VALUES (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Accuracy (% Recovery)	Reporting Limit (Default MDC)
Technetium - 99	Water	DOE RP 550; SOP 755/724	65 – 135	10pCi/L
	Solid	DOE RP 550; SOP 756/724	65 – 135	3.0 pCi/g
	Other	DOE RP 550; SOP 756/724	65 – 135	3.0 pCi / aliquot

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.27. SUMMARY OF PRECISION AND ACCURACY CRITERIA & REPORTING LIMITS FOR KINETIC PHOSPHORESCENCE ANALYSIS PARAMETERS -- REPRESENTATIVE VALUES
 (SUBJECT TO UPDATE AS NECESSARY)

Parameter	Matrix	Reference	Precision (RPD - %)	Accuracy (% Recovery)	Reporting Limit (Default MDC)
Total Uranium	Water	ASTM D-5174-91; SOP 741/747	20 %	80 - 120	1.0 µg/L
	Solid	ASTM D-5174-91-mod; SOP 741/747	20 %	80 - 120	0.10 µg /g
	Other	ASTM D-5174-91-mod; SOP 741/747	20 %	80 - 120	2.0 µg / aliquot

Note: Precision, accuracy, and reporting limits values shown in the table above are subject to change. Consult the QA Department and LIMS database for current values.

Table 3.28. SAMPLE PREPARATION METHOD SUMMARY

Sample Preparation Method Number	Description	Matrix	Sample Preparation for Methods
EPA 3005	Acid Digestion - HNO ₃ & HCl	Water	EPA 6010B
EPA 3020	Acid Digestion - HNO ₃	Water	EPA 7060A, 7421, 7741A, 7761, 7841
EPA 3050	Acid Digestion - HNO ₃ & HCl	Solid	EPA 6010B, 7060A, 7421, 7741A, 7761, 7841
EPA 3060	Alkaline Digestion	Solid	EPA 7196
EPA 3510 ¹	Separatory Funnel Liquid-Liquid Extraction	Water	EPA 8081A, 8141A, 8270C
EPA 3520	Continuous Liquid-Liquid Extraction	Water	EPA 8081A, 8141A, 8270C, 8310
EPA 3540	Soxhlet Extraction	Solid	EPA 8081A, 8141A, 8270C, 8310
EPA 3550	Sonication Extraction	Solid	EPA 8081A, 8270C, 8310
EPA 5030	Purge-and-Trap	Water, Solid	EPA 8021B, 8015-G, 8260B
EPA 3620	Florisil Cleanup	Water, Solid	EPA 8081A
EPA 3630	Silica Gel Cleanup	Water, Solid	EPA 8081A, 8310
EPA 3640	Gel-Permeation Cleanup	Water, Solid	EPA 8081A, 8270C
EPA 3660	Sulfur Cleanup	Water, Solid	EPA 8081A, EPA 8082
EPA 3665	Sulfuric Acid Cleanup	Water, Solid	EPA 8082 (PCBs only)

¹ Not preferred extraction technique.

4. SAMPLE PRESERVATION, HOLDING TIMES AND HANDLING PROCEDURES

4.1 INTRODUCTION

Obtaining representative samples and maintaining their integrity are critical elements of any monitoring program. Analytical methods have been standardized but the results of analyses are only as good as the sampling and the sample preservation methods. Defining the magnitude and the nature of an environmental problem requires collecting representative samples for laboratory analysis and data evaluation. The careful collection of samples is key to obtaining an accurate assessment of the site's environmental impact and to developing the appropriate remedial solution. Defining in detail the numerous available sampling procedures and their associated quality elements applicable to environmental testing is beyond the scope of this document. Quality elements required to meet the DQO's for a given sampling event must be contained in a project specific sampling plan or within an overall site work plan. The plans should present the best approved techniques currently available for sampling and sample preservation.

In sampling, the objective is to remove a small portion of an environment that is representative of the entire body. After the sample has been taken, the constituents of the sample must stay in the same condition as when collected. The length of time that these constituents will remain stable is related to their character and the preservation method used. As preservation methods relate to the parameters to be analyzed, these techniques are classified by parameter.

4.2 FIELD SUPPORT

Paragon provides shipping containers, custody documents, custody seals, sample bottles, labels, chemical preservatives for water samples, "blue ice" packs to maintain thermal preservation, and trip and field blanks to support field sampling events. Table 4-1

chemical preservative was added is measured (with the exception of sample collected for volatile organic compounds) and recorded. A disposable pipette is used to remove an aliquot of the sample for the pH check. When deviations from the required chemical or thermal preservation are noted, the Project Manager is notified. The Project Manager notifies the client immediately of any chain of custody inconsistencies, temperature excursions, broken bottles, or preservation issues.

Water samples for GC and GC/MS volatile aromatics determinations are monitored for pH prior to analysis, at which time the pH of each individual sample bottle used is checked. The portion of sample used for the analytical determination is removed from the vial prior to checking the sample's pH. Sample pH measurements are recorded on laboratory chronicles as they are taken.

Sample holding times begin with the collection of samples and continue until analysis is complete. Holding times are presented in Table 4-1 through Table 4-5.

4.4 SAMPLE CONTAINERS

Paragon provides precleaned and certified sample bottles in the shipping containers for sample collection. Used sample bottles are never reused by the laboratory. Vendor prepared I-Chem 200™, 300™, Eagle Pitcher (level 3) or equivalent bottles are provided.

The Sample Control Department maintains certificates of cleanliness. These certificates will be provided to the client upon request. Containers are stored in clean areas to prevent exposure to fuels, solvents, and other contaminants.

4.5 SAMPLE RECEIPT SCHEDULE

Samples are normally delivered to Paragon during business hours within one day following field sampling unless different arrangements are made in advance with an authorized Paragon representative. Shipping containers received at the laboratory on business holidays, weekends or after normal work hours will be placed in the walk-in

through 5-5 list sample container types, preservatives and holding times. The laboratory may be able to provide pick up and delivery services to clients.

Upon receipt of the field samples at the laboratory, Paragon laboratory personnel ensure that sample bottles are maintained according to preservation requirements and that sample storage conditions do not contribute to the presence of test analytes in the samples. Separate storage areas are provided for samples to be analyzed for the following parameter groups: metals/inorganics, semi-volatile organics, volatile organics and radiochemical analyses. Sample segregation in this manner is one way Paragon minimizes the chance for cross-contamination of samples.

Paragon typically uses commercial coolers for the transport of environmental samples from the field to the laboratory. Chain-of-custody seals and forms, employed for each cooler packed at Paragon, ensure complete documentation and provide evidence of unbroken custody of the cooler contents. Coolers meet or exceed all protocol requirements (i.e., DOT, USEPA, ASTM) for shipping. Coolers are prepared at the laboratory to provide the client with all of the sample containers needed for the analyses required by a project.

4.3 SAMPLE PRESERVATION AND HOLDING TIMES

Paragon provides the required chemical preservatives for water samples and “blue ice” packs, for thermal preservation at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, in the shipping containers during the shipping process. High quality reagent grade chemical preservatives are added to sample bottles. The ice packs are supplied at ambient temperatures. It is the responsibility of those collecting the samples to properly use these materials and ensure that proper preservation techniques (chilling) are performed and preservative (chemical preservation) requirements are met.

Upon receipt of samples at the laboratory, the temperature of each cooler is measured and recorded on the chain of custody documents. Similarly, the pH of bottles to which

refrigerator and opened on the next regular business day unless prior arrangements are made in advance for that day's receipt and log-in.

Table 4-1 REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR AQUEOUS INORGANIC TESTS

NAME	CONTAINER ¹	PRESERVATION ^{2,3}	MAXIMUM HOLDING TIME
Acidity	P,G	Cool, 4 ⁰ C	14 days
Alkalinity	P,G	Cool, 4 ⁰ C	14 days
Ammonia	P,G	Cool, 4 ⁰ C, H ₂ SO ₄ to pH<2	28 days
Biochemical Oxygen Demand (BOD)	P,G	Cool, 4 ⁰ C	48 hours
Biochemical Oxygen Demand, Carbonaceous	P,G	Cool, 4 ⁰ C	28 days
Bromide	P,G	None Required	28 days
Chemical Oxygen Demand (COD)	P,G	Cool, 4 ⁰ C, H ₂ SO ₄ to pH<2	28 days
Chloride	P,G	None Required	28 days
Chlorine, Total Residual	P,G	None Required	Analyze immediately
Chromium, Hexavalent	P,G	Cool, 4 ⁰ C	24 hours
Color	P,G	Cool, 4 ⁰ C	48 hours
Cyanide, Total	P,G	Cool, 4 ⁰ C, NaOH to pH>12 0.6g ascorbic acid	14 days
Fluoride	P	None Required	28 days
Hardness	P,G	HNO ₃ , to pH<2, H ₂ SO ₄ to pH<2	6 months
Nitrogen, Total Kjeldahl	P,G	Cool, 4 ⁰ C, H ₂ SO ₄ to pH<2	28 days
Mercury	P,G	HNO ₃ to pH<2	28 days
Metals, Except Cr(+6) & Hg	P,G	HNO ₃ to pH<2	6 months
Nitrate	P,G	Cool, 4 ⁰ C	48 hours
Nitrate + Nitrite	P,G	Cool, 4 ⁰ C, H ₂ SO ₄ to pH<2	28 days
Nitrite	P,G	Cool, 4 ⁰ C	48 hours
Oil & Grease	G	Cool, 4 ⁰ C, H ₂ SO ₄ to pH<2	28 days
Organic Carbon, Total (TOC)	P,G	Cool, 4 ⁰ C, H ₂ SO ₄ to pH<2	28 days
Orthophosphate	P,G	Filter immediately, Cool, 4 ⁰ C	48 hours
pH	P,G	None Required	Analyze immediately
Phenolics	G only	Cool, 4 ⁰ C, H ₂ SO ₄ to pH<2	28 days
Phosphorus, Total	P,G	Cool, 4 ⁰ C, H ₂ SO ₄ to pH<2	28 days
Residue, Total	P,G	Cool, 4 ⁰ C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4 ⁰ C	7 days
Residue, Nonfilterable(TSS)	P, G	Cool, 4 ⁰ C	7 days
Specific conductance	P,G	Cool, 4 ⁰ C	28 days
Sulfate	P,G	Cool, 4 ⁰ C	28 days
Sulfide	P,G	Cool, 4 ⁰ C, add zinc acetate & NaOH to pH>9	7 days
Turbidity	P,G	Cool, 4 ⁰ C	48 hours

¹ Polyethylene (P) or Glass (G)

² Free chlorine must be removed prior to addition of HCl by the appropriate addition of sodium thiosulfate.

³ Adjust to pH<2 with H₂SO₄, HCl.

Table 4-2 REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR AQUEOUS ORGANIC TESTS

NAME	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME
Purgeable Halocarbons	G, Teflon-Lined Septum	Cool, 4° C Na ₂ S ₂ O ₃	14 days
Formaldehyde (8315, 554)	G	Cool, 4° C	3 days until extraction, 3 days after extraction
Purgeable Aromatic Hydrocarbons	G, Teflon-Lined Septum	Cool, 4° C 0.008% Na ₂ S ₂ O ₃ , HCl pH <2	14 days
Acrolein and acrylonitrile	G, Teflon-Lined Septum	Cool, 4° C 0.008% Na ₂ S ₂ O ₃ , Adjust pH to 4-5	14 days
Phenols	G, Teflon-Lined Septum	Cool, 4° C 0.008% Na ₂ S ₂ O ₃ cap	7 days until extraction, 40 days after extraction
Benzidines	G, Teflon-Lined Septum	Cool, 4° C 0.008% Na ₂ S ₂ O ₃ cap	7 days until extraction, 40 days after extraction
Phthalate esters	G, Teflon-Lined Cap	Cool, 4° C	7 days until extraction, 40 days after extraction
Nitrosamines	G, Teflon-Lined Cap	Cool, 4° C, store in dark, 0.008%, Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
PCBs	G, Teflon-Lined Cap	Cool, 4° C	7 days until extraction, 40 days after extraction
Nitroaromatics and cyclic ketones	G, Teflon-Lined Cap	Cool, 4° C, store in dark, 0.008%, Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Polynuclear aromatic hydrocarbons	G, Teflon-Lined Cap	Cool, 4° C, store in dark, 0.008%, Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Haloethers	G, Teflon-Lined Cap	Cool, 4° C, store in dark, 0.008%, Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Chlorinated Hydrocarbons	G, Teflon-Lined Cap	Cool, 4° C, store in dark, 0.008%, Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Dioxins and Furans	G, Teflon-Lined Cap	Cool, 4° C, 0.008%, Na ₂ S ₂ O ₃	7 days until extraction, 40 days after extraction
Pesticides	G, Teflon-Lined Cap	Cool, 4° C ² pH 5-9	7 days until extraction, 40 days after extraction

Table 4-3 REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR AQUEOUS RADIOLOGICAL TESTS

NAME	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME
Alpha, Beta, Radium, Gamma,U,Th,Pu,Am,Cm, Np,Sr,Tc	P,G	HN0 ₃ < pH 2	*180 days
Gross Alpha/Beta	P	HN0 ₃ < pH 2	*180 days
Radium 226/228/Total	P	HN0 ₃ < pH 2	*180 days
Uranium/Plutonium, Thorium, Americium, Neptunium, Curium, Strontium 89/90/Total	P	HN0 ₃ < pH 2	*180 days
Tritium	G	Cool 4° C	*180 days
Tc99	P	HN0 ₃ < 2	*180 days
Gamma Spec	P	HN0 ₃ < 2	*180 days
C-14	G	pH>7 w/NaOH	*180 days
RadioIodine	P,G	0.008% Na ₂ S ₂ O ₃	*180 days

* - Paragon internal guideline; not regulatory. For Iodine-131, analyze as soon as possible (< 8 days).

Table 4-4 REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR NON-AQUEOUS LIQUIDS, SOIL OR SOLID MATRICES

NAME	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME
<i>Semivolatile Organics/Organochlorine Pesticides/PCBs and Herbicides</i>			
Concentrated waste samples	8 oz. wide mouth glass w/Teflon liner	None	14 days until extraction, 40 days after extraction
Soil/sediments and sludges	8 oz. wide mouth glass w/Teflon liner	Cool, 4° C	14 days until extraction, 40 days after extraction
<i>Volatile Organics</i>			
Concentrated waste samples	8 oz. wide mouth glass w/Teflon liner	None	14 days
Liquid samples, no residual Chlorine present	3x40 mL vials w/Teflon lined septum caps	Cool, 4° C ⁴	14 days
Liquid samples, residual chlorine present	3x40 mL vials w/Teflon lined septum caps	Collect sample in a 4 oz. soil VOA container which has been pre-preserved w/4 drops of 10% sodium thiosulfate. Gently mix sample & transfer to a 40 mL VOA vial. Cool to 4° C	14 days
Acrolein & Acrylonitrile	3x40 mL vials w/Teflon lined septum caps	Adjust to pH 4-5, Cool to 4° C	14 days
Soil/sediments and sludges	4 oz. (120 mL), wide mouth glass w/Teflon liner or wide mouth glass container sealed w/a septum	Cool to 4° C	14 days

⁴ Adjust pH<2 w/H₂SO₄, HCl.

Table 4-5 REQUIRED CONTAINERS, PRESERVATION, AND HOLDING TIMES
 FOR AIR METHODS

TEST	MEDIA	PRESERVATION	MAXIMUM HOLDING TIME
TO1	Tenax tubes	Freezer - 20° C	14 days
TO2	Carbo Sieve	Cool to 4° C	14 days
VOST	Tenax/Tenax-Charcoal	Cool to 4° C	14 days
TO4	Puf 3" long, 60mm diameter	Freezer - 10° C or below	Extracted 7 days after collection
TO10	Puf 10 cm long, 20 mm diameter	Cool to 4° C	Extracted 7 days after collection
TO11	Absorbent cartridge	Cool to 4° C	30 days
TO13	Puf XAD/XAD		Extracted 7 days after collection

5. SAMPLE CUSTODY

5.1 SAMPLE RECEIPT

Sample shipments are received at the sample receiving area. Sample custodians verify the number of shipping containers received against the numbers listed on the shipping manifest/chain-of-custody. Any damage to the shipping containers or other discrepancy observed is noted on the chain-of-custody before signing it.

The external chain-of-custody must be signed by the carrier for relinquishment of samples and signed by sample custodian personnel for sample receipt. The actual chain-of-custody may be supplied by Paragon, or may be the client's own form. The chain-of-custody remains in the project file at all times.

5.2 CHAIN-OF-CUSTODY AND SAMPLE LABELS

Chain-of-Custody encompasses three major elements: field sampling, laboratory analysis, and final data file. A Chain-of-Custody (COC) document may be the means in some types of legal proceedings by which evidence of custody of samples from time of receipt to completion of analysis is proved in the courts. The Paragon laboratory has implemented standard operating procedures (SOPs) to ensure that sample custody objectives of traceability and responsibility are achieved for every project. This section covers quality related activities from the receipt of samples at the laboratory through the issuance of final analytical data and the storage of data in its final data file.

The National Enforcement Investigations Center (NEIC) of EPA defines evidence of custody in the following manner:

- It is in your actual possession, or
- It is in your view, after being in your physical possession, or
- It was in your possession and then you locked or sealed it up to prevent tampering,
or
- It is in a secure area.

Samples may be physical evidence and should be handled according to certain procedural safeguards. Field personnel or client representatives complete a Chain-of-Custody Form for all samples. Samples are received by the laboratory accompanied by these forms.

The sampler should provide the following information on the Chain of Custody Form and sample label:

- Client project name
- Project location
- Field sample number/identification
- Date and time of sample collection
- Sample matrix
- Container type and number of containers for each sample
- Preservative
- Analysis requested
- Sampler signature
- Signature of person relinquishing samples
- Date and time relinquished
- Sampler remarks
- Custody Seal Number (if applicable)
- Designation of MS/MSD

The record is filled out completely and legibly. Errors are corrected by drawing a single line through and initialing and dating the error. The correct information is then recorded with indelible ink. All transfers of samples except to and from commercial couriers must be recorded on the Chain-of-Custody via the "relinquished" and "received by" sections. All information except signatures may be printed.

5.3 SAMPLE VERIFICATION

Upon arrival of a sample shipment and following initial screen for radioactivity, sample control personnel perform sample inspection. Paragon's Sample I.D. and Condition Sheet (Figure 5-1) serves as a check list of procedures to follow and as documentation of the following:

- Presence/absence of custody seals or tapes of the shipping containers and the condition of the seals (i.e., intact, broken).
- Presence/absence of chain-of-custody; (if present, is it complete?)
- Presence/absence of sample tags; (if present, are they removable?)
- Agreement/non-agreement between the sample tags, chain-of-custody, and any client documentation.
- Condition of the samples when received, including:
 - Sample temperature
 - Intact, broken/leaking
 - Headspace in VOA vials
 - Sample holding time
- Sample pH when required completion of radiological screen to ensure compliance with DOT regulations

If discrepancies are found, the Paragon Project Manager is contacted immediately. If the Project Manager is not available, the Quality Assurance Manager is contacted for further directions. A copy of a Discrepancy Report Form is included in the project file.

5.4 SAMPLE LOGIN POLICIES

Upon completing sample receipt/custody procedures, all sample and analysis data must be complete and documented on the chain of custody or accompanying forms for input into the Laboratory Information Management System (LIMS).

Figure 5-1 CONDITION OF SAMPLE UPON RECEIPT FORM

CLIENT: _____	SHIPPING CONTAINER		
#: _____			
WORK ORDER NO. _____	INITIALS: _____		
DATE: _____			
1.	Does this project require special handling according to NEESA, Level 3, or CLP protocols? If yes, complete a. and b. a. Cooler Temperature _____ b. Lot No's. _____ c. Airbill Number _____		Yes No
2.	Are custody seals on the cooler intact? If so, how many _____	N/A	Yes No
3.	Are custody seals on sample containers intact?	N/A	Yes No
4.	Is there a Chain of Custody (COC) or other representative documents, letters or shipping memos?		Yes No
5.	Is the COC complete? Relinquished: Yes No Requested Analysis: Yes No	N/A	Yes No
6.	Is the COC in agreement with the samples received? No. of Samples: Yes ___ No ___ Sample ID's: Yes ___ No ___ Matrix: Yes No No. of Containers: Yes No		Yes No
7.	Are the samples requiring acid preservation preserved correctly?	N/A	Yes No
8.	Is there enough sample? If so, are they in the proper containers?		Yes No
9.	Are all samples within holding times for the requested analyses?		Yes No
10.	Were the sample received on ice?	N/A	Yes No
11.	Were all sample containers received intact? (not broken or leaking, etc.)		Yes No
12.	Are samples requiring no headspace, headspace free?	N/A	Yes No
13.	Do the samples require quarantine?		Yes No
14.	Do samples require Paragon disposal?		Yes No
15.	Did the client return any unused bottles?		Yes No
Describe "NO" items (except No's 1, 13, & 14): _____ _____ _____			
Was the client contacted? Yes _____ No _____ If yes, Date: _____ Name of person contacted: _____			
Describe actions taken or client instructions: _____ _____ _____			
Group Leader's Signature: _____ Date: _____			

Sample and analysis data must include:

- Client name and contact
- Client number
- Paragon Work Order Number
- Paragon Project Manager
- Sample descriptions
- Due date
- List of analyses requested

Sample and requested analyses data are then input into the LIMS. All samples received before 1200 hours are logged into the LIMS on the day of receipt. Samples received after 1200 hours are logged in before 1200 hours the following business day.

A Work Order is generated immediately by the LIMS. The Work Order, Chain-of-Custody, Condition Upon Receipt form and Program Specifications (if applicable) are distributed as follows:

- To the Paragon Project Manager;
- To the project file;
- To each department; and
- To the client via facsimile.

Work Order is to be reviewed against the chain of custody by the Project Manager. Any discrepancies discovered by the Project Manager will be noted on the Work Order, which will be returned to the Sample Receiving Department for correction. After the Project Manager approves the Work Order it will be distributed as described above.

Sample containers are labeled with the corresponding Paragon sample ID and the stamped date of receipt. After the containers are labeled, samples are ready for storage in the appropriate area.

5.5 WHEN SAMPLES ARE RECEIVED WITH INADEQUATE DOCUMENTATION

If delivered by a client: Client is asked if previous arrangements were made for analysis (and with whom). The client completes a chain of custody and/or request for analysis, relinquishes samples to sample custodian personnel, and is given a copy of the C-O-C.

If received by courier or shipping:

- Routine Client File is checked
- Paragon key client contact is consulted
- General Manager is consulted to determine the designated Paragon Project Manager
- Information is requested from the Paragon Project Manager

If analysis information cannot be determined on the day of sample receipt, sample data entry personnel proceed to assign sample numbers and put samples on hold. Follow-up with Project Manager occurs until the analyses are determined and samples can be properly logged in.

5.6 RESPONSIBILITIES FOR SAMPLE LOG IN

5.6.1 SAMPLE CUSTODIAN

- Has the primary responsibility of ensuring that sample information is input into the LIMS as described in the SOP.
- Has the responsibility to make recommendations to the Quality Assurance Manager for revising the SOP.

5.6.2 PROJECT MANAGER

- Has the overall responsibility for ensuring that this procedure is implemented for all samples received into the laboratory.
- Has overall responsibility for ensuring that samples are logged in correctly (given that appropriate information has been supplied).

5.7 SAMPLE STORAGE

5.7.1 GENERAL PROCEDURES

Samples are stored immediately upon receipt to prevent sample degradation.

5.7.2 REFRIGERATED STORAGE AREA MAINTENANCE

All refrigerated storage areas are maintained at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The temperature is monitored and recorded daily. If the temperature falls outside the limit of 2° - 6°C , corrective action is to be taken as follows and appropriately documented.

- Temperature is monitored at 30 minute intervals with the refrigerator door closed.
- Quality Assurance Manager is notified if the problem persists longer than one hour.
- Samples are relocated to a proper storage environment if temperature cannot be maintained after corrective actions are implemented.

5.7.3 ROUTINE SAMPLE STORAGE

- Samples within each project are stored in sample number order.
- Waters and soils are generally stored on labeled separate shelves.

5.7.4 SPECIFIC PROCEDURES

- Volatiles

Samples within a project are stored in numerical order in vial containers. The holders are then stored where space permits in one of the designated volatile organic refrigerated storage areas.

- Semi-Volatiles

Samples within a project are stored in numerical order in a designated, refrigerated storage area.

- Hazardous Materials

Pure product or potentially heavily contaminated samples are tagged as "hazardous" and stored within a secured area, separate from other samples. This area is used only for

hazardous samples and is labeled per Occupational Health and Safety Administration (OSHA) requirements.

- Radiochemistry

Samples designated for radiochemistry analyses only are segregated and maintained at ambient temperature. Those samples having suspected activity and scheduled for chemical analyses are segregated and refrigerated. Access to this area is limited.

5.7.5 RESPONSIBILITIES FOR SAMPLE STORAGE

- Project Manager has overall responsibility for ensuring that the Standard Operating Procedure (SOP) is followed, samples are stored properly upon receipt, and refrigerated storage area temperatures are maintained.
- Sample custodians are responsible for storing all samples upon receipt into the appropriate storage area, maintaining high level security for those samples under custody, and for keeping a current custody sample inventory.
- Analytical personnel have the responsibility of daily sample storage area maintenance, disposal of old samples, and providing space for incoming samples in routine storage areas.

Assigned individuals are responsible for maintaining and documenting: (a) refrigerated storage area temperatures, and (b) corrective actions.

5.8 SAMPLE/DATA ACCESS AND INTERNAL CHAIN-OF-CUSTODY

5.8.1 GENERAL POLICIES AND PROCEDURES

Paragon has implemented standard operating procedures to ensure the integrity of samples and data so that they are not degraded or disclosed to unauthorized personnel. In order to ensure that this policy is maintained, the laboratory facilities are under controlled access. Only employees are allowed into the laboratory facilities; visitors must register at the front desk.

Samples are removed from their proper location by designated personnel and returned to the storage area immediately after the required sample quantity has been taken. This procedure minimizes unnecessary time spent searching for samples and helps prevent matrix degradation from prolonged exposure to room temperature. After the final report is sent and clients are allowed adequate time to review the results, the samples are properly discarded or returned to the client.

Upon client request, additional and more rigorous chain-of-custody protocols for samples and data can be implemented. For samples involving a high degree of confidentiality or potential litigation, Paragon has developed extensive sample and data handling protocols to ensure the scientific and legal defensibility of the report submitted. These protocols include those specified by the USEPA Contract Laboratory Program.

Analysts and technicians follow strict internal chain-of-custody procedures to further ensure the validity of all data. All samples are signed out in a sample custody log book when they are removed for analysis. The sample ID, date, time, analyst, and lab of analysis is recorded in the sample custody log or equivalent. Samples are signed back in noting date, time, and storage location, upon return.

5.8.2 RESPONSIBILITIES FOR INTERNAL CHAIN-OF-CUSTODY SOP COMPLIANCE

- The Quality Assurance Manager has the overall responsibility for ensuring that the SOP is implemented and followed.
- The sample custodian personnel have the responsibility for ensuring that the SOP is properly followed, and to notify the Quality Assurance Manager of problems.
- All employees checking out samples are required to follow procedures.

5.9 SUBCONTRACTING ANALYTICAL SERVICES

Every effort is made to perform analyses within the Paragon laboratory. Should subcontracting be necessary, samples are placed at other certified labs -- only after

receiving the client's *written* approval. See SOP 103 for additional information on evaluating a subcontract laboratory and data.

When subcontracting becomes necessary, a preliminary verbal communication with an appropriate laboratory is undertaken. Work performed under specific protocols may involve special consideration. For instance, work involving AFCEE, NFESC, or USACE samples may be subcontracted only to agency-approved laboratories. The contact and preliminary arrangements and terms of agreement are made between the Paragon Project Manager and the appropriate subcontract laboratory personnel (i.e., laboratory manager, customer services contact, or the appropriate laboratory section manager). The specific terms of the subcontract laboratory agreement should include (when applicable):

- Method (EPA or otherwise) of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required (hardcopy, electronic)
- Applicable laboratory certification status
- Price per analysis
- Turn around time requirements

Chain-of-Custody forms must be generated for samples which require subcontracting to other laboratories. The sample management personnel repackage the samples for shipment, create a transfer chain-of-custody form and record the following information:

- Paragon Work Order Number.
- Matrix.
- Requested analysis.
- Special instructions (quick turn around, required detection limits, anything unusual known about the samples or analytical procedure).
- Signature in "Relinquished By" block of the COC form.

All subcontracted sample data reports are sent to the Paragon Project Manager.

5.10 SAMPLE DISPOSAL

After completion of sample analysis and submission of the analytical report, unused portions of samples are retained by the laboratory for a minimum of 60 days from date of invoice. Samples will be disposed of or returned to the client according to the nature of the samples. Samples are considered hazardous waste or mixed waste and are handled by state and federally licensed hazardous waste or mixed waste disposal firms.

5.11 EXCESS SAMPLE DISPOSITION

Samples not consumed during the analyses are returned to the client or disposed of by Paragon. It is the Sample Custodian's responsibility to ensure that proper disposal has taken place.

If samples are to be returned to the client or held longer than 60 days, a sample disposition form is generated. Otherwise, samples are disposed of 60 days after invoice.

6. ANALYTICAL PROCEDURES

Paragon is capable of analyzing the full range of environmental samples from all media, including surface and groundwater, soil, sediment, tissue, and waste. Methodologies are employed with guidance from agencies such as EPA, ASTM, and - in certain instances - regulatory agencies. Paragon also develops and validates methodologies which are more applicable to a specific problem or objective.

Analytical procedures are detailed descriptions of any and all processing, preparation and analysis of samples in the laboratory. In some instances, data format, presentation and delivery are also described. All analytical procedures shall be conducted in strict adherence with written Standard Operating Procedures manuals which have been reviewed and approved by the Department Manager, the Quality Assurance Manager, and Laboratory Manager. Documents from which SOPs are developed include the references listed in Table 6-3. Additional SOPs may be adapted from other sources or generated in-house as project needs require.

6.1 ANALYTICAL METHODS

Numerous sources of information are available to offer guidance in analytical methods. Selection of the appropriate method is dependent upon data usage and the regulatory requirements during the analysis. Table 6-3 describes the analytical references routinely used by Paragon. Paragon may modify existing methods based on the following considerations: 1) in order to meet project specific objectives; 2) in order to incorporate modifications or improvements in analytical technology; 3) in order to comply with changing regulations and requirements; 4) in order to address unusual matrices not covered in available methods, 5) to provide analytical capabilities for analytes for which there are no promulgated methodologies.. Paragon will make every effort to disclose to its clients any instances in which modified methods are being used in the analysis of samples.

The following subsections contain method synopses for representative methods most frequently performed at Paragon. For clarity purposes, certain method summaries also

contain calibration criteria, several of which are also described in Section 7, Calibration Procedures and Frequency.

6.1.1 COMPLIANCE

6.1.1.1 DEFINITION

Compliance is the proper execution of recognized, documented procedures which are either approved or required. Adherence to these procedures is required in order to provide data products acceptable to a regulatory body of competent jurisdiction in a specific regulatory context. Compliance is separate from, but not inconsistent with, technical scientific quality. Paragon accepts compliance as an integral part of the definition of quality. Paragon understands that the expectations of our clients commonly include the assumption that data and reports will satisfy a regulatory purpose and will be found acceptable and compliant with regulatory requirements for the performance of tests and generation of data.

6.1.1.2 UNDERSTANDING THE REGULATORY FRAMEWORK

Compliance is not likely to be achieved in the absence of an understanding of the regulatory framework. Paragon will attempt to ascertain, prior to beginning a project, what regulatory jurisdiction (USEPA, NJDEPE, etc.) pertains to a project; within the regulatory jurisdiction, what body of regulation is meant to be satisfied (RCRA, SDWA, 21E, etc.); and finally, within this context, what protocols are required/expected (CLP, AFCEE, NEESA, USACE, etc.). Paragon will work with its clients to achieve a mutual understanding of all requirements.

6.1.1.3 COMMITMENT

Paragon makes the following commitments to its clients:

- Paragon will proactively attempt to identify and understand the regulatory context of clients' needs.
- Paragon will strive to be expert in understanding and executing the regulatory requirements for compliance.

- Paragon will identify and disclose to clients instances of non-compliance in a forthright fashion.

6.1.1.4 RESOLVING COMPLIANCE CONTRADICTIONS AND HIERARCHIES

It is a common occurrence that multiple regulatory jurisdictions overlap in a specific case. This causes uncertainty or even contradictions to arise in a work plan. Paragon will make every effort to detect such inconsistencies, and will communicate them to clients so that an informed decision can be made by the client regarding execution of the project. Similarly, methods and protocols will often be prescribed in a scope of work or QAPP which either will not achieve stated or implied DQOs or which are in conflict with the regulatory requirements. Paragon will attempt to detect these inconsistencies, and upon detection, disclose same to our client. Paragon voluntarily accepts a responsibility to provide advice to clients; however, the primary responsibility for resolving inconsistencies with regulators remains with the client.

6.1.1.5 DISCLOSURE OF NONCOMPLIANCE

As stated, it is Paragon's policy to disclose in a forthright manner any detected noncompliance that may affect the usability of data produced by Paragon. It is not within our expertise to predict the manner in which a specific regulator or regulatory body will interpret the rules governing analysis; therefore, Paragon is unable to guarantee compliance. It is Paragon's policy that our responsibility begins with a bona fide and competent attempt to evaluate potential compliance issues and ends with disclosure of any findings that may be useful to our client in their making the final judgment.

6.2 NON-STANDARD METHODS VALIDATION

When non-promulgated methods (i.e. methods other than EPA, NIOSH, ASTM, AOAC, etc.) are required for specific projects or analytes of interest, or when the laboratory develops a method, the laboratory must establish the validity of the method prior to applying it to client samples. Method validity is established by meeting criteria for precision and accuracy. Method development and validation must include the following: MDL study; validated extraction and analytical criteria; SOP generation and approval; and

comparison studies on matrix spike samples. The method development study will be treated as a work order and all data filed appropriately (see Section 10.7 of the LQAP).

6.3 SUMMARY OF SAMPLE PREPARATION METHODS

6.3.1 DIGESTION OF AQUEOUS SAMPLES FOR METALS - METHOD 3005A

This method is an acid digestion procedure used for the preparation of water samples for metals analysis. The digested samples can be analyzed for dissolved and total recoverable metals by flame (FLAA) or furnace (GFAA) atomic absorption spectrophotometry or by inductively coupled argon plasma emission spectroscopy (ICP). Method 3005 may be used to prepare samples for analysis of the following metals:

Aluminum	Cobalt	Potassium
Antimony	Copper	Selenium
Arsenic	Iron	Silver
Barium	Lead	Sodium
Beryllium	Magnesium	Thallium
Cadmium	Manganese	Vanadium
Calcium	Molybdenum	Zinc
Chromium	Nickel	

For the analysis of total recoverable metals, the entire sample is acidified at the time of collection with nitric (HNO₃) acid. Sample preparation involves heating the sample with nitric acid and concentrating to a specified volume. The sample is not allowed to boil because some elements are in a volatile state and may be easily lost. The digestate is then filtered (if necessary) and diluted to the desired final volume for analysis. For the analysis of dissolved metals in water samples, some analysis methodologies do not require sample digestion prior to analysis. If the client clearly identifies samples as dissolved water samples, they laboratory may analyze these samples without digestion.

For the analysis of dissolved metals, the samples are filtered through a 0.45-um filter immediately upon collection and prior to acidification with nitric acid. In the lab, the sample is heated with acid and the volume is reduced. The digestate is filtered again (if necessary) and diluted to volume.

6.3.2 DIGESTION OF AQUEOUS SAMPLES FOR METALS - METHOD 3010A AND THE CLP SOW

These methods describe the preparation of aqueous samples for total metals determination by flame atomic absorption spectrophotometry (FLAA) and by inductively coupled argon plasma emission spectroscopy (ICP). By Method 3010A, samples are vigorously digested with nitric and hydrochloric acids.

6.3.3 DIGESTION OF AQUEOUS SAMPLES FOR METALS - METHOD 3020A AND THE CLP SOW

These methods describe the preparation of aqueous samples for total metals determination by graphite furnace atomic absorption spectroscopy (GFAA). By Method 3020A, samples are vigorously digested with nitric acid and hydrogen peroxide. By CLP protocol, samples are digested with a mixture of nitric acid and hydrogen peroxide.

6.3.4 DIGESTION OF SOLID SAMPLES FOR METALS - METHOD 3050A AND THE CLP SOW

These methods are applicable to the preparation of sediment, sludge, and soil samples for metals determination by FLAA or GFAA or by ICP. One gram of solid sample is digested with nitric acid and hydrogen peroxide. The digestate is then refluxed with nitric or hydrochloric acid, depending on the analysis performed. When using hydrochloric acid as the final refluxing acid, the digests may not be boiled because antimony is in a volatile state and may be easily lost. A separate sample aliquot is dried to determine the percent moisture in the sample.

6.3.5 SEPARATORY EXTRACTION - METHOD 3510C

Method 3510C is designed to quantitatively extract nonvolatile and semivolatile organic compounds from liquid samples using separatory funnel techniques. The sample and extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method that will be used to analyze the extract. Samples are pH-adjusted and serially extracted by vigorous shaking for 1-2 minutes with the appropriate solvent for the analytical method.

Samples are extracted three times, the combined extracts are dried with anhydrous sodium sulfate, and concentrated in a Kuderna-Danish apparatus.

6.3.6 CONTINUOUS LIQUID/LIQUID EXTRACTION - METHOD 3520C

Method 3520C is designed to quantitatively extract nonvolatile and semivolatile organic compounds from liquid samples using continuous liquid-liquid extractors. The sample and extracting solvent must be immiscible in order to recover target compounds. Subsequent cleanup and detection methods are described in the organic analytical method that will be used to analyze the extract. Samples are pH-adjusted and extracted with the appropriate solvent for the analytical method. Samples are extracted for 18 to 24 hours and the extracts dried with anhydrous sodium sulfate and concentrated in a Kuderna-Danish apparatus.

6.3.7 SOXHLET EXTRACTION - METHOD 3540C

Method 3540C is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent. Extraction is accomplished by mixing the solid sample with anhydrous sodium sulfate, placing it in an extraction thimble or between two plugs of glass wool, and extracting it with an appropriate solvent in the Soxhlet extractor for 18 to 24 hours. The extract is dried and concentrated and then treated using a cleanup method or analyzed directly by the appropriate measurement technique.

6.3.8 SONICATION EXTRACTION - METHOD 3550B

Method 3550B is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The Sonication process ensures intimate contact of the sample matrix with the extraction solvent. A weighed sample of the solid waste is mixed with sodium sulfate, then dispersed into the solvent using sonication. The extract is dried with anhydrous sodium sulfate and concentrated with a Kuderna-Danish apparatus. The resulting solution may then be cleaned up or analyzed directly using the appropriate technique.

6.3.9 WASTE DILUTION - METHOD 3580A

Method 3580A is a technique for solvent dilution of non-aqueous waste samples prior to sample cleanup and/or analysis. It is designed for wastes that may contain organic constituents at concentrations greater than 20,000 ug/kg and that are soluble in the dilution solvent.

6.3.10 PURGE-AND-TRAP SAMPLE INTRODUCTION - METHOD 5030B/5035

Method 5030B/5035 is used to determine the concentration of volatile organic compounds in a variety of liquid and solid waste matrices using a purge and trap gas chromatographic procedure. The success of this method depends on the level of interferences in the sample. Results may vary due to the large variability and complexity of various matrices.

Inert gas is bubbled through a 5-mL or 25-mL aqueous sample aliquot at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept to a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is flash heated and backflushed with inert gas to desorb and transfer the volatile components onto the head of a GC column. The column is heated to elute the volatile components, which are detected by the appropriate detector for the analytical method used.

Solid samples may be analyzed using one of two techniques. For GC and GC/MS analysis, 5 g of solid sample is dispersed into 5 mL of blank laboratory water and the sample is purged as described above. This technique is referred to as the direct purge method. If analyte concentrations in the sample are above the upper calibration range, a medium-level methanol extraction may be performed. In this procedure, an aliquot of solid sample is dispersed in methanol to dissolve the volatile constituents, and a portion of the methanol extract is combined with blank purge water and purged as described above.

6.3.11 EXTRACTION PROCEDURE TOXICITY TEST (EP-TOX) - METHOD 1310A

This method is used to determine whether a waste exhibits the characteristics of extraction procedure (EP) toxicity. If a representative sample of the waste contains >0.5% solids, the solid phase of the sample is ground to pass a 9.5 mm sieve and extracted with deionized (DI) water that is pH adjusted with acetic acid. Wastes containing <0.5% solid material are extracted and analyzed as a single phase.

6.3.12 TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) - METHOD 1311

This method is used to determine whether a waste exhibits toxicity leaching characteristics. The procedure includes a leaching extraction for semivolatile compounds and metals and a zero-headspace extraction for volatile compounds.

6.3.13 CALIFORNIA ASSESSMENT MANUAL WASTE EXTRACTION TEST (CAM WET)

This waste extraction test, described in the California Administrative Code, Title 22, Article 11, Section 66700, can be used to determine the amount of extractable substance in a waste or other material.

6.3.14 PREPARATION OF SAMPLES FOR THE DETERMINATION OF ACTINIDES

Preparation of water samples for actinide analysis begins with generating an iron hydroxide co-precipitate. The iron hydroxide precipitate is dissolved in strong acid (either HCl or HNO₃). Purification of the various actinides is then accomplished using anion exchange procedures. Following anion exchange, the analytes are mounted for alpha counting/analysis by forming a lanthanum fluoride co-precipitate and collecting the precipitate on a membrane filter.

Preparation of solid samples for this determination begins with a total dissolution of the solid matrix using hydrofluoric, nitric and hydrochloric acids. An initial separation is performed by generating an iron hydroxide co-precipitate. The iron hydroxide precipitate

is dissolved in strong acid (either HCl or HNO₃). Purification of the various actinides is then accomplished using anion exchange procedures. Following anion exchange, the analytes are mounted for alpha counting/analysis by forming a lanthanum fluoride co-precipitate and collecting the precipitate on a membrane filter.

6.3.15 PREPARATION OF SAMPLES FOR GROSS ALPHA AND GROSS BETA DETERMINATION

Water samples are prepared for gross alpha and gross beta analysis by evaporation of an aliquot on a stainless steel planchet. The samples will be stabilized with acid to maximize the solubility of the radionuclides. The aliquot size used for analysis varies since the maximum amount of residue permitted for counting is 100 mg.

Soil samples are prepared for gross alpha and gross beta analysis by leaching the soil with a nitric acid solution on a steam bath. An aliquot of the leachate (containing no more than 100 mg of solid) is then evaporated on a stainless steel planchet for counting.

6.3.16 PREPARATION OF SAMPLES FOR THE DETERMINATION OF RADIOSTRONTIUM

The preparation of water samples for radiostrontium analysis begins by concentration of the analyte trapping it on a cation exchange column. Strontium (and other cations) are then stripped from the column with a nitric acid solution, and the eluent collected.

Strontium is then further concentrated and purified using ion exchange columns packed with a resin that has a high affinity for strontium (Sr - spec). The purified/concentrated strontium is then driven off the Sr - spec resin with dilute nitric acid, and the eluent collected. This eluted solution is evaporated on a stainless steel planchet for beta counting. Chemical yield is determined by Inductively Coupled Argon Plasma analysis of the chemical species of stable strontium.

Analysis of radiostrontium in solids is done by performing a nitric acid leach on the solid sample. The leachate is processed to purify strontium following a procedure identical to the analysis for radiostrontium in water described above.

6.3.17 PREPARATION OF SAMPLES FOR THE DETERMINATION OF TRITIUM

Water samples are prepared for tritium analysis by distillation. Potassium permanganate and sodium hydroxide are added prior to distillation to remove interferences arising from organic constituents. An aliquot of the distillate is mixed with a liquid scintillation cocktail for beta counting.

Solid samples are prepared for tritium analysis by distillation of water from the solid using a microwave distillation apparatus. If insufficient water is available (dry soil), additional water is added and allowed to equilibrate before the distillation is performed. An aliquot of the distillate is mixed with a liquid scintillation cocktail for beta counting.

6.3.18 PREPARATION OF WATER SAMPLES FOR THE DETERMINATION OF RADIUM 228

Radium in a water sample is collected by co-precipitation with barium and lead sulfate. The precipitate is collected, washed and redigested. Further purification of radium is accomplished by reprecipitation of analyte with an ethylenediamine-tetraacetic acid (EDTA) solution. Both Ra-226 and Ra-228 are collected in this precipitate. After a 36-hour ingrowth of Ac-228 (created from the radioactive decay of Ra-228), the Ac-228 is co-precipitated with yttrium oxalate, purified and beta counted immediately after final collection.

6.3.19 PREPARATION OF SAMPLES FOR THE DETERMINATION OF TOTAL URANIUM BY KINETIC PHOSPHORESCENCE ANALYSIS (KPA)

Water samples are prepared for KPA analysis by evaporation to dryness followed by digestion with nitric acid. After digestion is complete, the samples are restored to their original volume. An aliquot is then mixed with URAPLEX complexing reagent prior to measurement by KPA.

Soil samples are prepared for KPA analysis by dissolution using hydrogen peroxide and hydrofluoric, hydrochloric, nitric acids. After digestion is complete, the digestates are

brought to a known volume. an aliquot of the digestate is then mixed with URAPLEX complexing reagent and analyzed in the same manner as water samples by KPA.

6.3.20 PREPARATION OF SAMPLES FOR THE DETERMINATION OF NI-63

Nickel-63 is dissolved from solids using a nitric acid leach procedure. Nickel in the leachate is purified by formation of a complex with dimethyl glyoxime (Ni - DMG). The Ni - DMG is extracted from the sample with chloroform and evaporated to dryness. The Ni - DMG is dissolved in concentrated HNO_3 , diluted with water and mixed with scintillation cocktail for beta counting.

Nickel-63 in water samples is preconcentrated by passing the sample through a cation exchange column, which will immobilize the nickel on the resin carrier. Sorbed nickel is stripped from the cation exchange column with a nitric acid solution. The collected eluent (enriched with Ni-63) is then processed through the Ni-DMG extraction process described above for the solid analysis.

6.3.21 PREPARATION OF SAMPLES FOR THE DETERMINATION OF PB-210

Lead in solids is solubilized by the total dissolution of the matrix using nitric, hydrochloric and hydrofluoric acids. Lead in the solid digestates and water samples is preconcentrated by passing the sample through a cation exchange column, which will immobilize the lead on the resin carrier. The selected chromatographic resin is chosen to have a high affinity for lead, which will allow potentially interfering radionuclides to be removed from the sample matrix. Lead is then stripped from the cation exchange column with hydrochloric acid. The purified solution containing lead is reduced in volume and transferred to a planchet, where it is evaporated to dryness. Lead-210 is determined by measuring the ingrowth of bismuth-210 (the radioactive decay product of lead-210). Non-radioactive lead is spiked into the samples at the initial stages of processing to monitor chemical recovery of the sample preparation process.

6.3.22 PREPARATION OF SAMPLES FOR CARBON-14 ANALYSIS

Carbon is separated from sample matrices by conversion to carbon dioxide with a heated potassium permanganate and sulfuric acid digestion. The evolved carbon dioxide gas is trapped in a basic water solution. An aliquot of this distillate trap solution is then mixed with liquid scintillation cocktail for counting.

6.3.23 PREPARATION OF SAMPLES FOR TECHNETIUM-99 ANALYSIS

Technetium-99 is leached from solids with 1 N nitric acid, which will mobilize this analyte and bring it into the aqueous phase. Tc-99 is then selectively absorbed onto a TEVA-Spec chromatographic resin, which will separate this analyte from potential interferences. The analyte is then driven off the resin with 12 N nitric acid, and the eluent collected. The eluent is reduced in volume, and transferred to a planchet and evaporated to dryness. The dried planchet is then presented to the analytical instrumentation for beta counting.

The processing steps for water samples is identical to the soil procedure except there is no need for an initial acid leach.

6.3.24 PREPARATION OF WATER SAMPLES FOR IODINE-129 ANALYSES

A stable (non-radioactive) iodate carrier solution is spiked into an acidified water sample. The iodate (with I-129) is reduced to the iodide state with sodium sulfite and the iodide is precipitated with silver as AgI. The precipitate is filtered, which removes interferences and precipitate is dissolved with zinc powder and sulfuric acid. This resolubilizes the iodide, which is then reprecipitated as palladium iodide, leaving most of the halide anions in solution. The PdI₂ is then filtered, and the filter submitted to the analytical instrumentation for beta counting.

6.4 CALIBRATION AND ANALYSIS PROCEDURES FOR ORGANICS

6.4.1 AROMATIC VOLATILE ORGANICS - METHOD 8021B

Aromatic volatile organics in water and soil samples are analyzed using Method 8021B, which is a gas chromatography (GC) method using purge and trap sample introduction (Method 5030B/5035). An inert gas is bubbled through a water matrix to transfer volatile aromatic hydrocarbons from the liquid to the vapor phase. Volatile aromatics are collected on a sorbent trap, then flash thermally desorbed and transferred to a GC column. Target analytes are detected using a photoionization detector (PID). Soil samples are extracted by direct purge or with methanol; then an aliquot of sample extract is added to blank reagent water for purge and trap GC analysis.

Positive results are confirmed by GC analysis using a second GC column of dissimilar phase. When second column analysis is performed, peak RTs on both columns must match expected RTs within the calculated RT windows.

Calibration for 8021B - Calibration standards are prepared and analyzed at five concentration levels. A linear calibration curve is developed for each analyte of interest. If the RSD is less than 20% for that analyte, an average response factor may be calculated and used for sample concentration. Otherwise, a linear curve function is used (with a calibration correlation coefficient that is ≥ 0.995) to calculate analyte concentrations. Each working day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by $\pm 15\%$ in order for the run sequence to continue.

6.4.2 ORGANOCHLORINE PESTICIDES AND PCBS - METHOD 8081A/8082 AND THE CLP SOW

Organochlorine pesticides and PCBs are analyzed by gas chromatography following either methods 8081A/8082 or the CLP Organic SOW. Each of these analyses involves solvent

extraction of the sample followed by analysis by gas chromatography with electron capture detection (GC-ECD).

Confirmatory techniques, such as a dissimilar analytical column, must be used to confirm the tentative identification of a peak. For an analyte to be considered confirmed, the peak RTs on both columns must match the expected RTs. For analysis by CLP protocol, the results are flagged with a "P" if the two quantitations differ by more than 25 %. In addition, the breakdown of 4,4'-DDT and endrin is monitored. If the breakdown of either of these compounds is found to be > 15%, the analytical sequence must be discontinued. For analysis by CLP protocol, the combined breakdown must not exceed 30 %.

Calibration for Methods 8081A/8082: Calibration standards are prepared and analyzed at five concentration levels for single component pesticides. A single low-point (level 1) standard is analyzed for multicomponent standards (a five point curve is acquired if any patterns are detected). A linear calibration curve not forced through the origin is developed for each analyte of interest. This function is used for the calibration curve if the RSD is less than 20% for that analyte. Otherwise, a curve function is used that meets this criterion. Each working day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by <15% in order for the run sequence to continue.

Calibration for the CLP SOW - Calibration and analysis are performed in strict accordance with the CLP Organic SOW.

6.4.3 ORGANOCHLORINE HERBICIDES - METHOD 8151A

Method SW8150 is a GC method for determining selected chlorinated acid herbicides. The esters are hydrolyzed and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to esters. The esters are determined by GC employing an electron capture detector. The results are

reported as the acid equivalents. Any compounds identified tentatively in the primary analysis are confirmed on a second GC column.

Calibration for Method 8151A - Calibration procedures for this method is very similar to those described above for methods 8081A/8082. The exceptions are that there are no multi-component herbicides, so separate calibrations for these types of materials are not required. Additionally, herbicides are not susceptible to breakdown when injected into the GC instrumentation, therefore there is no requirement to check for analyte breakdown.

6.4.4 ORGANONITROGEN/ORGANOPHOSPHOROUS PESTICIDES - METHOD 8141A

Method 8141A is a GC method used to determine the concentrations of various organophosphorus pesticides. This analytical method involves extraction of the samples. An aliquot of the extract is then injected into a GC, and compounds in the GC effluent are detected with a flame photometric or nitrogen-phosphorus detector. Any compounds identified tentatively in the primary analysis are confirmed on a second GC column.

Calibration for Method 8141A - Calibration procedures for this method is very similar to those described above for methods 8081A/8082. The exceptions are that there are no multi-component ONOP pesticides, so separate calibrations for these types of materials are not required. Additionally, pesticides are not susceptible to breakdown when injected into the GC instrumentation, therefore there is no requirement to check for analyte breakdown.

6.4.5 POLYNUCLEAR AROMATIC HYDROCARBONS - METHOD 8310

Method SW8310 is used to determine the concentration of ppb levels of selected polynuclear aromatic hydrocarbons (PAHs) in groundwater and soils by HPLC. Samples are extracted then analyzed by direct injection. Detection is by ultraviolet and fluorescent detectors.

Calibration for Method 8310 - Calibration standards are prepared and analyzed at a minimum of five concentration levels. Calibration data for each analyte will be collected from a specific detector (fluorescence or UV-Vis), and this same detector will be used for sample quantitation as well. A linear calibration curve is developed for each analyte of interest. If the RSD is less than 20% for that analyte, an average response factor may be calculated and used for sample concentration. Otherwise, a linear curve function is used (with a calibration correlation coefficient that is ≥ 0.995) to calculate analyte concentrations. Each working day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by <15% in order for the run sequence to continue.

6.4.6 VOLATILE ORGANICS - METHOD 8260B AND THE CLP SOW

Samples may be analyzed for volatile organics by gas chromatography/mass spectrometry (GC/MS) following the procedure described in Method 8260B or the CLP Organic SOW. Analyte identification and quantitation are accomplished using response factors and retention times generated from a (minimum) five-point calibration curve, relative to the closest eluting internal standard. The three internal standards used for these methods are:

- Pentafluorobenzene
- 1,4-Dichlorobenzene-d4
- 1,4-Difluorobenzene
- Chlorobenzene-d5

If requested by the client, non-target analytes are reported as tentatively identified compounds (TICs), but only when an acceptable match is obtained between the spectrum of the analyte and a spectrum found by library search. Unidentified TICs are labeled "unknown". The TICs are quantitated using assumed response factors of 1 relative to the nearest eluting internal standards.

Instrument Performance Check - The mass spectrometer is tuned daily and after every 12 hours of operation to yield an acceptable spectrum for bromofluorobenzene (BFB).

Relative ion abundance criteria for BFB are given in Table 6-1.

Calibration for Method 8260B - After passing the instrument performance check criteria and prior to analyzing samples, a 5-point initial calibration is performed. From that calibration, the calibration check compounds (CCCs) must meet the RSD criteria of <30% and the system performance check compounds (SPCCs) must meet the minimum RRF criteria given in the method. A continuing calibration standard is analyzed after every 12 hours of operation. In that standard, the CCC compounds must meet the %D criteria of <20% and SPCC compounds must meet the minimum RRF criteria listed in the method.

Calibration for the CLP SOW - After passing the instrument performance check criteria and prior to analyzing samples, a 5-point initial calibration is performed. From that

Table 6-1 BFB KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40 % of mass 95
75	30 to 60 % of mass 95
95	Base peak, 100 % relative abundance
173	Less than 2 % of mass 174
174	Greater than 50 % of mass 95
175	5 to 9 % of mass 174
176	Greater than 95 % but less than 101 % of mass 174
177	5 to 9 % of mass 176

Table 6-2 DFTPP KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30 to 60 % of mass 198
68	< 2 % of mass 69
70	< 2 % of mass 69
127	40 to 60 % of mass 198
197	< 1 % of mass 198
198	Base peak, 100 % relative abundance
199	5 to 9 % of mass 198
275	10 to 30 % of mass 198
365	> 1 % of mass 198
441	Present but less than mass 443
442	> 40 % of mass 198
443	17 to 23 % of mass 442

calibration, the compounds listed in Table 2 of Exhibit D, Section IV (VOA) of the CLP SOW must meet the RSD criteria of <20.5% and the minimum RRF criteria listed in the method. A continuing calibration standard is analyzed after every 12 hours of operation. In that standard, the Table 2 compounds must meet the %D criteria of <25% and the minimum RRF criteria listed in the method.

6.4.7 SEMIVOLATILE ORGANICS - METHOD 8270C AND THE CLP SOW

Semivolatile extracts are analyzed by gas chromatography/mass spectrometry following Method 8270C or the CLP Organic SOW. All samples are prepared following extraction methods described in the applicable protocol. Identification and quantitation is performed using response factors and retention times generated from a (minimum) five-point calibration curve, relative to the closest eluting of six internal standards. The six internal standards are:

- 1,4-Dichlorobenzene-d4
- Naphthalene-d8
- Acenaphthene-d10
- Phenanthrene-d10
- Chrysene-d12
- Perylene-d12

If requested by the client, non-target analytes are reported as tentatively identified compounds (TICs), but only when an acceptable match is obtained between the spectrum of the analyte and a spectrum found by library search. Unidentified TICs are labeled "unknown". The TICs are quantitated using assumed response factors of 1 relative to the nearest eluting internal standards.

Instrument Performance Check - The mass spectrometer is tuned daily and after every 12 hours of operation to give an acceptable spectrum for DFTPP. DFTPP ion abundance criteria are given in Table 6-2.

Calibration for Method 8270C - After passing the instrument performance check criteria and prior to analyzing samples, a 5-point (minimum) initial calibration is performed. From that calibration, the CCC compounds must meet the RSD criteria of <30% and the SPCC compounds must meet the minimum RRF criteria given in the method. A continuing calibration standard is analyzed after every 12 hours of operation. In that standard, the CCC compounds must meet the %D criteria of <30% and SPCC compounds must meet the minimum RRF criteria listed in the method.

Calibration for the CLP SOW - After passing the instrument performance check criteria and prior to analyzing samples, a 5-point initial calibration is performed. From that calibration, the compounds listed in Table 5 of Exhibit D, Section IV (SV) of the CLP SOW must meet the RSD criteria of <20.5% and the minimum RRF criteria listed in the method. A continuing calibration standard is analyzed after every 12 hours of operation. In that standard, the Table 5 compounds must meet the %D criteria of <25% and the minimum RRF criteria listed in the method.

6.4.8 PURGEABLE PETROLEUM HYDROCARBONS - METHOD 8015M

Gasoline and volatile aromatic compounds, including benzene, toluene, ethylbenzene, and the xylenes (BTEX), are analyzed by a modified method 8015M using the direct purge technique described above for Method 5030B/5035. Analysis is performed on a GC equipped with a photoionization detector (PID) and a flame ionization detector (FID) connected in series. If BTEX compounds are found without the associated presence of gasoline, confirmation analysis is performed with a second GC column of dissimilar phase and retention characteristics in accordance with the requirements of Method 8021B.

Calibration for 8015M- Calibration standards are prepared and analyzed at five concentration levels. A linear calibration curve is developed for each analyte of interest. If the RSD is less than 20% for that analyte, an average response factor may be calculated and used for sample concentration. Otherwise, a linear curve function is used (with a calibration correlation coefficient that is ≥ 0.995) to calculate analyte concentrations. Each working

day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by <15% in order for the run sequence to continue.

6.4.9 EXTRACTABLE PETROLEUM HYDROCARBONS - METHOD 8015M

Aqueous samples analyzed for diesel, kerosene, jet fuel, and motor oil are prepared using a separatory liquid/liquid microextraction. Solid samples are processed with a liquid-solid extraction utilizing a wrist action shaker extraction (similar to the California LUFT method). One liter of water or 30 grams of soil/sludge are extracted and concentrated to a volume of 1 mL. Analysis is performed by a modified method 8015M on a GC equipped with a capillary or megabore column and an FID detector.

Calibration - Calibration standards are prepared and analyzed at five concentration levels. A linear calibration curve not forced through the origin is developed for each analyte of interest. This function is used for the calibration curve if the RSD is less than 20% for that analyte. Otherwise, a curve function is used that meets this criterion. Each working day, the calibration is verified with the analysis of a continuing calibration standard at the beginning and end of the run sequence and after every 10 analyses. For each analyte of interest, the %D of the response in the continuing calibration standard must agree with the expected response by <15% in order for the run sequence to continue.

6.5 REPRESENTATIVE CALIBRATION AND ANALYSIS PROCEDURES FOR INORGANICS

6.5.1 METALS BY ICP - METHOD 6010B AND THE CLP SOW

These methods describe the simultaneous or sequential determination of metal elements using ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is passed through a plasma torch. Element-specific atomic-line emission spectra are produced which are dispersed by a grating spectrometer and monitored for intensity by photomultiplier tubes.

Calibration for Method 6010B - Prior to the analysis of samples, an initial multipoint calibration is performed for all elements of interest. The initial calibration is checked with an initial calibration verification standard (ICV). For each element, the ICV responses must agree with the initial calibration within $\pm 10\%$ for the calibration to be verified. Following the ICV and after the analysis of every 10 samples, a continuing calibration verification standard (CCV) is analyzed. The response for each element in the CCV must agree within $+10\%$ of the expected value for the analysis to continue.

Calibration for CLP SOW - The calibration procedure for ICP is detailed in Exhibit D, Section IV, Part A of the Inorganic CLP SOW ILM04.0.

6.5.2 METALS BY GFAA - METHODS 7000 SERIES AND THE CLP SOW

Graphite furnace atomic absorption (GFAA) techniques may be used for the determination of various metals including arsenic, lead, selenium, and thallium. Following sample digestion, an aliquot of sample is placed in a graphite tube in the furnace, evaporated to dryness, charred, and atomized, where the absorption of light by the atomized metal is monitored with a photomultiplier tube.

Calibration for Methods 7000 Series - Calibration procedures for the GFAA analyses are detailed in the respective methods in SW-846. For the element of interest, a multipoint initial calibration is performed. The calibration correlation coefficient must be >0.995 to be acceptable. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within $+10\%$ in order for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within $+20\%$ in order for the analysis to continue.

Calibration for CLP SOW - The calibration procedure for GFAA metals is detailed in Exhibit D, Section IV, Part B of the Inorganic CLP SOW ILM04.0.

6.5.3 MERCURY BY CVAA - METHODS 7470A, 7471A, AND THE CLP SOW

Cold-vapor atomic absorption (CVAA) techniques are used for the determination of mercury. Sample preparation is specified in the method. Following dissolution, mercury in the sample 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.

Calibration for Methods 7470A and 7471A - The digestion and calibration procedure is detailed in SW-846 and the CLP SOW. Prior to the analysis of samples, a multipoint initial calibration is performed. The calibration correlation coefficient must be >0.995 to be acceptable. The initial calibration is verified by the reanalysis of a calibration standard (ICV) response of the ICV must agree with the expected response within $+5\%$ in order for the calibration to be verified. A CCV check standard prepared from a second source is analyzed following the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within $\pm 20\%$ in order for the analysis to continue.

Calibration for CLP SOW - The calibration procedure for Mercury analysis by CVAA is detailed in Exhibit D, Section IV, Part D of the Inorganic CLP SOW ILM03.0.

6.5.4 TOTAL AND AMENABLE CYANIDE - METHOD 9010A AND THE CLP SOW

These methods are used to determine concentrations of both total cyanide and cyanide amenable to chlorination in aqueous or solid samples. Cyanide, as hydrocyanic acid (HCN), is released by refluxing the sample with strong acid and distilling the HCN into an absorber-scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by UV spectrophotometry.

Calibration for Method 9010A - Prior to sample analysis, a multipoint initial calibration is performed. The calibration correlation coefficient must be >0.995 to be acceptable. The

initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within +15% for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within +15% for analysis to continue.

Calibration for CLP SOW - The calibration procedure for Cyanide by UV spectrophotometry is detailed in Exhibit D, Section IV, Part E of the Inorganic CLP SOW ILM03.0.

6.5.5 ANIONS - METHOD 9056

Method 9056 is used to analyze anions, including chloride, nitrite, nitrate, o-phosphate, fluoride, and sulfate, in aqueous samples by ion chromatography (IC). A volume of sample is injected into the ion chromatograph. The anions of interest are separated and measured using a chromatography system consisting of a guard column, separator column, suppresser device and conductivity detector. Samples must be refrigerated at 4 ° C and analyzed within 48 hours of sample collection if nitrate, nitrite, and/or o-phosphate are analyzed, or within 28 days of sample collection if chloride, fluoride and/or sulfate are analyzed.

Calibration - Prior to sample analysis, a multipoint initial calibration is analyzed. The calibration correlation coefficient must be >0.995 to be acceptable. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within +10% for the calibration to be verified. A CCV check standard is analyzed and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within +10% for analysis to continue.

6.5.6 PH - METHODS 9040B/9045C

These methods are used to measure the pH of aqueous, multiphase and soil samples. The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

Calibration - The pH meter is calibrated with three standard buffer solutions. The reading must be within +0.05 pH units of the true value of each buffer solution.

6.5.7 FILTERABLE RESIDUE - METHOD 160.1

This method is applicable to drinking, surface and saline waters, and domestic and industrial wastes. A well mixed sample is filtered through a glass fiber filter. The residue that passes through the filter is dried and measured gravimetrically.

Calibration - The analytical balance must be checked each day of use with Class S weights. Balance readings must read within +0.001 grams of the true weight.

6.5.8 NON-FILTERABLE RESIDUE - METHOD 160.2

This method is applicable to drinking, surface and saline waters, and domestic and industrial wastes. A well mixed sample is filtered through a glass fiber filter. The residue on the filter is dried and measured gravimetrically.

Calibration - The analytical balance must be checked each day of use with Class S weights. Balance readings must read within +0.001 grams of the true weight.

6.5.9 NITRATE-NITRITE - METHOD 353.2

Method 353.2 is used to determine the concentrations of nitrate and nitrite in aqueous samples. Nitrite concentration is determined by diazotization with sulfanilamide and complexation with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Combined nitrate-nitrite concentration is determined by first carrying out a copper-cadmium reduction step. A filtered sample is passed through a column containing granulated copper and cadmium to reduce nitrate to

nitrite. Nitrate concentration is determined from the difference of the nitrate-reduced nitrite value and the nitrite value. Samples must be preserved with sulfuric acid to pH <2 and refrigerated at 4 °C.

Calibration - Prior to sample analysis, a multipoint initial calibration is analyzed. The calibration correlation coefficient must be >0.995 to be acceptable. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within +15% for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 10 samples. The response of the CCV must agree with the expected value within +15% for analysis to continue.

6.5.10 TOTAL ORGANIC CARBON (TOC) - METHODS 9060 AND 415.1

Methods 9060 and 415.1 are used to determine the concentration of total organic carbon in samples. TOC is analyzed by combustion of organic material in the sample to carbon dioxide, followed by IR detection of the carbon dioxide.

Calibration - The instrument is calibrated by analyzing five standards and a blank. The initial calibration is verified by the analysis of an ICV standard prepared from a source independent of the calibration standards. The response of the ICV must agree with the expected response within +10 % for the calibration to be verified. A CCV check standard is analyzed following the analysis of the ICV and after the analysis of every 20 samples. The response of the CCV must agree with the expected value within $\pm 10\%$ for analysis to continue.

6.5.11 OIL AND GREASE - METHODS 9070/9071A AND 413.1

Methods 9070/9071A and 413.1 are used to determine the concentration of oil and grease in waters and wastes. The aqueous sample is acidified with HCl to pH <2 and extracted with methylene chloride in a separatory funnel. Sample extracts are evaporated to dryness and measured gravimetrically on an analytical balance. Method 9071A is used to prepare solid samples for gravimetric analysis of oil and grease. By this method, solid samples are

Soxhlet extracted with Freon and the extracts are evaporated to dryness and measured gravimetrically on an analytical balance.

Calibration - A balance calibration check is performed at the beginning and end of each analytical sequence with 1 g and 100 g Class S weights. Measurements must agree to within +0.001 g of the true weight.

6.5.12 OIL AND GREASE - METHOD 413.2

This method is used to determine the concentration of oil and grease in waters and wastes. Samples are acidified with HCl to pH <2 and extracted with Freon in a separatory funnel. Sample extracts are analyzed by infrared spectrometry (IR).

Calibration - Prior to sample analysis, a multipoint initial calibration is performed. The calibration correlation coefficient (r) must be >0.995 for the calibration to be acceptable. A continuing calibration standard is analyzed after the analysis of every 10 samples. The continuing calibration must agree with the initial calibration within +20%.

6.5.13 TOTAL RECOVERABLE PETROLEUM HYDROCARBONS (TRPH) - METHOD 418.1

This method is used to determine the concentration of total petroleum hydrocarbons in waters and wastes. The sample is acidified with HCl to pH <2 and extracted with Freon in a separatory funnel. Extracts are shaken with silica gel to remove interferences, then the extracts are analyzed by infrared spectrometry (IR).

Calibration - Prior to sample analysis, a multipoint initial calibration is performed. The calibration correlation coefficient (r) must be >0.995 for the calibration to be acceptable. A continuing calibration standard is analyzed after the analysis of every 10 samples. The continuing calibration must agree with the initial calibration within +20%.

Table 6-3 ANALYTICAL PROTOCOLS

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, October 26, 1984.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846. 3rd edition (1986) and Final Update III (1996), Office of Solid Waste and Emergency Response, U.S. EPA.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, 1979 Revised 1983, U.S. EPA.
- "Methods for the Determination of Inorganic Substances in Environmental Samples," EPA/600/R-93/100, August, 1993.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, SOW 2/88, OLM01.8, 8/91, OLM02.0, and OLM03.0.
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, SOW ILM03.0.
- "Standard Methods for the Examination of Water and Wastewater", 18th Edition, 1992, APHA-AWWA-WPCF.
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society for Testing and Materials, 1987.
- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society for Testing and Materials, 1987.
- "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory - Cincinnati (September 1986).
- New York State Department of Environmental Conservation. Analytical Services Protocol, September, 1989.

7. CALIBRATION PROCEDURES AND FREQUENCY

All instruments and equipment used in the laboratory must follow a well defined calibration routine. Calibration may be accomplished by laboratory personnel using certified reference materials traceable to NIST, EPA, or equivalent certified materials or by external calibration agencies or equipment manufacturers. The discussion presented here is general in nature because the requirements for calibration are instrument (or equipment) and method specific. Details of calibrations can be found in Paragon Standard Operating Procedures, analytical methods, and operations manuals. In addition to the summary calibration information pertaining to general analysis categories contained in the following subsections, Table 7-2 , Table 7-3 and Table 7-4 list detailed calibration information for representative methods and applications most frequently performed at the Paragon laboratory.

7.1 EQUIPMENT LIST

Appendix B of this LQAP lists all major instrumentation currently available at the Paragon Analytics laboratory.

7.2 STANDARDS AND TRACEABILITY

Analytical standards are prepared from pure compounds or nuclides or mixtures of compounds or nuclides or are purchased prepared from reputable vendors, if available. NIST certificates (or equivalent) must be supplied by vendors. They are used to prepare serial dilutions that are used as calibration and spiking standards. Each department is responsible for the preparation, storage and disposal of its standards. The preparation information is recorded into section specific Standards Notebooks. The analyst records all information needed to maintain traceability in the Standards Notebook. Table 7-1 summarizes the standard sources and preparation of calibration solutions used for most methods implemented at the laboratory.

Each standard is given an internal identification number. The preparation of all stock standards shall be documented in a Standards Notebook which is used to record the date of

preparation, the analyst, the source of the reference material, amounts used, final volume,
etc. and the serial

Table 7-1 STANDARD SOURCES AND PREPARATION

<u>Inst. Type</u>	<u>Std. Source(s)</u>	<u>How Received</u>	<u>Storage Cond's</u>	<u>Source/Prep'n from Source</u>	<u>Lab Storage</u>	<u>Preparation Frequency</u>
ICAP	Various	1,000 & 10,000 ppm	Room Temp.	Intermediate (I) Working (W)	Room Temp.	(I) 3 Months (W) 3 Months
GFAA	EM Sci. & Plasma Chem.	1,000 & 10,000 ppm	Room Temp.	Intermediate (I) Working (W)	Room Temp.	(I) 3 Months (W) Daily
CVAA	Various	1,000 ppm	Room Temp.	Intermediate (I) Working (W)	Room Temp.	(I) 3 Months (W) Daily
Ion Chrom.	Various	1,000 ppm	Room Temp.	Intermediate (I) Working (W)	Room Temp.	(I) 3 Months (W) Daily ¹
Cyanide	Various	KCN Solid	Room Temp.	Intermediate (I) Working (W)	Room Temp.	(I) Annually (W) Daily
Infrared	Various	Neat (oil)	Chill (4 °C).	Intermediate (I) Working (W)	Chill (4 °C).	(I) Annually (W) Semiannually
GC Semi-Volatiles	Various	Neat, & Mixed Soln's 100 to 50,000 µg/mL	Freezer ²	Neat (N) Primary (P) Intermediate (I) Working (W)	Chill (4 °C)	5 years (N) Annually (P) Semiannually (All others)
GC Volatiles	Various (Ultra & Supelco)	Neat, & Mixed Soln's 2,000 µg/mL	Freezer	Primary (P) Intermediate (I) Working (W)	Freezer	Semiannually (All)
HPLC	Various	Mixed or Indiv. Soln's 1.0 to 2,000 µg/L	Freezer	Neat (N) Primary (P) Intermediate (I) Working (W)	Chill (4 °C)	5 years (N) Annually (P) Semiannually (I, W)
GC/MS Semivolatiles	Various (Ultra, NSI, Restek)	Internal Std's - 2,000 µg/mL Standards - 200 µg/mL	Freezer	Intermediate (I) Working (W)	Freezer	Semiannually (I) Semiannually (W)
GC/MS Volatiles	Various (Supelco, Restek)	Mixed Soln. 2,000 µg/L	Freezer	Intermediate (I) Working (W)	Freezer	Quarterly (I) Monthly (W)

¹ Initial multi-point calibration and daily one-point calibration standards prepared each day of use.

² Organonitrogen/Organophosphorous analytes' initial storage conditions are at room temperature.

reference number of that stock solution. All standards shall be labeled with the standard serial reference number, with the name, concentration, date of preparation and expiration date of the stock standards. All diluted working standards not consumed during an analytical session shall be labeled fully, including the serial reference number of any stock standard used in its preparation.

For organic and inorganic standards, if no expiration date has been assigned by the manufacturer, then an expiration date of one year from the date of preparation (or the date first opened in the case of sealed ampoules) is reported unless degradation prior to this date is observed. Radiochemical standards are generally assigned expiration dates no longer than five radioactive half-lives; longer or shorter expiration dates may be assigned following consideration of the chemical stability and availability or cost of the material. A short-lived or difficult to obtain standard may be used past five half-lives if there is sufficient data to verify the integrity of the standard.

To help determine if a standard has degraded, one must note inconsistencies. For instance, low recoveries from newly prepared quality control spikes or abnormally low instrument response to a specific standard are indications of possible standard degradation. However, for some standards, degradation is more easily noted. For instance 4,4'-DDT breaks down to form 4,4'-DDD and 4,4'-DDE. In this example, one can discern, on a chromatogram, the degradation of 4,4'-DDT by the increased concentrations of 4,4'-DDD and 4,4'-DDE. If degradation is observed before the default expiration date, it should be noted in the Standard Notebook for that standard and the standard removed from service.

Before standards can be utilized in a calibration curve they must be verified by a secondary means:

- Analysis of an EPA QC Check Sample, or
- Analysis of an independently prepared check standard.

7.3 GENERAL CALIBRATION PROCEDURES

Calibration standards are prepared from materials of the highest available purity. To establish instrument calibration, working standards are prepared from more concentrated working stock solutions. All organic standards are refrigerated or frozen. Inorganic standards are refrigerated as necessary. Radiochemical standards are generally prepared in a matrix which recreates the preservation of the primary (or parent) standard. Data regarding their preparation is recorded in the each laboratory section's Standards Notebook.

For most organic and inorganic analyses, calibration standards are chosen to bracket the expected concentrations of those parameters in the sample and to demonstrate the calibration range of the method and instrument. Samples that fall outside the calibration range are diluted until bracketed by the calibration standards. Calibration standards are prepared typically at a minimum of three concentration levels (usually chosen at three to five times and five to ten times the estimated method detection limit plus a calibration blank) with the exception of most organic analyses which do not require a calibration blank. Either an internal standard or external standard quantitation technique may be utilized. The reporting limit is verified by analyzing a standard at the reporting limit.

Instrumental responses to calibration standards for each parameter are subjected to an appropriate statistical test of fitness (least squares linear regression, quadratic equation, or relative standard deviation of response factors) or as required by the method or LQAP. The calibration must reflect an acceptable correlation of data points or linearity to be acceptable. In cases where the calibration data are outside these criteria, the analyst must reanalyze and/or redilute the calibration standards (meeting the same criteria) and change instrumental conditions as necessary before acquiring sample data.

For analyses which are performed frequently and for which substantial calibration data is available, a complete recalibration is not required each time an analysis is performed providing that the following criterion is met: one calibration standard is analyzed at the beginning of the analysis which may vary from the expected response (based on the initial,

most recent calibration curve) by no more than +25% or as specified by the method, SOP or QAPP. If this criterion is not met, a complete recalibration is necessary.

During the course of analysis, calibration standards are routinely analyzed to ensure that the instrumental response has not changed. Again the criterion stipulated in each method or SOP for expected response is used by the analyst to determine whether the instrument must be recalibrated or the instrument conditions further optimized.

The accuracy of prepared standards is periodically checked by comparison with a standard from an independent source. In addition, a second source standard (initial calibration verification or ICV) is acquired after the initial calibration and the responses of the second source and the calibration standards are compared against one another. Method specific acceptance criteria must be met for this comparison, or the analysis will be halted, the problem identified and corrected, and recalibration of the instrument initiated.

Certain pieces of equipment such as balances, pH meters, and turbidity meters are calibrated daily with NIST traceable standard reference material.

Radiochemical determinations employ two basic calibration types: external and internal calibrations. An instrument is calibrated by external means prior to introduction of the sample to be measured. Homogeneous standards prepared in accurately defined geometries having exact or acceptably equivalent radiometric characteristics of the sample matrix/geometry combination are measured in the instrument to establish response of 'efficiency' factors. The level of activity of the calibration source should be sufficient to minimize uncertainty attributable to background and to counting statistics. The source is then measured under conditions equivalent to that being measured in the analysis.

Wherever feasible, sufficient counts (usually 10000 counts) should be gathered to provide for less than one percent counting uncertainty. Once instrument calibration is established, an instrument quality control chart may be established, and instrument reliability tests performed to demonstrate that the operating or response characteristics of the instrument

have remained within accepted tolerances. Re-calibration should only be necessary in the event that the instrument has malfunctioned, or the results of QC or daily reliability checks indicate that the operating or response characteristics of the instrument have exceeded acceptable tolerances for the use intended. A quality control chart is used to determine if the response of the instrument has changed statistically; the magnitude of the statistical change may or may not be significant when compared to the required accuracy and precision criteria for the overall technique.

Numerous radiochemical techniques employ the use of radioisotopic tracers as internal standards; for example while alpha spectrometric measurement use an external standard to achieve the energy and efficiency calibration of the instrument, actual quantitation requires addition of standard radioisotopic tracers to permit measurement of sample activity using the method of internal standards ratio. The second type of internal calibration involves standard addition of a radioactive tracer or stable carrier to a sample or split of a sample. Often, combinations of external and internal standards, and of stable and radioisotopic tracers, may be employed in one measurement. Specific requirements for calibration is discussed in the specific standard operating procedure for each method.

Background determinations: Because laboratory instrumentation typically operates over several orders of magnitude or more of sample activity, careful control of instrument background is an integral part of the radiochemistry laboratory's control program. A routine program for monitoring of instrumental and extraneous background is conducted to ensure that background values used in the calculation of sample results are representative of the instrumental background during that measurement. Specific requirements for the measurement and application of background measurements are addressed in the specific standard operating procedure for each method.

7.4 RADIOCHEMICAL INSTRUMENT CALIBRATION PROCEDURES

7.4.1 GAS FLOW PROPORTIONAL COUNTER

The minimum operations necessary to satisfy analytical requirements associated with the detection and measurement of gross alpha and beta emissions from appropriate radiochemical sample preparations are listed below. The following operations should be performed periodically in the laboratory and carefully documented:

- Plateau measurement determines operating voltage and discriminator settings
- Initial calibration is conducted for specific methods according to SOP
- Daily reliability checks verify Continuing Calibration
- Daily background measurements verify background integrity and provide data for use in background correction calculations.

Prior to initial calibration of the Gas Flow Proportional Counter, it is necessary to measure operating plateaus and establish optimum operating voltages and discriminator settings for each detector and each instrument. Procedures, frequency, and tolerances for these activities are found in the Instrument Operating Procedure for Proportional Counters and in the respective Operating Manual for the Instrument. Following instrument set-up, several data points are collected for daily response checks ($\alpha + \beta$) and for background counts. The data are used to establish interim operating limits for daily reliability checks (mean \pm 2 and 3 SD). Initial calibrations may then be conducted as described in the specific SOP for each method in question (e.g. Gross Alpha, Gross Beta, Radiostrontium, Radium-228, Total Radium, Carbon-14, Technetium-99, etc.). Daily reliability checks are run daily when the instrument is in use to assess the stability of the instrument prior to analyzing samples. When 20-30 checks have been gathered, final operating limits are established and used to assess the stability of the instrument prior to analyzing samples. Tolerance limits may also be established which may exceed historical operating limits, but are conservative enough to ensure that the most conservative project DQO's are met (usually \pm 15 %).

If the daily checks exceed the tolerance limits listed in the Operating Procedure for Proportional Counters, or if other indications of possible instrument malfunction are noted, analysis of samples is halted until corrective actions have been completed and the daily reliability checks and other appropriate measures verify proper operation of the instrument and the continuity of instrument response. If following corrective actions, it is determined that the instrument response has drifted relative to the point of initial calibration, the daily reliability operating limits and specific method calibrations will be repeated prior to analysis of further samples.

7.4.2 GAMMA SPECTROMETER

The minimum operations necessary to satisfy analytical requirements associated with the detection and measurement of gamma spectral analysis on the high purity germanium gamma spectrometer from appropriate radiochemical sample preparations are listed below. The following operations should be performed periodically in the laboratory and carefully documented:

- Periodic (weekly) background measurements verify background integrity and provide data for use in background correction calculations.
- Initial calibrations are conducted for specific methods according to SOP
- Daily reliability checks verify Continuing Calibration

Prior to initial calibration of the HPGe gamma spectrometer, it is necessary to establish the instrument internal QC database. Procedures, frequency, and tolerances for these activities are found in the Operating Procedure for Gamma Spectrometers and in the respective Operating Manual for the Instrument. 20 data points are collected using a long-lived mixed gamma standard. These data are used to establish instrument internal operating limits for daily reliability checks (mean +/- 2 and 3 SD). Additionally, a long background count is conducted. Initial calibrations may then be conducted as described in the specific SOP. Reliability checks are run daily when the instrument is in use to assess the stability of the instrument prior to analyzing samples.

If the daily checks exceed the tolerance limits listed in the Operating Procedure for Gamma Spectrometers, or if other indications of possible instrument malfunction are noted, analysis of samples is halted until corrective actions have been completed and the daily reliability checks and other appropriate measures verify proper operation of the instrument and the continuity of instrument response. If following corrective actions, it is determined that the instrument response has drifted relative to the point of initial calibration, the daily reliability operating limits and specific method calibrations will be repeated prior to analysis of further samples.

7.4.3 ALPHA SPECTROMETER

The minimum operations necessary to satisfy analytical requirements associated with the detection and measurement of alpha spectral analysis from appropriate radiochemical preparations are listed below. The following operations should be performed periodically in the laboratory and carefully documented:

- Weekly background measurements verify background integrity, monthly background measurements, and provide data for use in background correction calculations.
- Monthly energy and efficiency calibrations are conducted
- Initial calibrations are conducted for specific methods according to SOP
- Daily reliability checks verify Continuing Calibration

7.4.4 LIQUID SCINTILLATION COUNTER

The minimum operations necessary to satisfy analytical requirements associated with the detection and measurement of alpha spectral analysis from appropriate radiochemical preparations are listed below. The following operations should be performed periodically in the laboratory and carefully documented:

- Periodic normalization
- Daily Instrument Performance Assessment (IPA) verify instrument operation and response

- Daily background measurements verify background integrity
- Method specific initial calibrations are conducted for specific methods according to SOP
- Representative backgrounds are counted with each batch of samples and provide the basis for background correction of sample results

7.5 INORGANIC INSTRUMENT CALIBRATION PROCEDURES

7.5.1 INDUCTIVELY COUPLED ARGON PLASMA (ICAP)

ICAP systems are calibrated at least daily using a calibration blank and one working standard (at a minimum). Using linear interpolation, a calibration equation is developed from these data points. This information is compared to historical calibration data to verify that instrument performance is consistent with past capabilities. If this evaluation shows that instrument responses have changed significantly, the analyst will recalibrate the instrument by generating an “initial” calibration curve. An initial calibration curve consists of analyzing three to five working calibration standards which have been prepared from NIST traceable stock solutions. The calibration standards define the working range of the system; sample results that are beyond that range must be diluted into the working range. Immediately following instrument calibration, the calibration is verified by analysis of a second source independent standard which must fall within the acceptance limits prescribed by the method or an investigation and corrective action shall be initiated, including complete recalibration, if necessary. After instrument calibration has been established and independently verified, calibration slope and baseline shall be monitored for drift using amid-range standard (CCV) and a calibration blank (CCB), respectively. The CCV and CCB shall be analyzed at a 10% frequency throughout as well as at the end of the analytical run and must fall within the acceptance limits prescribed by the method, or an investigation and corrective action shall be initiated, including complete recalibration, if necessary.

7.5.2 GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA)

GFAA systems are calibrated each day of use using a calibration blank and three to four working calibration standards which have been prepared from NIST traceable stock

solutions. The calibration standards define the working range of the system; sample results that are beyond that range must be diluted into the working range. Immediately following instrument calibration, the calibration is verified by analysis of a second source independent standard (ICV) which must fall within the acceptance limits prescribed by the method. If this criteria is not met, an investigation and corrective action shall be initiated, including complete recalibration, if necessary.

After instrument calibration has been established and independently verified, calibration slope and baseline shall be monitored for drift using a mid-range standard (CCV) and a calibration blank (CCB), respectively. The CCV and CCB shall be analyzed between every 10 field samples (at a minimum) throughout the analysis run sequence as well as at the end of the analytical run. The results for these QC check samples must fall within the acceptance limits prescribed by the method, or an investigation and corrective action shall be initiated, including complete recalibration if necessary.

7.5.3 COLD VAPOR ATOMIC ABSORPTION

CVAA systems are calibrated at least daily using a calibration blank and a minimum of five working calibration standards which have been prepared from NIST traceable stock solutions. The calibration standards define the working range of the system; sample results that are beyond that range must be diluted into the working range. Immediately following instrument calibration, the calibration is verified by the reanalysis of a calibration standard which must fall within the acceptance limits prescribed by the method or an investigation and corrective action shall be initiated, including complete recalibration, if necessary. Verification of the accuracy of preparation of the calibration standards will be done by analyzing a second-source reference standard (CCV), which will also be used to assess instrument/detector drift during the analysis sequence.

After instrument calibration has been established, calibration slope and baseline shall be monitored for drift using amid-range standard (CCV) and a calibration blank (CCB), respectively. The CCV and CCB shall be analyzed at a 10% frequency throughout as well

as at the end of the analytical run and must fall within the acceptance limits prescribed by the method, or an investigation and corrective action shall be initiated, including complete recalibration, if necessary.

7.5.4 ION CHROMATOGRAPHY

The ion chromatograph (IC) is initially calibrated at using a calibration blank and five working calibration standards which have been prepared from NIST traceable stock solutions. The calibration standards define the working range of the system; sample results that are beyond that range must be diluted into the working range. Immediately following instrument calibration, the calibration is verified by analysis of a second source independent standard (ICV) which must fall within the acceptance limits prescribed by the method. If this criteria is not met, an investigation and corrective action shall be initiated, including complete recalibration, if necessary.

Daily calibration of the IC instrument is performed by analyzing a calibration blank and a high-level calibration verification standard (ICV). Using the calibration curves generated earlier, the concentrations of the target analytes in the ICV, which are compared to the known concentrations. ICV concentrations calculated in this manner must agree to within 10 % of their target values for the analysis to proceed. If this acceptance criteria is not met, an investigation and corrective action shall be initiated, including complete recalibration, if necessary.

After instrument calibration has been established and independently verified, instrument stability shall be monitored for drift using a mid-range standard (CCV) and a calibration blank (CCB), respectively. The CCV and CCB shall be analyzed between every 10 field samples (at a minimum) throughout the analysis run sequence as well as at the end of the analytical run. The results for these QC check samples must fall within the acceptance limits prescribed by the method, or an investigation and corrective action shall be initiated, including complete recalibration if necessary.

7.6 ORGANIC INSTRUMENT CALIBRATION PROCEDURES

7.6.1 GAS CHROMATOGRAPHY

Initially, a three or five point calibration curve, consisting of all compounds of interest plus a calibration blank, is established to define the usable range of the instrument. Calibration may be accomplished as best-fit line, quadratic equation, or average RF. The curve is determined to be linear if the correlation coefficient is > 0.995 . Linearity may also be determined using response factors. Response factors are calculated for each compound at each concentration level. These RF will be averaged to generate the mean RF for each compound over the range of the standard curve. The curve is determined to be linear if the RSD of the response factors is $< 20\%$ or as specified by the method. The mean response factor will be used to calculate the sample concentration of the compound of interest.

When sample responses exceed the range of the standard curve, the sample will be diluted to fall within the range of the standard curve and be reanalyzed. The results of the daily gas chromatography standardization will be tabulated and filed with the corresponding sample analyses. Daily full calibration is not necessary if a calibration check standard validates the initial calibration curve. If the response to a calibration check standard differs from the initial calibration by more than $+15\%$ for any analyte being quantitated or as specified by the method, then investigation and corrective action will be performed, including complete recalibration, if necessary.

7.6.2 GAS CHROMATOGRAPHY/MASS SPECTROMETRY

The minimum operations necessary to satisfy analytical requirements associated with the determination of organic compounds in water and soil/sediment samples are listed below.

The following operations should be performed routinely in the laboratory:

- Documentation of GC/MS mass calibration and abundance pattern
- Documentation of GC/MS response factor stability
- Internal standard response and retention time

Prior to initiating data collection, it is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP) for base/neutral and acid (BNA) compounds or p-bromofluorobenzene (BFB) for volatile compounds. The ion abundance criteria for each calibration compound must be met before any samples, blanks, or standards can be analyzed.

Each GC/MS system used for the analysis of semivolatile organic compounds by EPA methods must be tuned to meet method or program specific abundance criteria. This criteria must be demonstrated as required in the appropriate Paragon semivolatile SOPs.

Each GC/MS system used for the analysis of volatile organic compounds by EPA methods must be tuned to meet method or program specific abundance criteria. The criteria must be demonstrated as required in the appropriate Paragon volatiles SOPs.

Prior to the analysis of samples and after tuning criteria have been met, the GC/MS system must be initially calibrated with a minimum of five concentrations of each compound being analyzed to determine the linearity of response. USEPA and/or QAPP criteria may specify both the concentration levels for initial calibration and the specific internal standard to be used on a compound-by-compound basis for quantitation. The response factor (RF) for each compound at each concentration level is calculated using the following equation:

$$R_f = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

Where:

A_x = area of the characteristic ion for the compound to be measured.

A_{is} = area of the characteristic ion for the specific internal standards.

C_{is} = concentration of the internal standard (ng/ μ L).

C_X = concentration of the compound to be measured
(ng/ μ L).

Using the RF from the initial calibration, the percent relative standard deviation (%RSD) for compounds identified as Calibration Check Compounds (CCC) is calculated using the following equation :

$$\% RSD = \frac{S}{\bar{X}} \times 100$$

Where:

RSD = relative standard deviation

S = standard deviation of initial five response factors (per compound).

\bar{X} = mean of initial five response factors (per compound).

The %RSD for each individual CCC should be less than 30% for volatile organics and less than 30% for semivolatile organics or as specified by the method. This criteria must be met for the initial calibration to be valid.

A calibration check standard containing all compounds of interest as well as all required surrogates is analyzed each day of analysis. The RF data from the standard is compared each day against the average RF from the initial calibration for a specific instrument. If the response to a calibration check standard differs from the initial calibration by more than $\pm 25\%$ for volatile organics and $\pm 30\%$ for semivolatile organics (or as specified by the method), then investigation and corrective action must be performed including a complete recalibration if necessary before samples are analyzed.

7.6.3 HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

Initially, a five point calibration curve, consisting of all compounds of interest plus a calibration blank, is established to define the usable range of the instrument. Calibration may be accomplished as best-fit line, quadratic equation, or average RF. The curve is determined to be linear if the correlation coefficient is > 0.995 . Linearity may also be

determined using response factors. Response factors are calculated for each compound at each concentration level. These RF will be averaged to generate the mean RF for each compound over the range of the standard curve. The curve is determined to be linear if the RSD of the response factors is <20% or as specified by the method. The mean response factor will be used to calculate the sample concentration of the compound of interest.

When sample responses exceed the range of the standard curve, the sample will be diluted to fall within the range of the standard curve and be reanalyzed. The results of the daily HPLC standardization will be tabulated and filed with the corresponding sample analyses. Daily full calibration is not necessary if a calibration check standard validates the initial calibration curve. If the response to a calibration check standard differs from the initial calibration by more than +15% for any analyte being quantitated or as specified by the method, then investigation and corrective action will be performed, including complete recalibration, if necessary.

7.7 MISCELLANEOUS INSTRUMENT CALIBRATION PROCEDURES

7.7.1 ANALYTICAL BALANCES

Every 12 months, calibration of the entire analytical range shall be checked by a qualified service technician. The calibration of each balance is checked each day of use using weights traceable to the National Institute of Standards and Technology (NIST).

Calibration weights are Class S or better and are recertified every two years. If balances are calibrated by an external agency, verification of their weights shall be provided. All information pertaining to balance maintenance and calibration is found in the individual balance logbook.

7.7.2 pH/ELECTROMETER

The meter is calibrated before use each day and once after each four hours of continuous use. At least three fresh buffer solutions are used to calibrate the meter.

7.7.3 UV/VISIBLE/INFRARED SPECTROPHOTOMETER

During use, spectrophotometer performance is checked at established frequencies in analysis sequences against CCVs and ICVs. The instrument operating capability is also evaluated annually by an outside instrument maintenance service.

7.7.4 TOTAL ORGANIC CARBON ANALYZER

The TOC instrument is calibrated daily by analyzing five calibration standards plus a blank. Instrument responses versus concentrations are modeled using a linear calibration curve algorithm. Acceptance criteria for the calibration curve is that the correlation coefficient must be $\geq .995$. If this criteria is passed, the analysis may proceed. If this criteria is not met, the analysis is halted, the problem of poor curve fit investigated and corrected, and the instrument recalibrated.

After an acceptable calibration curve is generated, an independent calibration verification solution (second source) is analyzed. Using the daily calibration curve, the TOC value of the calibration verification solution is compared to the target value. The second source verification solution's calculated concentration must be within 20% of the expected (target) value. If this criteria is met, sample analysis may proceed. If this criteria is not met, the analysis is halted, the problem investigated and corrected, and the instrument recalibrated.

7.7.5 TITRIMETRIC/ELECTROMETRIC METHODS

For titrimetric analyses (such as alkalinity, sulfide, etc.), titrant solutions are prepared from commercially-available reagents. These solutions are prepared initially to target concentrations (or normalities) that the laboratory staff has determined are analytically useful over a wide range of expected sample concentrations for the target analyte. High-purity standardization reference materials are then used to accurately establish the normality of the titrant solution. This is done by performing the standardization titration in triplicate. The results of the triplicate titration are then averaged, and this value is used to calculate the actual concentration of the titrant.

With each analysis batch, a reference material will be prepared in the same manner as was used to establish the initial normality of the titrant. This check sample (also called a Laboratory Control Sample or LCS) will be analyzed with the field samples. Acceptance criteria is established for all methods used by the laboratory, and this criteria must be met. If the acceptance criteria is not met, the analysis must be halted, the source of the problem located and corrected, and the analysis restarted.

7.7.6 FLASHPOINT TESTER CALIBRATION

The instrument is calibrated against the flashpoint of p-xylene, which is 27 ° C. The observed reading from the flashpoint tester should be between 26.0 ° C and 28.0 ° C. If this criteria is not met, check the condition and operation of the apparatus, especially the tightness of the lid, the action of the shutter, and the position of the test flame. After adjustment, repeat the test with the p-xylene standard. If the acceptance criteria for the p-xylene is still not met, replace the instrument's thermometer with another calibrated thermometer and repeat the p-xylene calibration procedure.

7.8 OVEN TEMPERATURE MONITORING

All ovens in use in the laboratory will use a calibrated laboratory thermometer to monitor temperature. The thermometers will be placed in such a fashion so as to continuously monitor the interior temperature of the oven. Personnel using the ovens will monitor its temperature, and will ensure that a constant temperature is achieved before using it. For ovens operating in a temperature range of 40 ° C to 200 ° C, oven temperature must be able to remain within a range of ± 5 ° C around the setpoint. For ovens whose operating range is above 200 ° C, the stability acceptance criteria is ± 20 ° C. If the oven is unable to maintain a stable temperature within the required range, the oven should be placed out of service and the Department Manager to which the unit is assigned notified that the oven needs service.

7.9 SAMPLE REFRIGERATOR MONITORING

Sample storage refrigerators will be equipped with a calibrated thermometer that will monitor the interior temperature of the storage unit. During every day of business, a temperature reading will be made in the refrigerator, and the results of the measurement will be entered on a refrigerator temperature log. The date the reading was made, the actual reading and the initials of the person making the reading will be entered on the temperature log. For environmental samples that must be chilled for proper preservation, the storage units must maintain a temperature of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. If the temperature reading is above or below the criteria, the Department Manager to which the unit is assigned notified that the refrigerator has exceeded the acceptance criteria.

7.10 THERMOMETER STANDARDIZATION

Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are recertified every three years with equipment directly traceable to the NIST.

Working thermometers are compared with the reference thermometers every 12 months. Each thermometer is tagged and individually numbered. In addition, working thermometers are visually inspected by laboratory personnel prior to use.

Calibration temperatures and acceptance criteria are based upon the working range of the thermometer and the accuracy required for its use. Laboratory thermometer inventory and calibration data are maintained by the QA department.

Table 7-2 SUMMARY OF RADIOCHEMISTRY CALIBRATION REQUIREMENTS

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
Gas Flow Proportional Counter	Plateau and Initial Set-up; and Operating Voltage and Discriminator Settings	Slope <2.5%/100V a->b crosstalk: <50% b-> a crosstalk: 0.1 - 2%	1) Recount plateau, reset operating voltage, and/or discriminator 2) Determine cause, repair, repeat set-up
	Daily background check	a < 0.5 cpm b < 2.5 cpm	1.) Clean planchet, sample holder, recount 2.) determine cause, repair, repeat measurement, repeat set-up
	Daily Reliability Check Am-241, Sr/Y-90	± 5% rel./ monitor historical performance for trends	1) Repeat Check 2) Determine cause, repair, repeat check, repeat set-up
	Initial Method Specific Calibration	Meets Criteria in SOP	1) Consult with supervisor
HPGe Gamma Spectrometer	Energy and Shape Calibration:	To manufacturer's specifications	1) Check Detector bias, cables, 2) Determine cause, repair, Pole-zero, recalibrate, repeat calibration
	Efficiency Calibration	Meets criteria in SOP	1) Consult with supervisor
	Monthly long background check w/ empty chamber	To meet sensitivity requirements	1) Clean chamber, detector, recount 2) Determine cause, repair, repeat measurement
	Daily Reliability Check, Mixed Standard	± 10% rel./monitor historical performance for trends	1) Repeat check 2) Determine cause, repair, repeat check, repeat set-up
	Initial specific geometry calibration	Meets criteria in SOP	1) Consult with supervisor
Alpha Spectrometer	Weekly Energy and Efficiency:	± 50 keV and ± 20% relative	1) Repeat measurement

Table 7-2 SUMMARY OF RADIOCHEMISTRY CALIBRATION REQUIREMENTS

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
	Mixed Alpha Source		2) Determine cause, repair 3) Repeat measurement
	Quarterly Pulser Check	FWHM < 50 keV	1) Repeat measurement 2) Determine cause, repair 3) Repeat measurement
	Weekly long background check	To meet sensitivity requirements	1) Clean chamber, detector, recount 2) Determine cause, repair, repeat measurement
	Daily Vacuum Check	< 0.1 torr	1) Check/replace seals 2) Locate and repair leak 3) Call service
Liquid Scintillation Counter	Periodic normalization and: Daily Instrument Performance Assessment	Within established control limits	1) Repeat measurement 2) Determine cause, repair 3) Call service
	Daily background check	Within established control limits	1) Determine cause, repair 2) Call service
	Initial specific calibration	Meets criteria in SOP	1) Consult with supervisor
	Representative backgrounds with each batch	Within established control limits	1) Repeat measurement 2) Prepare new bknd sample 3) Call service

Table 7-3 SUMMARY OF GC AND GC/MS CALIBRATION REQUIREMENTS

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
GC/MS (8270)	Tune: DFTPP	Meets Criteria	Re-tune instrument Repeat DFTPP analysis
	Initial: 5 level (20, 50, 80, 120, 160 ppb)	RSD < 30% for RF's	Repeat Calibration
	Daily: 1 level (50 ppb) (every 12 hours)	RF \geq 0.050 (SPCC)	Evaluate system Repeat Calibration
		% Difference <30% of the average five-point RF (CCC)	Evaluate system Repeat Calibration
GC/MS (8240)	Tune: BFB	Meets Criteria	Re-tune instrument Repeat BFB Analysis Repeat Calibration
	Initial: 5 level (10, 20, 50, 100, 200 ppb)	RSD < 30% for RF's	Repeat Calibration
	Daily: 1 level (50 ppb) (every 12 hours)	RF \geq 0.300 (0.250 for bromoform) (SPCC)	Evaluate system Repeat Calibration
		% Difference <25% of the average five-point RF (CCC)	Evaluate system Repeat Calibration
Gas Chromatograph (8080)	Initial: 5 level (concentration based)	Std curve or calibration factor (CF)	Make new standards or establish new calibration

Table 7-3 SUMMARY OF GC AND GC/MS CALIBRATION REQUIREMENTS

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
	upon instrument response)	%RSD \leq 20%	curve
	Mid-level DDT/Endrin standard	DDT/Endrin breakdown < 20%	Reanalyze standard once Perform system maintenance
	Daily: 1 level of check standard (mid-range)	CF \pm 15% of initial calibration	Repeat initial calibration
	Standard check every 10 samples	CF \pm 15% of initial calibration Retention times within RT windows	Reanalyze all samples since last acceptable check standard
Gas Chromatograph (8150)	Initial: 5 level (concentration based upon instrument response)	Std curve or calibration factor (CF) %RSD \leq 20%	Make new standards or establish new calibration curve
	Daily: 1 level of check standard (mid-range)	CF \pm 15% of initial calibration	Repeat initial calibration
	Standard check every 10 samples	CF \pm 15% of initial calibration Retention times within RT windows	Reanalyze all samples since last acceptable check standard
High Performance Liquid Chromatograph (8310)	Initial: 5 level (concentration based upon instrument response)	Std curve or calibration factor (CF) %RSD \leq 20%	Make new standards or establish new calibration curve
	Daily: 1 level of check standard (mid-range)	CF \pm 15% of initial calibration	Repeat initial calibration
	Standard check every 10 samples	CF \pm 15% of initial calibration Retention times within RT windows	Reanalyze all samples since last acceptable check standard

Table 7-4 SUMMARY OF INORGANIC CALIBRATION REQUIREMENTS

Instrument	Calibration Standards Used, Initial and Daily Minimum	Acceptance Limits	Corrective Actions
Ion Chromatograph (9056)	Initial: Blank + 5 levels	Linear or Polynomial	Recalibrate system
	Daily: 1 level of check standard (mid-range)	ICV 90% - 100% of target value	1) Reanalyze ICV once 2) Regenerate multi-point calibration curve
	Continuing Calibration Verification (CCV) between every 10 samples	CCV 90% - 100% of target value	1) Reanalyze CCV once 2) Recalibrate and reanalyze all samples after last acceptable CCV
ICAP (6010)	Initial: Blank + 3 standards	Calibration Verification Standard: 95% - 105% of true value	Recalibrate system
	Daily: Initial Calibration Verification (ICV)	ICV: 90% - 110% of true value	Recalibrate system
	Continuing Calibration Verification (CCV) between every 10 samples	CCV: 90% - 110% of true value	1) Reanalyze CCV once 2) Recalibrate and reanalyze all samples after last acceptable CCV
GFAA (7000 - series methods)	Multi-point calibration of four or more calibration standards plus calibration blank	Correlation coefficient ≥ 0.995	Make new standards or establish new calibration curve
	Initial Calibration Verification (ICV)	ICV: 90% - 110% of true value	Make new standards or establish new calibration curve
GFAA (C)	Continuing Calibration Verification	CCV: 80% - 120% of true value	1) Reanalyze CCV once

Table 7-4 SUMMARY OF INORGANIC CALIBRATION REQUIREMENTS

Instrument ontinued)	Calibration Standards Used, Initial and Daily Minimum (CCV) between every 10 samples	Acceptance Limits	Corrective Actions
			2) Recalibrate and reanalyze all samples after last acceptable CCV
pH Meter (9040/9045)	Daily: 3 pH buffer levels	± 0.05 pH units of target value	Clean or replace electrode; recalibrate
	Acid/Base check solutions between every 10 samples	± 0.05 pH units of target value	Clean or replace electrode; recalibrate
UV-Vis Spectrophotometer	Daily: five standards plus blank	Correlation coefficient ≥ 0.995	Recalibrate system
	Initial Calibration Verification (ICV)	ICV: 90% - 110%	1) Reanalyze ICV once 2) Recalibrate system
	Mid-range check standard between every 10 samples	CCV: 90% - 110%	1) Reanalyze CCV once 2) Recalibrate and reanalyze all samples after last acceptable CCV
CVAA (7000 - series methods)	Multi-point calibration of four or more calibration standards plus calibration blank	Correlation coefficient ≥ 0.995	Make new standards or establish new calibration curve
	Initial Calibration Verification (ICV)	ICV: 90% - 110% of true value	Make new standards or establish new calibration curve
	Continuing Calibration Verification (CCV) between every 10 samples	CCV: 80% - 120% of true value	1) Reanalyze CCV once 2) Recalibrate and reanalyze all samples after last acceptable CCV

8. PREVENTIVE MAINTENANCE

The objective of Paragon's preventive maintenance program is to establish a system of instrument care that prevents the loss of analytical quality control and results in a minimum of lost productivity due to instrument failure. This program includes a system for documenting all routine and non-routine instrument maintenance and repairs. Analysts maintain calibration and maintenance records of all equipment and instruments that generate analytical data. Paragon maintains service contracts for most major analytical equipment including: gas chromatographs, mass spectrometers, balances, atomic absorption spectrometers, and inductively coupled plasma spectrometers.

8.1 MAINTENANCE RESPONSIBILITIES

The Department Manager is responsible for providing technical leadership to all staff involved with chemical analysis. This leadership role includes: (1) serving as a technical resource to help solve equipment and method problems; (2) evaluating and recommending investments in new technologies; (3) improving efficiency; and (4) coordinating instrument repair and maintenance.

The primary responsibility for the maintenance of instruments and equipment rests with each analytical Department Manager. The Department Manager is further responsible for developing procedures and schedules for maintaining each major instrument or piece of equipment and for delegating specific maintenance responsibilities to department staff.

8.2 MAINTENANCE DOCUMENTATION

All routine and non-routine instrument maintenance is documented in maintenance logbooks or in run sequence logbooks assigned to each instrument. To provide a clear and complete history of repairs and maintenance associated with each instrument, each entry must include the following elements:

- The reason for the maintenance or repair (e.g., was this action taken to fix a problem or was this action routine instrument maintenance);
- A full description of the maintenance or repair;

- A description of how the analyst demonstrated that the analytical system was operating in control after completion of the maintenance and *before* the resumption of sample analysis; and
- The initials of the analyst making the entry, date of entry, and the dates that maintenance actions were performed.

8.3 MAINTENANCE SCHEDULES

The effectiveness of the maintenance program relies heavily on adherence to prescribed schedules for maintaining each instrument or piece of equipment. A schedule is established for all routine maintenance. Other maintenance activities may also be identified as requiring attention on an as-needed basis. Manufacturers' recommendations provide the primary basis for developing these schedules, and manufacturers' service contracts provide primary maintenance for several major instruments (e.g., spectrophotometers, gas chromatographs, analytical balances, etc.).

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument.

SOPs are written for each instrument that cover basic operation and maintenance procedures. The following are brief summaries of maintenance for each major instrument. This information is also listed by major instrumentation system in Table 8-1.

8.4 RADIOCHEMICAL INSTRUMENT MAINTENANCE PROCEDURES

8.4.1 GAS FLOW PROPORTIONAL COUNTER

Regularly performed maintenance includes, but is not limited to, the following for gas flow proportional counters:

- Maintenance of the P-10 gas supply;
- Periodic background checks;
- Periodic checks of detector stability (efficiency and cross-talk);
- Detector plateau determinations;

- Operating bias adjustment;
- Discriminator adjustment;
- Inspection and cleaning of slide and sample holders;
- Inspection and renewal of detector windows;
- Inspection and adjustment of electrical connections;
- Cleaning and checks of system components;
- Checks, adjustment and repair of automatic sample changer components;
- Maintenance of software, analysis protocols and system backup.

8.4.2 HIGH PURITY GERMANIUM GAMMA SPECTROMETERS

Regularly performed maintenance includes, but is not limited to, the following for HPGe spectrometers:

- Maintain Dewar liquid nitrogen level;
- Periodic resolution checks;
- Periodic checks of detector stability (energy and efficiency);
- Pole-zero adjustment and processing adjustments;
- Inspect and clean detector chamber;
- Periodic long background counts;
- Power supply and NIM bin checks;
- Clean and check system components;
- Check and adjust electrical connections;
- Recondition detector as needed (pump and bake);
- Maintenance of software, analysis protocols and backup data files.

8.4.3 SURFACE BARRIER ALPHA SPECTROMETER

Regularly performed maintenance includes, but is not limited to, the following for alpha spectrometers:

- Periodic checks of system stability (efficiency and energy);
- Periodic energy calibration;

- Periodic efficiency calibration;
- Periodic checks of system integrity (pulser checks);
- Detector background counts;
- Detector maintenance and cleaning;
- Inspection and cleaning of sample holders;
- Inspection and renewal of sample chamber;
- Inspection and adjustment of electrical connections;
- Clean and check system components;
- Periodic checks of chamber vacuum;
- Check, adjust and repair vacuum manifold;
- Change oil, check, repair vacuum pump;
- Maintenance of software, analysis protocols and system backup.

8.4.4 LIQUID SCINTILLATION COUNTER

Regularly performed maintenance includes, but is not limited to, the following for liquid scintillation spectrometers:

- Periodic background checks;
- Periodic checks of detector stability (C-14 and H-3 efficiency);
- Periodic detector normalization and voltage adjustments (automatic);
- Inspection and cleaning of instrument;
- Periodic cleaning of PMT's and optical systems;
- Inspection and adjustment of electrical connections;
- Inspection and maintenance of refrigeration systems;
- Cleaning and checks of system components;
- Checks, adjustment and repair of automatic sample changer components;
- Maintenance of software, analysis protocols and system backup.

8.4.5 ALPHA SCINTILLATION SYSTEM

Regularly performed maintenance includes, but is not limited to, the following for alpha scintillation systems:

- Periodic dark count background checks;
- Periodic checks of detector stability;
- Detector plateau determinations;
- Threshold adjustment;
- Operating bias adjustment;
- Inspection and cleaning of instrument;
- Periodic cleaning of PMT and optical systems;
- Inspection and adjustment of electrical connections;
- Cleaning and checks of system components;
- Maintenance of printer apparatus;
- Alpha Scintillation Flask background checks;
- Alpha Scintillation Flask recalibration;
- Alpha Scintillation Flask maintenance;
- Alpha Scintillation Flask scintillation material renewal.

8.5 INORGANIC INSTRUMENT MAINTENANCE PROCEDURES

8.5.1 INDUCTIVELY COUPLED ARGON PLASMA (ICAP)

Regularly performed maintenance includes, but is not limited to, the following for ICAP systems:

- Monitor argon gas tank pressure daily;
- Replace argon gas tank when contents fall below 1/8 full volume;
- Clean nebulizer and spray chamber as needed;
- Replace and realign plasma torch monthly;
- Clean air filters monthly;
- Monitor vacuum pump oil level and add oil as needed;
- Check cooling system water level;

- Empty waste reservoir daily;
- Clean components to prevent acid corrosion as needed;
- Monitor instrument response daily;
- Replace peristaltic pump tubing as needed.

8.5.2 GRAPHITE FURNACE SPECTROPHOTOMETER

Regularly performed maintenance includes, but is not limited to, the following for graphite furnaces:

- Clean components to prevent acid corrosion as needed;
- Align source lamps to ensure maximum sensitivity whenever elemental emission lamps are changed;
- Clean and inspect graphite tube sample platform daily and replace it when surface appears excessively burned or cracked or a buildup of minerals is evident;
- Clean and inspect contact ring, replacing when excessively worn;
- Clean optical path of the mirrors and sensors as needed;
- Check autosampler injector alignment to ensure placement of sample in correct location on the graphite tube;
- Monitor instrument response daily;
- Replace rinse and waste water as needed;
- Replace fume extraction water and filter as needed.

8.5.3 COLD VAPOR SPECTROPHOTOMETER

Regularly performed maintenance includes, but is not limited to, the following for mercury analyzers:

- Replace drying tube each morning before sample analysis;
- Replace pump tubing weekly or when deterioration is evident;
- Replace mercury lamp after ~2000 hr of use (when relative absorbance of a standard has changed significantly while the optical cell is clean);
- Align source lamp as needed;

- Remove and clean sample cell and connecting tubes;
- Clean and lubricate autosampler to ensure smooth operation of the servo mechanisms as needed;
- Check sparger for proper operation;
- Clean sample compartment windows.

8.5.4 ION CHROMATOGRAPH

Regularly performed maintenance includes, but is not limited to, the following for IC systems:

- Check nitrogen and helium pressure;
- Check eluent level;
- Check pump pressure and flow rate;
- Check retention time for fluoride (earliest) and sulfate (latest);
- Check for system leaks within column and valve compartment;
- Rinse off dried eluents and reagents;
- Check air and liquid transfer lines for discoloration, replace lines if discolored;
- Replace guard column as needed;
- Replace suppresser as needed.

If instrument sensitivity has decreased, clean detector with a dilute solution of nitric acid to restore the sensitivity of the cell.

8.6 ORGANIC INSTRUMENT MAINTENANCE PROCEDURES

8.6.1 GAS CHROMATOGRAPHS

Regularly performed maintenance includes, but is not limited to, the following for GC instrumentation:

- Clip ~12 inches from the injection end of the capillary columns as needed to remove active sites;

- Replace injection port liner as needed to remove active sites;
- Replace septum as needed when symptoms of septum deterioration are noted;
- Replace carrier and detector gases when the supply of gas in the cylinder falls between 400-500 psi to prevent contaminants from reaching the column and detector;
- Leak check instrument after maintenance;
- Replace molecular sieves and oxygen traps to remove accumulations when decline in instrument performance is noted;
- NRC wipe test ECD (semiannually) to ensure that the housing of the radioactive source remains effective in preventing any release of any radioactive substance;
- Replace vent traps (semiannually) to ensure that organic vapors are trapped.

Instrument calibration curves will be monitored and compared to historical performance. Excessive noise, low response, and poor precision may be indications of dirty injection liners, columns, and/or detectors. Replacing injection liners, clipping the front end of the column, and cleaning detectors are activities that may correct some of these problems. Spare columns, packing materials, and instrument cables will be available in case of breakage or malfunction to minimize instrument downtime.

8.6.2 GAS CHROMATOGRAPH/MASS SPECTROMETERS

Regularly performed maintenance includes, but is not limited to, the following for GC/MS instrumentation:

- Hard tune with calibration gas (PFTBA);
- Clip ~12 inches from the injection end of the capillary columns as needed to remove active sites;
- Replace injection port liner as needed to remove active sites;

- Replace septum as needed when symptoms of septum deterioration are noted;
- Replace carrier and detector gases when the supply of gas in the cylinder falls between 400-500 psi to prevent contaminants from reaching the column and detector;
- Leak check instrument after maintenance;
- Replace molecular sieves and oxygen traps to remove accumulations when decline in instrument performance is noted;
- Clean ionizing source as needed to prevent loss of sensitivity;
- Change vacuum pump oil (semiannually);
- Replace vent traps (semiannually) to ensure that organic vapors are trapped.

Instrument calibration curves will be monitored and compared to historical performance. Excessive noise, low response, and poor precision may be indications of dirty injection liners, columns, and/or detectors. Replacing injection liners, clipping the front end of the column, and cleaning detectors are activities that may correct some of these problems. Spare columns, packing materials, and instrument cables will be available in case of breakage or malfunction to minimize instrument downtime.

8.6.3 HIGH-PERFORMANCE LIQUID CHROMATOGRAPHS

Regularly performed maintenance includes, but is not limited to, the following for HPLC instrumentation:

- Replace sparging and autosampler gases when the supply of gas in the cylinder falls between 400-500 psi to prevent contaminants from reaching the solvent reservoirs;
- Replace pre-column filter and guard column as needed;
- Replace water every 2 weeks to prevent algae formation;
- Change the cup filter in the inlet valve when it becomes difficult to prime the pump.

Instrument calibration curves will be monitored and compared to historical performance. Excessive noise, low response, and poor precision may be indications of dirty columns, dirty guard columns, and/or dirty detectors. Replacing the column, changing the guard column, or rinsing the system with 6 N nitric acid, followed by ultra-pure water are activities that may correct some of these problems. Spar columns, guard columns, and pre-column filters will be available in case of malfunction to minimize instrument downtime.

8.7 MISCELLANEOUS INSTRUMENT MAINTENANCE PROCEDURES

8.7.1 ANALYTICAL BALANCES

Analytical balances are calibrated annually by a certified technician. A dated sticker, certifying the calibration, is placed on each balance. Records for balance calibration/servicing are maintained in Paragon QA files. Multi and single point calibration checks are regularly performed to ensure the accuracy of each balance. The results are recorded in dedicated logbooks that are maintained at each balance location. Balances that do not satisfy specifications are taken out of service for replacement or repair. Class "S" weights must be verified/calibrated every two years.

8.7.2 LABORATORY OVENS AND REFRIGERATORS

Refrigerators and ovens are monitored once or twice daily or as used, depending upon the function of the unit. Logbooks are maintained to record monitoring and results. For sample refrigerators (excluding standards or extract storage refrigerators) the internal temperature will be monitored and record every business day. The acceptance criteria for the sample storage refrigerators is $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. For the extract and standards storage refrigerators, temperature reading should be 4°C or less.

If a unit fails acceptance criteria, monitoring is continued until the temperature stabilizes within the range or appropriate corrective actions are taken. Monitoring occurs at one (1) hour intervals for a maximum four (4) hour period; if the reading following the temperature

control adjustment is out, the unit is considered “out of order,” and is emptied and serviced. It is not put back into service until shown to be stable at the required temperature range.

When not in use, laboratory ovens and kilns will be turned off and allowed to cool to room temperature. Prior to use, ovens will be turned on and given sufficient warm-up time to allow them to meet their operating temperature. For kilns used to dry/clean glassware at > 400 °C, glassware will be loaded in the cool kiln, and power applied to bring the unit to final temperature. Ovens used for drying glassware should be maintained at a temperature between 105 °C and 200 °C.

8.7.3 TOTAL ORGANIC CARBON ANALYZER

Regularly performed maintenance includes, but is not limited to, the following for a total organic carbon analyzer:

- Replace injector septum every 100 injections or whenever leakage is observed;
- Fill reservoir with fresh solution when low;
- Monitor sparge/carrier gas daily;
- Inspect tin sample scrubber daily and replace/repack scrubber if color change is observed.

8.7.4 GENERAL LABORATORY AREAS

- Calibrate automatic pipettes and burettes weekly;
- Clean, check, calibrate to manufacturer’s specifications all pH, DO, conductivity and turbidity meters and spectrophotometers semiannually;
- General housekeeping: keep counter tops, hoods, and floors clean; and
- Check airflow in hoods quarterly.

8.8 SPARE PARTS

An adequate inventory of spare parts is required to minimize equipment downtime. This inventory should emphasize those parts and supplies that:

- Are subject to frequent failure,

- Have limited useful lifetimes, or
- Cannot be obtained in a timely manner should failure occur.

Department Managers are responsible for maintaining an adequate inventory of necessary spare parts for all major instruments and equipment items. Examples of spare parts maintained for major instrumentation systems are listed in Table 8-1.

8.9 CONTINGENCY PLAN

In the event of a catastrophic instrument failure, every effort will be made to analyze samples within holding times by alternate means. If the redundancy in equivalent instrumentation is insufficient to handle the affected samples, then the analyst and/or Department Manager will notify the Project Manager in a timely fashion (i.e., several days before holding times expire) to ensure that the Project Manager has adequate time to notify the client and discuss options.

Table 8-1 SCHEDULED MAINTENANCE PROCEDURES AND REPRESENTATIVE SPARE PARTS FOR MAJOR INSTRUMENTATION

INSTRUMENT	MAINTENANCE PROCEDURE	SPARE PARTS
Gas Chromatograph Mass Spectrometer (GC/MS)	<ol style="list-style-type: none"> 1. Change septa and liner 2. Clip column 3. Replace pump oil 4. Change gas line dryers 5. Clean ionizing source 6. Replace electron multiplier 7. Replace gases 8. Replace vent traps 	<ol style="list-style-type: none"> 1. Syringes 2. Septa and inserts 3. GC columns 4. Various electronic components 5. Plumbing supplies - tube fittings 6. Filaments 7. Source parts
Gas Chromatograph (GC)	<ol style="list-style-type: none"> 1. Change septa and liners 2. Clip column 3. Change gas line dryers 4. Leak check when installing new analytical column 5. Replace gases 6. Replace vent traps 	<ol style="list-style-type: none"> 1. Syringes 2. Septa and inserts 3. GC columns 4. Various electronic components 5. Plumbing supplies - tube fittings
Purge and Trap Sample Concentrator	<ol style="list-style-type: none"> 1. Replace trap 2. Decontaminate system as required by blank analysis 3. Leak check system 4. Measure flowrates for each sparging position 	<ol style="list-style-type: none"> 1. Spare traps 2. Various electronic components and circuit boards 3. Plumbing supplies - tube fittings
Ion Chromatograph (IC)	<ol style="list-style-type: none"> 1. Replace guard column 2. Replace separator column 3. Replace suppresser 4. Replace valve system 	<ol style="list-style-type: none"> 1. Guard column 2. Separator column 3. Suppresser 4. Valve
Inductively Coupled Argon Plasma Spectrometer (ICP)	<ol style="list-style-type: none"> 1. Clean torch assembly and mixing chamber when discolored or after 8 hours of running high dissolved solids samples 	<ol style="list-style-type: none"> 1. Spare torch and mixing chamber 2. Spare coil 3. Plumbing supplies - tube fittings
Graphite Furnace Atomic Absorption Spectrophotometer	<ol style="list-style-type: none"> 1. Change graphite contact rings 2. Clean quartz windows 	<ol style="list-style-type: none"> 1. Contact rings 2. Graphite cups and electrodes 3. Autosampler tubing
Hg Analyzer	<ol style="list-style-type: none"> 1. Clean tubing and quartz cell 2. Clean aspirator 3. Replace drying tube media 	<ol style="list-style-type: none"> 1. Quartz cells 2. Aspirator 3. Plumbing supplies

9. QUALITY CONTROL PROCEDURES

A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out-of-control events, and requiring corrective action measures to prevent or minimize the recurrence of these events. QC procedures are implemented to ensure that sample data meet the quality objectives of the laboratory and the client. An effective QC program must be able to control the quality of the data through the monitoring of QC indicators. Criteria frequently applied to environmental QC data include measurements of accuracy and precision. Precision measures the randomness associated with an analytical measurement and reflects the inherent variability in that measurement system. Accuracy reflects the degree to which the measured value approximates the actual or "true" value for a given parameter and reflects the influence of systematic biases in the measurement. Thus, the "quality" of QC data can be said to be a measure of both the randomness and biases in a specific measurement system.

For all analyses performed by Paragon, the QC checks described in this section are mandatory. Table 9-1 through Table 9-4 summarize minimum QC sample requirements. Section 3 of this LQAP contains the acceptance criteria that will be used by the laboratory. Department Managers are responsible for reviewing QC acceptance criteria for each method performed by their department. EPA precision and accuracy data should be used as a basis for developing acceptance criteria for assessing the precision and accuracy of generated data. However, once an adequate database of analysis batches for a given method has been established, the control chart procedure described in Section 9.4 will be used to control the analysis.

Table 9-1 MINIMUM QC SAMPLE REQUIREMENTS FOR METALS AND INORGANICS ANALYSIS

Analysis Type	Method Blank	Laboratory Control Sample	Sample Replicate	Sample Matrix Spike/Spike Replicate
Metals Analysis (ICAP, GFAA, CVAA)	Every Batch or 5 %	Every Batch or 5 %	Every Batch or 5 %	1 Pair Every Batch or 5 %
Nonmetallic Inorganics Analysis (Anions, Autoanalyzer & Colorimetric Methods)	Every Batch or 5 %	Every Batch or 5 %	--	1 Pair Every Batch or 5 %
Cyanide ¹ Analysis	Every Batch or 5 %	Every Batch or 5 %	Every Batch or 5 %	1 MS Every Batch or 5 %
Nonmetallic Inorganics Analysis (Titrimetric Methods)	Every Batch or 5 %	Every Batch or 5 %	Every Batch or 5 %	--
Gravimetric Methods (TDS, TSS, % Moisture, Oil & Grease, etc.)	Every Batch or 5 %	Every Batch or 5 %	Every Batch or 5 %	--
Total Organic Carbon	Every Batch or 5 %	Every Batch or 5 %	--	1 MS Every Batch or 5 %
Electrometric Methods	Every Batch or 5 %	Every Batch or 5 %	Every Batch or 5 %	--
TCLP	Every Batch or 5 %	Every Batch or 5 % ²	--	1 MS Every Batch or 5 % ³

¹ Cyanide analyzed by CLP SOW will follow requirements specified in the SOW.

² QC sample is created by spiking appropriate matrix (blank or sample) after TCLP extract generation.

Table 9-2 MINIMUM QC SAMPLE REQUIREMENTS FOR GC AND GC/MS ORGANICS ANALYSIS

Analysis Type	Method Blank	Laboratory Control Sample	Surrogate Spike	Sample Matrix Spike/Spike Replicate ³
GC/MS Analyses (VOA's, Semivolatiles)	Every Batch or 5 %	Every Batch or 5 %	Every Sample	1 Pair Every Batch or 5 %
GC/HPLC Analyses (Surrogates Avail.)	Every Batch or 5 %	Every Batch or 5 %	Every Sample	1 Pair Every Batch or 5 %
GC/HPLC Analyses (Surrogates N/A)	Every Batch or 5 %	Every Batch or 5 %	--	1 Pair Every Batch or 5 %
TCLP	Every Batch or 5 %	Every Batch or 5 % ⁴	Every Sample ⁴	1 MS Every Batch or 5 % ⁴

³ Client may specify which field samples are to be selected for MS/MSD pairs. If not, laboratory will select field sample.

⁴ QC sample is created by spiking appropriate matrix (blank or sample) after TCLP extract generation.

Table 9-3 MINIMUM QC SAMPLE REQUIREMENTS FOR RADIOCHEMICAL ANALYSES

Analysis Type	Method Blank	Laboratory Control Sample	Internal Isotopic Tracer	Sample Matrix Spike	Sample Replicate ⁵
Gas Flow Proportional Counting	Every Batch or 5 %	Every Batch or 5 %	--	--	Every Batch or 10 %
Gamma Spectroscopy	Every Batch or 5 %	Every Batch or 5 % ⁶	--	--	Every Batch or 10 %
Alpha Spectroscopy	Every Batch or 5 %	Every Batch or 5 %	Every Sample	--	Every Batch or 10 %
Liquid Scintillation	Every Batch or 5 %	Every Batch or 5 %	--	--	Every Batch or 10 %
Alpha Scintillation	Every Batch or 5 %	Every Batch or 5 %	--	--	Every Batch or 10 %
Kinetic Phosphorescence Analysis	Every Batch or 5 %	Every Batch or 5 %	--	Every Batch or 5 %	Every Batch or 10 %

⁵ Client may specify which field samples are to be selected for duplicate analyses. If not, laboratory will select field sample.

⁶ LCS supplied in instrumentation laboratory.

Table 9-4 MINIMUM QC SAMPLE REQUIREMENTS FOR WET CHEMISTRY ANALYSES

Methods	Method No.	QC Required ⁷
Ammonia	350.3	MB, LCS, MS/MSD
Alkalinity (Total)	310.1	MB, LCS, DUP
Bicarbonate	310.1 mod	MB, LCS, DUP
Carbonate	310.1 mod	MB, LCS, DUP
Chloride	325.3	MB, LCS, DUP
Total Cyanide	9010	MB, LCS, MS/MSD
Amenable Cyanide	9010	MB, LCS, MS/MSD
Fluoride	340.2	MB, LCS, MS/MSD
Hexavalent Chromium	7196	MB, LCS, DUP
NO ₂ + NO ₃ as N	353.3	MB, LCS, MS/MSD
NO ₃ as N	353.3	MB, LCS, MS/MSD
NO ₂ as N	354.1	MB, LCS, MS/MSD
Reactive Cyanide	SW 846 (7)	MB, LCS, DUP
Reactive Sulfide	SW 846 (7)	MB, LCS, DUP
Ortho Phosphate as P	365.3	MB, LCS, MS/MSD
Total Phosphate as P	365.3	MB, LCS, MS/MSD
Sulfate	375.4	MB, LCS, DUP
Sulfide	376.1	MB, LCS, DUP
TDS	160.1	MB, LCS, DUP
TSS	160.2	MB, LCS, DUP
Ion Chromatography		
Fluoride	300	MB, LCS, MS/MSD
Chloride	300	MB, LCS, MS/MSD
Bromide	300	MB, LCS, MS/MSD
Nitrate as N	300	MB, LCS, MS/MSD
Nitrite as N	300	MB, LCS, MS/MSD
Ortho Phosphate as P	300	MB, LCS, MS/MSD
Sulfate	300	MB, LCS, MS/MSD

⁷ Legend: MB = Method Blank; LCS = Laboratory Control Sample; MS/MSD = Matrix Spike/Matrix Spike Duplicate; DUP = Sample Duplicate.

9.1 DEFINITION OF ANALYSIS BATCH

For QC purposes, field samples and qc samples processed as a unit constitute a batch. A batch may contain as many as 20 field samples of similar matrix. The field samples must be processed with the appropriate qc samples (eg, method blank, laboratory control samples, matrix spike samples, duplicate samples). The number and type of QC samples specified in this section will apply to a batch of samples. For example, a group of field and qc samples that are extracted on the same day and (if required) undergo concentration and clean-up procedures on subsequent days would be considered one batch for QC purposes.

Some analyses (such as volatile organics by GC or GC/MS, anions by ion chromatography, etc.) require no preparation before analysis; therefore, the batch definition presented above does not apply. For GC/MS volatiles analyses, the determination of a batch of samples is further restricted to the number of samples that may be analyzed during the 12-hour period that follows the injection of the tuning standard, BFB.

9.2 QC SAMPLE DEFINITION AND USE

The results of quality control samples created in the laboratory represent estimates of accuracy and precision for the preparation and analysis steps of sample handling. This section describes the quality control information provided by each of these analytical measurements. Information on the procedures to follow in preparation of the samples or spiking solutions is described for each method and matrix in the respective method's Standard Operating Procedure.

9.2.1 METHOD BLANK

A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix, carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or weight processed. Analysis of the blank verifies that method interferences caused by

contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Optimally, a method blank should contain less than the reporting limit for all parameters unless otherwise specified in the method or this LQAP. For radiochemical analyses, a suitable blank solid matrix has not been identified; therefore deionized water will be used for the blank matrix for analyses of solid matrices.

9.2.2 LABORATORY CONTROL SAMPLE

Laboratory Control Samples (LCS) consist of aliquots of ideal matrices (water, sand, etc.) spiked with analytes of interest and processed through each sample preparation and analysis procedure. (Where sample pretreatment is not required, such as with ion chromatography or GC volatiles in water for example, the Initial Calibration Verification or other appropriate control standard can be employed as the LCS). LCSs for methods with extensive lists of analytes that may interfere with one another may include a limited number of analytes, but the analytes included must be representative of as many analytes as is practical. In the case of metals analysis, all analytes of interest must be included.

Laboratory pure water is used to prepare most LCSs for methods for analysis of water. Highly characterized solids, where available, are used for LCSs for methods for analysis of solids. Where no such solid LCS is available, spiked laboratory pure water or spiked reagent blanks may be substituted. LCSs provide an estimate of bias based on recovery of the compounds from a clean, control matrix. They provide evidence that the laboratory is performing the method within accepted guidelines without potential non-matrix interferences.

9.2.3 SAMPLE MATRIX SPIKE/MATRIX SPIKE REPLICATE

These QC samples are generated in a manner similar to Laboratory Control Samples, except that instead of spiking into a well-characterized standard matrix, replicate aliquots of field samples are used as the spike matrix. These QC samples incorporate sample matrix effects and field conditions. To generate the MS/MSD pairs for any

analysis, there must be an adequate volume/weight of field sample available. Inadequate sample volumes preclude the possibility of generating this set of QC samples. For this reason, it will be necessary for the field sampling contractor to designate which samples will be used for MS/MSD analysis, and to ensure that adequate sample volumes for the designated samples are collected.

Note that for some inorganic analysis techniques (such as hardness, alkalinity, TOC, etc.) changing the composition of the sample in any way invalidates the analysis method to be performed. Under these circumstances, a MS/MSD pair cannot be generated. Normally, duplicate sample aliquots are analyzed in order to generate an estimate of a method's precision.

9.2.4 SURROGATE SPIKES

Surrogates are compounds that exhibit chemical characteristics that are similar to a method's target analytes, but are unlikely to be present in actual field samples. They provide an estimate of bias based on recovery of similar compounds, for a given extraction technique/analysis method combination. These bias estimates will incorporate sample matrix effects and field sampling conditions, as well as the variability/bias of the laboratory analysis process. When used in the laboratory, surrogate spikes are introduced into all field and QC samples in a batch, prior to the commencement of sample processing for analysis.

9.2.5 SAMPLE DUPLICATE

A Sample Duplicate is a sample that has been split into two equal portions before the method sample preparation process. It measures sample precision associated with an analysis method from the preparation through final analysis. For organic analyses the MS/MSDs fill this function and provide an measure of overall precision.

9.2.6 CHEMICAL YIELD MONITORS

This type of QC tool is used primarily for radiochemical analyses, and is similar in concept to the surrogate spike discussion above. The primary difference between this QC

sample type and surrogates is that sample analysis results are corrected for observed chemical yields, and surrogates are not used for this correction. A chemical yield monitor is a substance that has similar chemical characteristics as the parameter being measured, and is introduced into all field and QC samples in a batch prior to commencing the analysis process. Chemical yield monitors also provide information regarding the performance of a method on a sample by sample basis. If the yield is outside the acceptance requirements of the method, corrective action by the laboratory is mandated.

9.3 UTILIZATION OF QUALITY CONTROL DATA

The purpose for preparing and analyzing quality control samples is to demonstrate, through the known entities, how accurate and precise the investigative sample data are. The tables in Section 3.0 of this LQAP summarize the quality control assessment criteria by matrix for the most commonly used methods by Paragon . Different criteria may be dictated by different methods or by project QA plans. All assessments of quality control data will be performed after all rounding and significant figure truncations have been performed.

9.4 CONTROL CHARTS

Control charts are a vital tool that can assist the laboratory in evaluating method control and assessing trends. Diligent use of control charts by the laboratory analyst can be an aid in understanding the routine performance expectations for a method, and can help prevent a measurement system from drifting into an out-of-control situation; corrective action can be taken before any sample analyses are jeopardized.

Accuracy and precision control charts are generally maintained for each method that utilizes a laboratory control spike as the QC sample for assessment of method control. For methods that cannot use LCS samples for method control (such as pH, specific conductivity, flashpoint, etc.), other acceptance criteria will be used to assess method control. For certain low-volume methods (less than 10 analysis batches performed by Paragon in one calendar year) tabulated control limits are used to monitor acceptability of quality control measurements instead of control charts.

9.4.1 ACCURACY CONTROL CHARTS

Accuracy (or recovery) control charts will be evaluated by plotting the individual percent recovery point for a control analyte on a control chart and comparing its value against the current control limits. If the QC spike recovery value for the current analytical batch meets the acceptance criteria for that method, the data point will be incorporated into the control chart database for that method. This database will be used to calculate and update the control limits for the method for each of the control analytes.

Starting with the 20th analysis batch, the precision data from the previous batches will be pooled, and an average precision value will be calculated. Control chart limits will be calculated from this average recovery value from the pooled data set, and from the standard deviation of the pooled data. The upper and lower warning limits of the control chart will be values equal to the average recovery plus or minus two times the standard deviation, and the upper and lower control limits of the control chart will be values equal to the average recovery plus or minus three times the standard deviation. These four limits, plus the average recovery, will be plotted on the control chart, along with the individual recoveries for all the analysis batches.

At this time, the control limits for the parameter will be fixed, and the data points for the next 20 analysis lots will be plotted and compared to these fixed control limits. For each subsequent set of 20 analysis batches, the precision control limits will be calculated using the data from the previous 40 data batches, and the limits will be fixed for the following 20 batches.

The frequency of updating control limits may vary somewhat for some very high-frequency use methods that process high volumes of analysis in short periods of time. For these high-use methods, the control limit update frequency may be extended to once every 50 or 100 analysis batches. The goal for determining the update frequency of any method will be to try to recalculate control charts no more frequently than once every three months, but have limits recalculated at least once per year.

9.4.2 PRECISION CONTROL CHARTS

Precision control charts will be generated from recovery data from the laboratory control sample that will be part of every analysis batch performed at Paragon. The accuracy values for the batch currently being evaluated and the last two analysis batches will be examined and the highest and lowest values for these three batches will be identified. A percent range value will be calculated by subtracting the lowest recovery value from the highest recovery value. This range value will be compared to the current control chart control limits for that method and that analyte. Since the range value for precision will never be less than zero, there will only be an upper control limit for this measurement; there is no lower control limit.

Precision control limits will be calculated using procedures developed by the U.S. Environmental Center (Source, USAEC IRPQAP, January, 1990). Starting with the 20th analysis batch, the precision data from the previous batches will be pooled, and an average precision value will be calculated. The upper control limit (UCL) of the precision control chart will be calculated by multiplying the average precision value by the factor 2.575. The upper warning limit (UWL) will be calculated by multiplying the average precision value by 2.050. At this time, the control limits for the parameter will be fixed, and the data points for the next 20 analysis lots will be plotted and compared to these fixed control limits. For each subsequent set of 20 analysis batches, the precision control limits will be calculated using the data from the previous 40 data batches, and the limits will be fixed for the following 20 batches.

9.5 EVALUATION OF CONTROL CHARTS

Plotting and connecting successive data points on control charts enables the laboratory to detect many types of suspicious and out-of-control situations. These events can be caught by monitoring the following: outliers, runs, trends, and periodicity.

There are two types of outliers: any particular point that falls outside the control limits or any point that falls outside the warning limits. A point that falls outside the control

limits is classified as an out-of-control event; a point that falls outside the warning limits is classified as a suspicious event. When two consecutive control chart points fall within the same warning limit band, this will be classified as an out-of-control event.

A run is defined as a series of points that line up on one side of the central line (the mean). Any run that has a length of seven points is indicative of a potential abnormality in the process, a suspicious event. A run can suggest several potential problems such as a leak in the system, elevated contamination, or incorrect dilutions of standards.

A trend is defined as a series of points that are marked by an unbroken rise or fall. Any trend with a length of five points is classified as a suspicious event. A trend may indicate a change in instrument sensitivity due to a dirty source or injection port or standard degradation, to name a few.

Periodicity is a term used to describe a recurring pattern of change over equal intervals. This occurrence may be of any length or amplitude; thus, careful observation of the control chart is necessary.

9.5.1 OUTLIER REJECTION

For the generation of control charts, and with the evaluation of other QC data sets, it is important to prevent spurious or erroneous data from being incorporated into the data being examined. On occasion it may be necessary to reject data as being an outlier to keep it from having an adverse impact on the values being calculated from the data set. This is especially true with control chart data, and other data sets from which calculations of the laboratory's standard performance for methods and processes.

For the purposes of statistically determining whether a data point is an outlier or not, Paragon will use the procedures discussed in the Dixon Rank Sum Test ("Processing Data

for Outliers”, by W.J. Dixon, Biometrics, Vol. 9, No. 1, 1953) for the identification of outliers in control charts, and other data sets. If a control chart data point is identified as an outlier, the following procedure will be followed:

The data point for that analysis lot will be plotted on the control chart, but will be identified (with a flag or notation) as an outlier. The data point will not be incorporated into the control chart database when updating limits, nor will it be used to calculate the 3-day precision values for the following data batches.

9.5.2 DIXON OUTLIER TEST PROCEDURE

Dixon’s test expresses the gap between an outlier and the nearest value as a fraction of the range between the smallest and largest value in the data set being tested.

The entire data set must be ordered from highest to lowest, with the highest value assigned a rank of 1 (X_1) and the lowest value a rank of n (X_n). The test criterion (r) varies with sample size, as follows:

For less than eight measurements, reject X_n (the lowest value) if

$$\frac{X_n - X_{(n-1)}}{X_n - X_1} > r(10);$$

For less than eight measurements, reject X_1 (the highest value) if

$$\frac{X_2 - X_1}{X_n - X_1} > r(10);$$

Between eight and ten measurements, reject X_n (the lowest value) if

$$\frac{X_n - X_{(n-1)}}{X_n - X_2} > r(11);$$

Between eight and ten measurements, reject X_1 (the highest value) if

$$\frac{X_2 - X_1}{X_{(n-1)} - X_1} > r(11);$$

Between eleven and thirteen measurements, reject X_n (the lowest value) if

$$\frac{X_n - X_{(n-2)}}{X_n - X_2} > r(21);$$

Between eleven and thirteen measurements, reject X_1 (the highest value) if

$$\frac{X_3 - X_1}{X_{(n-1)} - X_1} > r(21);$$

Over thirteen measurements, reject X_n (the lowest value) if

$$\frac{X_n - X_{(n-2)}}{X_n - X_3} > r(22);$$

Over thirteen measurements, reject X_1 (the highest value) if

$$\frac{X_3 - X_1}{X_{(n-2)} - X_1} > r(22)$$

The critical values for the test statistic at 98 percent confidence level are shown in Table 9-5. If the test statistic is greater than the critical value from the table, then the data point is an outlier. Once adequate data are available, n shall be kept constant at 20, with the 20 most recent data points being used.

On occasion, control chart data points may be flagged as an outlier by the laboratory even though they do not fail the Dixon outlier test, or may even be within control limits. This can be done only when there is a precisely defined cause as to why that data point should not be incorporated into the database. The reason that points would be identified in this

Table 9-5 CRITICAL VALUES FOR DIXON'S OUTLIER TEST

<u>Number of Measurements (n)</u>	<u>Criterion (r)</u>	<u>Critical Value of r</u>
3		0.976
4		0.846
5	r ₁₀	0.729
6		0.644
7		0.586
8		0.631
9	r ₁₁	0.587
10		0.551
11		0.638
12	r ₂₁	0.605
13		0.578
14		0.602
15		0.579
16		0.559
17		0.542
18		0.527
19	r ₂₂	0.514
20		0.502
21		0.491
22		0.481
23		0.472
24		0.464
25		0.457

Source; USATHAMA IRP Quality Assurance Plan, January, 1990.

fashion is to keep control data that is not characteristic of the normal performance of the method from being incorporated into the control chart database and improperly biasing the control limits.

An example of this situation might be if the control spike was accidentally prepared at twice the normal spiking concentration for the control spike. Even though the spike targets could be adjusted to compensate for the increased analyte concentration, it is not desirable to include this data into a database that was based on a different analyte target concentration. In order to maintain the integrity of the control chart database, this point would be flagged as an outlier in order to keep it from being incorporated into the database. Other circumstances could take place that could cause manual rejection of control data points. In every case, the cause of the outlier rejection must be clearly understood, and the Laboratory QA Manager must be informed of and agree to the manual rejection of any data point.

9.6 MINIMUM QUALITY CONTROL - INORGANICS

The sections that follow summarize the minimum QC procedures that will be followed for each inorganics analysis methodology. These sections summarize the minimum QC requirements for the methodology; additional QC procedures (such as method of standard additions, post-digestion fortification of samples, etc.) may be performed as necessary. In the event that EPA Contract Laboratory Program procedures are required for a client project, the procedures specified in this program's Statement of Work (SOW) will be followed by the laboratory, and will supersede the requirements that follow.

9.6.1 INDUCTIVELY COUPLED ARGON PLASMA (ICAP)

For each batch of samples analyzed by ICAP, the following QC checks will apply:

1. At least one method blank will be analyzed.
2. At least one LCS (spiked with all reported analytes) will be analyzed.
3. One MS/MSD pair will be analyzed.
4. One sample duplicate will be analyzed.
5. One sample serial dilution (Dilution Factor = 5) will be analyzed.

6. An initial multi-point calibration (three to six standards plus a calibration blank) must be generated for all methods. The calibration curve correlation coefficient must be equal to or greater than 0.995.
7. A one-point calibration verification standard may be analyzed and compared against the initial calibration curve. If the one-point calibration passes acceptance criteria, the analysis of samples may proceed, and sample results quantitated against the initial calibration curve.
8. A second-source calibration verification standard will be analyzed.
9. Two interference check standards will be analyzed, and these checks will be analyzed at the beginning and at the end of the analysis run.
10. A drift-check standard will be analyzed between every 10 field samples and at the end of the analysis run.
11. A continuing calibration blank will be analyzed between every 10 field samples and at the end of the analysis run.
12. Samples will be diluted as required to keep all analyte concentration ranges in the digestates within the calibration range of the initial multi-point calibration curve.

9.6.2 GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA)

For each batch of samples analyzed by GFAA, the following QC checks will apply:

1. At least one method blank will be analyzed.
2. At least one LCS (spiked with all reported analytes) will be analyzed.
3. One MS/MSD pair will be analyzed.
4. One sample duplicate will be analyzed.
5. A minimum of 3 calibration standards and a blank will be analyzed every day of analysis. The correlation coefficient of the calibration curve must be greater than or equal to 0.995.
6. A second-source calibration verification standard will be analyzed.
7. A drift-check standard will be analyzed between every 10 field samples and at the end of the analysis run.

8. A continuing calibration blank will be analyzed between every 10 field samples and at the end of the analysis run.
9. Samples will be diluted as required to keep all analyte concentration ranges in the digestates within the calibration range of the multi-point calibration curve.

9.6.3 COLD VAPOR ATOMIC ABSORPTION

For each batch of samples analyzed by CVAA for mercury, the following QC checks will apply:

1. At least one method blank will be analyzed.
2. At least one LCS will be analyzed.
3. One MS/MSD pair will be analyzed.
4. One sample duplicate will be analyzed.
5. A minimum of 5 calibration standards and a blank will be analyzed every day of analysis. The correlation coefficient of the calibration curve must be greater than or equal to 0.995.
6. A second-source calibration verification standard will be analyzed.
7. A drift-check standard will be analyzed between every 10 field samples and at the end of the analysis run.
8. A continuing calibration blank will be analyzed between every 10 field samples and at the end of the analysis run.
9. Samples will be diluted as required to keep the mercury concentration ranges in the digestates within the calibration range of the multi-point calibration curve.

9.6.4 ION CHROMATOGRAPHY

For each batch of samples analyzed by ion chromatography for anions, the following QC checks will apply:

1. At least one method blank will be analyzed.
2. One LCS (spiked at a fixed concentration) will be analyzed.
3. One MS/MSD pair will be analyzed.
4. An initial multi-point calibration (minimum of five standards plus a calibration blank) must be generated. The calibration curve correlation coefficient must be equal to or

greater than 0.995. If this criteria is met, analysis may commence immediately following the calibration curve.

5. A one-point calibration verification standard may be analyzed and compared against the latest initial calibration curve. If the one-point calibration passes acceptance criteria, the analysis of samples may proceed, and sample results quantitated against the initial calibration curve.
6. A second-source calibration verification standard will be analyzed.
7. A drift-check standard will be analyzed between every 10 field samples and at the end of the analysis run.
8. Samples will be diluted as required to keep the analyte concentration ranges in the samples within the calibration range of the multi-point calibration curve.

9.6.5 AUTOANALYZER METHODS

For each batch of samples analyzed using an autoanalyzer (such as TOC, TOX, TDC, etc.), the following QC checks will apply:

1. At least one method blank will be analyzed.
2. One LCS (spiked at a fixed concentration) will be analyzed.
3. One MS/MSD pair will be analyzed.
4. A multi-point calibration (minimum of five standards plus a calibration blank) must be generated each day of analysis. The calibration curve correlation coefficient must be equal to or greater than 0.995.
5. A drift-check standard will be analyzed between every 10 field samples and at the end of the analysis run.
6. Samples will be diluted as required to keep the analyte concentration ranges in the samples within the calibration range of the multi-point calibration curve.

9.6.6 SPECTROPHOTOMETRIC METHODS

For each batch of samples analyzed using UV-Visible or other spectrophotometric techniques, the following QC checks will apply:

1. At least one method blank will be analyzed.
2. One LCS (spiked at a fixed concentration) will be analyzed.

3. One sample MS/MSD pair will be analyzed.
4. A multi-point calibration (minimum of five standards plus a calibration blank) must be generated each day of analysis. The calibration curve correlation coefficient must be equal to or greater than 0.995.
5. A drift-check standard will be analyzed between every 10 field samples and at the end of the analysis run.
6. A continuing blank check (CCB) will be analyzed between every 10 field samples and at the end of the analysis run.
7. Samples will be diluted as required to keep the analyte concentration ranges in the samples within the calibration range of the multi-point calibration curve.

9.6.7 TITRIMETRIC/ELECTROMETRIC METHODS

For each batch of samples analyzed for titrimetric parameters, the following QC checks will apply:

1. At least one method blank will be analyzed.
2. One LCS will be analyzed.
3. One sample duplicate will be analyzed.

9.7 MINIMUM QUALITY CONTROL - RADIOCHEMISTRY

The sections that follow summarize the minimum QC procedures that will be followed for each radiochemistry analysis methodology. These sections summarize the minimum QC requirements for the methodology; additional QC may be performed as necessary.

9.7.1 GAS FLOW PROPORTIONAL COUNTING METHODS

This classification of methods applies to the analysis of the following parameters: Gross Alpha and Beta, Radio-Sr, Radium-228, Total Alpha Emitting Radium, Radio-Pb, Radio-I, C-14, etc. For each batch of samples analyzed for these parameters, the following QC checks will apply:

1. A plateau will be run on a quarterly basis. Slope at operating will be less than 2.5 % / 100 volts. Discriminator will be set for each detector to optimize beta to alpha crosstalk to < 1%.

2. A configuration check will be conducted weekly to verify that instrument set-up is intact (software settings, gasflow, etc.)
3. A test specific initial calibration for efficiency, crosstalk and mass absorption will be run yearly, as appropriate. Efficiency calibrations will verify to within 15% of known values. Fitted points for absorption curves will fall to within 15% of observed values. Fitted points for crosstalk curves will fall to within 25% of observed values.
4. Instrument Daily Response checks will be run and will fall within 10% tolerance or +/- 3 sigma of historical performance for each detector employed each day when used.
5. The weekly 1000 minute background calibration will be current and acceptable (alpha < 0.5 cpm, beta < 3 cpm or +/- 3 sigma of historical performance) for each detector each day when used.
6. The daily 60 minute background check will be within +/- 3 sigma of historical performance acceptable limits for each detector employed each day when used.
7. Sample residue mass will be within method prescribed limits, where applicable, and within the range of the method calibration.
8. Sample residue distribution in the planchet will be uniform and representative of the calibration geometry applied to analysis (by visual inspection).
9. Method blanks will be analyzed at one per batch. Results will be less than or equal to the MDC, the Required MDC (RMDC) or 1/5 the associated sample activity.
10. Laboratory Control Samples will be analyzed at one per batch and will be within limits defined in Section 3.0.
11. Duplicate samples (of client samples when available, or LCSD) will be analyzed at one per batch or every 10 samples whichever is more frequent and will meet DER criteria (3 sigma < 1.43).
12. For those methods where yield correction is performed (Radiostrontium, Radium-228, Total Alpha Emitting Radium, Radiolead, Radioiodine, Carbon-14, etc.), the calculated chemical yield must fall in the range of 40 % - 110 %.

9.7.2 GAMMA/X-RAY SPECTROSCOPY METHODS

This classification of methods applies to the analysis of the following parameters:

Gamma Spec, Fe-55, Ra-226/228, etc. For each batch of samples analyzed for these parameters, the following QC checks will apply:

1. A weekly configuration check will verify that instrument set-up is intact (detector bias, gain, software, etc.) and meets expected parameters.
2. Initial calibrations for Energy and shape and a geometry specific calibration for efficiency will have been run within 12 months of detector use. Fitted points verify to within 1 keV of observed values for major lines for shape and energy calibrations and to within 10% of observed values for efficiency.
3. Instrument Daily Response Checks will be run and within acceptable limits (+/- 10% tolerance or +/- 3 sigma of historical performance) for the detector employed each day when used.
4. The monthly background calibration will be current and acceptable (will meet sensitivity requirements or +/- 3 sigma of historical performance) for the detector each day when used.
5. The weekly background check will be run and acceptable (+/- 3 sigma of historical performance) for the detector employed each day when used.
6. The sample geometry is equivalent to calibration geometry to be applied to analysis.
7. Method blanks will be analyzed at one per batch or 5% whichever is more frequent.
8. Laboratory Control Samples will be analyzed at one per batch and will be within limits defined in Section 3.0.
9. Duplicate samples (of client samples when available, or LCSD) will be analyzed at one per batch or every 10 samples whichever is more frequent and will meet DER criteria (3 sigma < 1.43).
10. For those methods where yield correction is performed (Radium-226/228), the calculated chemical yield must fall in the range of 40 % - 110 %.

9.7.3 ALPHA SPECTROMETRY METHODS

This classification of methods applies to the isotopic determination of actinides. For each batch of samples analyzed for these parameters, the following QC checks will apply:

1. A weekly configuration check will be conducted to verify that instrument set-up meets expected parameters (bias, energy range, vacuum, pump, etc.)
2. The weekly recalibration for energy will be current when detector used..
3. An initial calibration for efficiency will be run within 6 months of detector use. Points fall within 20% of expected range or +/- 3 sigma of historical performance.
4. Instrument Weekly Response Checks will be run and within acceptable limits (+/- 20% of calibrated value or +/- 3 sigma of historical performance) for the detector employed each day when used.
5. The weekly background calibration and check will be current and acceptable (sufficient to meet sensitivity requirements) for the detector each day when used.
6. The sample geometry is equivalent to calibration geometry to be applied. geometry applied to analysis.
7. Method blanks will be analyzed at one per batch. Activity will be less than MDC, RMDC or 1/5 of associated sample activity.
8. Laboratory Control Samples will be analyzed at one per batch and will be within limits defined in Section 3.0.
9. Duplicate samples (of client samples when available, or LCSD) will be analyzed at one per batch or every 10 samples which ever is more frequent and will meet DER criteria (3 sigma < 1.43).
10. Chemical yield meets acceptance criteria (20-110%).
11. Regions of Interest correspond to observed spectral peaks, tailing does not compromise quantitation.
12. No significant contaminant peaks are present which may compromise quantification.
13. Tracer solution used is current and traceable.

9.7.4 LIQUID SCINTILLATION METHODS

This classification of methods applies to the analysis of the following parameters: H-3, C-14, Ni-63, etc. For each batch of samples analyzed for these parameters, the following QC checks will apply:

1. Quarterly detector 'calibration' (normalization with C-14) will have been run.
2. A test specific initial calibration for efficiency and quench (where applicable) will be run within 12 months of detector use.
3. Instrument Daily Response checks will be run and within acceptable limits ($\pm 15\%$ or ± 3 sigma of historical performance) for the detector employed each day when used.
4. The daily background check will be run and within acceptable limits (± 3 sigma of historical limits) for the detector employed each day when used.
5. Sample quench is within the range of the method calibration (H# ± 15).
6. Method blanks will be analyzed at one per batch. Activity will be $< \text{MDC}$, RMDC or $1/5$ associated sample activity.
7. Laboratory Control Samples will be analyzed at one per batch and will be within limits defined in Section 3.0.
8. Duplicate samples (of client samples when available, or LCSD) will be analyzed at one per batch or every 10 samples whichever is more frequent and will meet DER criteria ($3 \text{ sigma} < 1.43$).
9. The reagent blank is entered into control table and is within acceptable limits (where applicable).
10. For those methods where yield correction is performed (Ni-63, etc.), the calculated chemical yield must fall in the range of 40 % - 110 %.

9.7.5 KINETIC PHOSPHORESCENCE ANALYSIS

This classification of methods applies to the determination of total uranium. For each batch of samples analyzed for these parameters, the following QC checks will apply:

1. Instrument configuration will be verified weekly
2. Reference Cell Solution will provide sufficient intensity (intensity > 10 cts/pulse)

3. Daily background calibration will be conducted
4. A three point calibration will be current for each range used. The correlation coefficient of the calibration curve will be > 0.995 .
5. IDL/MDL studies will be run quarterly and to support reporting limits.
6. Daily Second Source Continuing Calibration Verification is performed for each range used. The checks will be within 15 % of the known value.
7. A CCV and CCB will be run prior to initiation and following completion of an analytical run and between each ten client samples and will fall within 15% of the known value.
8. Samples will be diluted as required to maintain analyte concentration within the range of the calibration curve.
9. The Sample R-squared for samples containing measurable uranium will be greater than > 0.95 for each acceptable sample result.
10. The sample lifetime for samples containing measurable uranium will be between 200 and 350 us.
11. Method blanks will be analyzed at one per batch. Results will be $< RL$, $< RDL$ or $1/5$ associated sample concentration.
12. Laboratory Control Samples will be analyzed at one per batch and will be within limits defined in Section 3.0.
13. Duplicate samples (client samples where available, or LCS-DUP) will be analyzed at one per batch or every 10 samples which ever is more frequent. The RPD will be within limits defined in Section 3.0.
14. Matrix Spike samples will be analyzed at one per batch and matrix type. The MS recovery will be within limits defined in Section 3.0.

9.8 MINIMUM QUALITY CONTROL - ORGANICS

The sections that follow summarize the minimum QC procedures that will be followed for each inorganics analysis methodology. These sections summarize the minimum QC requirements for the methodology; additional QC procedures (such as derivitization, post-extraction fortification of samples, etc.) may be performed as necessary.

9.8.1 GAS CHROMATOGRAPHY

For each batch of samples analyzed by GC methods, the following minimum requirements will apply:

1. At least one method blank will be analyzed.
2. At least one control spike in a standard matrix at a preset level will be analyzed.
3. An initial multi-point calibration (five standards plus a calibration blank) must be generated for all methods. The calibration curve correlation coefficient must be equal to or greater than 0.995. Samples can be analyzed immediately after this curve is run, and quantitated against this curve.
4. A one-point calibration verification standard (usually a mid-level standard) may be analyzed and compared against the initial calibration curve. If the one-point calibration passes acceptance criteria, the analysis of samples may proceed, and sample results quantitated against the initial calibration curve.
5. One degradation check standard will be analyzed with every analysis run (pesticides).
6. Samples will be diluted as required to keep all analyte concentration ranges in the extracts within the calibration range of the initial multi-point calibration curve.
7. A mid-level standard will be analyzed between every 10 field samples and at the end of the analysis run. Acceptance criteria of this drift-check QC sample is method-specific.
8. At a minimum, one MS/MSD set will be analyzed with every batch.

9.8.2 GC DEGRADATION CHECK

Prior to sample analysis for pesticides, the laboratory will perform a degradation check standard analysis to verify that the degree of breakdown of endrin and DDT is within acceptance criteria. The degradation check standard will consist of a calibration solution that contains only endrin and DDT. This standard will be injected into the GC instrument and a chromatogram with peak table generated.

The percent degradation of endrin will be calculated from the peak areas of endrin, endrin aldehyde and endrin ketone using the following equation:

$$\text{Degradation}(\%)_{\text{endrin}} = \frac{\text{Response}_{\text{aldehyde}} + \text{Response}_{\text{ketone}}}{\text{Response}_{\text{endrin}} + \text{Response}_{\text{aldehyde}} + \text{Response}_{\text{ketone}}} \times 100$$

The percent degradation of DDT will be calculated from the peak areas of DDT, DDE and DDD using the following equation:

$$\text{Degradation}(\%)_{\text{DDT}} = \frac{\text{Response}_{\text{DDE}} + \text{Response}_{\text{DDD}}}{\text{Response}_{\text{DDT}} + \text{Response}_{\text{DDE}} + \text{Response}_{\text{DDD}}} \times 100$$

9.8.3 GAS CHROMATOGRAPHY/MASS SPECTROMETRY

For GC/MS analyses the following QC checks will apply:

1. All samples, method blanks and QC spikes will be spiked with the surrogates.
2. At least one control spike in a standard (blank) matrix with selected target analytes at a preset level will be analyzed. The matrix spike compounds for volatiles and base/neutral/acids are listed in the appropriate tables in Section 4.0 of this LQAP.
3. At least one method blank (spiked with all required surrogates) will be analyzed with each sample batch.
4. A continuing calibration check shall be run and checked against criteria for Response Factors (RFs) for System Performance Check Compounds (SPCCs) and for RPD requirements specified in the appropriate method for Calibration Check Compounds (CCCs). The specific compounds and their acceptance criteria are specified in the individual method (624, 8270, 524.2, etc.) or in the applicable CLP SOW.
5. A BFB or DFTPP tune will be performed and be within criteria prior to analysis (see specific method or SOW for acceptance criteria).
6. At a minimum, one MS/MSD set will be analyzed with every batch

9.8.4 HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

For each batch of samples analyzed by HPLC methods, the following minimum requirements will apply:

1. At least one method blank will be analyzed.
2. At least one control spike in a standard matrix at a preset level will be analyzed.

3. An initial multi-point calibration (five standards plus a calibration blank) must be generated for all methods. The calibration curve correlation coefficient must be equal to or greater than 0.995. Samples can be analyzed immediately after this curve is run, and quantitated against this curve.
4. A one-point calibration verification standard (usually a mid-level standard) may be analyzed and compared against the initial calibration curve. If the one-point calibration passes acceptance criteria, the analysis of samples may proceed, and sample results quantitated against the initial calibration curve.
5. Samples will be diluted as required to keep all analyte concentration ranges in the extracts within the calibration range of the initial multi-point calibration curve.
6. A mid-level standard will be analyzed between every 10 field samples and at the end of the analysis run. Acceptance criteria of this drift-check QC sample is method-specific.
7. At a minimum, one MS/MSD set will be analyzed with every batch.

9.8.5 SECOND COLUMN CONFIRMATION PROCEDURES

Second column confirmation is performed for a number of GC and HPLC analysis techniques. Whenever two different chromatography columns with similar performance are available for a given method, the laboratory will perform second column confirmation analysis to confirm the identity of target analytes in field samples. When second column analysis is performed for any chromatography technique, the following procedures will apply:

1. Every attempt will be made to calibrate the second (confirmatory) column in the same manner as the quantitative (primary) column. The same calibration standards, calibration check (calibration verification solutions, calibration blanks, etc.) should be analyzed on the confirmation column in the same manner as the quantitation column. The purpose of this dual calibration requirement is to allow the possibility of reporting quantitative results from the confirmation column if interferences on the primary column prevent target analyte quantitation.

2. Both a method blank and a laboratory control sample must be analyzed with the samples that are reanalyzed to confirm tentative identification of target analytes. It is not necessary to confirm any MS/MSD samples unless there are special circumstances that warrant their reanalysis. Confirmatory analysis of these two QC samples are used to verify consistent performance between the two columns, but QC results from the confirmation column are not reported in the summary reports in the data deliverable packages.
3. Confirmation of the presence of target analytes in a sample depends solely on peak retention times observed in both primary and secondary chromatograms. If target analyte peaks are present at the proper retention times in both confirmation and quantitation chromatograms, and both values are above the reporting limits, the Paragon will determine this to be a “confirmed” analyte. If either chromatogram has target analyte peaks that are below the reporting limit for that parameter, a value of “less than” the reporting limit will be reported by the laboratory.

9.8.6 RETENTION TIME WINDOW DETERMINATION

Retention time (RT) windows are calculated for each target analyte peak(s) and for each GC/HPLC column used for sample analysis. To establish RT windows, the laboratory measures the RTs of each analyte peak (or of each selected peak for multi-component analytes) from three analyses of the continuing calibration standard over a 72-hour period. The RT window is determined as ± 3 times the standard deviation of the three measured RTs. Daily RT windows are established for each analyte peak using the RT in the daily calibration verification standard as the centerpoint of the window determined above. In successive continuing calibration standards, the RT of each analyte peak must fall within the prescribed RT window for the analysis sequence to continue. RT windows must be recalculated whenever a different GC/HPLC column is installed.

Because ion chromatography techniques exhibit a strong correlation between analyte concentration and retention time, retention times must be determined in a different manner than described above. To determine retention time windows for ion chromatography, the

laboratory will run a minimum of two calibration curves that span the normal concentration range of the method. The analyst will then examine the retention times for all target analytes, and will determine the minimum and maximum retention times observed for each analyte. This spread in retention times will serve as the basis for determining the retention time window for each target analyte.

Results calculated in the manner(s) described above will be evaluated by the analyst. On occasion the retention time windows generated by these statistical processes can be unrealistic when assessed against practical experience in analyzing actual field samples. The analyst's judgment will be used as necessary to modify the windows generated as discussed above prior to their implementation. Once retention time windows are established, they will be uniformly applied in the evaluation of chromatographic data.

9.8.7 EVALUATION OF SURROGATES

For a number of organic analysis methods (See Table 9-6), the introduction of surrogates is mandated by the analysis protocol. Surrogate recoveries in samples will be evaluated by the analyst, and will be compared to historical performance data (where available) generated by Paragon. If the observed recovery of the surrogate is outside the ranges of historical performance for the laboratory, the corrective actions discussed in Section 13 of this LQAP will be followed.

For new methods, or methods in which a significant historical database is not available, recovery ranges from published sources will be used by Paragon for the evaluation of surrogates. With continued performance of new methods, surrogate recovery information will be compiled until such time as laboratory-specific ranges can be calculated and implemented at Paragon. For all procedures that require surrogate evaluation, advisory ranges will be recalculated incorporating the latest available data, and will be used to update the criteria shown in Table 9-6 each time this LQAP is revised.

9.9 MANUAL INTEGRATION PROCEDURES

Many of the data collection systems in use for chromatographic analyses at Paragon allow the analyst to post-process chromatograms, and allow for the manual re-integration of analyte peaks. Although Paragon will make every attempt to set up the data collection systems to correctly process the chromatographic data, manual reprocessing of this data is frequently performed by the laboratory staff. To verify that sound analytical judgment was used to perform manual integrations, additional documentation is required to be generated by the laboratory staff.

Whenever manual integrations are performed, the analyst performing this process must include a display of the area that includes the change. This will usually require the generation of an expanded display the integration of the affected peak. If sufficient detail is available to allow the expansion of a region where several integrations were manually performed, and still allow a subsequent reviewer to verify that sound analytical judgment was used in this process, it will be permissible to allow one expanded display to cover multiple integrations. This “blow-up” of the affected area will be included in the raw data package generated by the laboratory.

To date, four areas of instrumentation have been identified in the laboratory as having the possibility of manual integrations being performed: Gas Chromatography/Mass Spectrometry, Gas Chromatography, High Performance Liquid Chromatography and Ion Chromatography. The requirements to provide additional documentation for manual integrations applies uniformly to all of these instrumentation types.

Table 9-6 ORGANIC SURROGATES AND ADVISORY RECOVERY RANGES

Method	Surrogate	Percent Recovery (Water)	Percent Recovery (Soil)
Volatiles (GC/MS) ⁸	Toluene-d(8)	83-118	87-118
	Bromofluorobenzene	77-131	83-119
	Dibromofluoromethane	75-128	81-122
	1,2-Dichloroethane-d(4)	79-114	71-127
Fuels (GC) - Diesel/PID ⁸	Hexacosane	57-125	50-152
Volatiles (GC) - BTEX/8021/Gas ⁸	2,3,4-Trifluorotoluene	83-119	69-119
Semi-Volatiles ⁸ (GC/MS): Base/Neutrals	Nitrobenzene-D(5)	23-109	24-104
	2-Fluorobiphenyl	24-110	25-112
	Terphenyl-D(14)	14-129	21-141
	Phenol-D(5)	14-118	27-110
Acids	2-Fluorophenol	21-100	21-100
	2,4,6-Tribromophenol	12-106	20-116
	Polynuclear Arom. Hydro. (HPLC) ⁸	2-Chloroanthracene	40-115
Carbamate Pesticides ⁸	Neburon	34-114	N/A
Explosives ⁸	1,2-Dinitrobenzene (NG/PETN Only)	31-109	56-136
	1,4-Dinitrobenzene (Explosives)	31-121	59-139
Herbicides ⁹	Dichlorophenylacetic acid	42-129	47-154
Nitrogen/Phosphorous Pesticides ⁹	Triphenyl Phosphate	16-116	5-203
Organochlorine Pesticides ⁸	Tetrachlorometaxylene	45-125	35-135
	Decachlorobiphenyl	34-133	25-143

⁸ Source: Paragon Analytics historical data.

⁹ Recovery limits based on small data set. Larger data set being developed

10. DATA REDUCTION, VALIDATION AND REPORTING

Data transfer and reduction are essential functions in summarizing information to support conclusions. It is essential that these processes are performed accurately and, in the case of data reduction, accepted statistical techniques are used.

10.1 CORRECTION OF ERRORS IN DOCUMENTS

During the course of processing and reviewing sample analysis results, it may be necessary to correct documentation errors discovered during this process. To maintain the integrity of the documentation generated by the laboratory in order to meet potential litigation requirements, changes to documents must be made in the following manner:

1. A single line will be struck through the entry to be changed;
2. A new entry with the correct information will be made;
3. The date the change was made will be recorded; and
4. The initials of the person making the change will be entered.

10.2 DATA REDUCTION

Data reduction is performed by Paragon's individual analysts and consists of calculating concentrations in samples from the raw data obtained from the measuring instruments. The complexity of the data reduction will be dependent on the specific analytical method and the number of discrete operations (e.g., digestions, dilutions, or concentrations) involved in obtaining a sample that can be measured. The analyst will reduce or calculate all raw data into the final reportable values or enter all necessary raw data into the LIMS in order for the data base system to calculate the final reportable values.

Data reduction calculations used for most projects are typically included in the standard operating procedures developed by the laboratory and associated with each individual method or groups of methods. The complete calculation procedures used in computer-based data reduction are based on the calculation procedures specified in each method and will not be covered here.

Copies of all raw data and the calculations used to generate the final results, such as hard-bound laboratory notebooks, spreadsheets, electronic data files and LIMS record files will be retained in the project file to allow reconstruction of the data reduction process at a later date.

10.3 DOCUMENTATION OF RAW DATA

All manual documentation of raw data will be performed on appropriate forms or in notebooks. All notebooks used will be hard-bound with pre-numbered pages. All raw data entries shall be made in indelible ink.

Because of the special demands that apply to litigation-defensible documentation, there are several minimum requirements that apply to all raw data documents. At a minimum, all raw data must display the following information:

1. The date when the process was performed must be present.
2. The name of the staff member who performed the process must be present.
3. All samples or standard solutions that were processed must be clearly identified.
4. The methodology for which the samples were being processed must be clearly indicated.

10.4 DATA VALIDATION

The laboratory system for ensuring valid data includes several levels of review. Each level commands specific action to prevent the unqualified release of erroneous data and to correct any problems discovered during the review process.

All analytical data generated at Paragon Analytics, Inc. are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and three levels of review, as described below.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following

protocols specified in laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample preparation information is correct and complete.
- Analysis information is correct and complete.
- The appropriate SOPs have been followed.
- Analytical results are correct and complete and are compliant with program specifications.
- QC samples are within established control limits; blanks are acceptable.
- Special sample preparation and analytical requirements have been met.
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented; all manual integrations are signed and dated and “before and after” plots submitted; out of control forms, if required, are complete, holding times are documented, etc.).

This initial review step, performed by the analyst is designated Level 1 review. The analyst then passes the data package to an independent reviewer who performs a Level 2 review.

Level 2 review is performed by a group leader or data review specialist whose function is to provide an independent review of the data package. This review is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented.
- QC samples are within established guidelines.
- Qualitative identification of sample components is correct.
- Quantitative results are correct.

- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented, out-of-control forms, if required, are complete, holding times are documented, etc.).
- The data are ready for incorporation into the final report.
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed, and all of the analytical results from 10 percent of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is considered complete. If any problems are found with the data package, an additional 10 percent of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety. Level 2 data review is documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

Before the report is released to the client, the Laboratory Project Manager who is responsible for interfacing directly with the client reviews the report to ensure that the data meets the overall objectives of the project. This review is labeled Level 3 review.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data are consistently of high quality.

10.5 DATA REPORTING

Reports will contain final results (uncorrected for blanks and recoveries), methods of analysis, level of detection, and method blank data. For solid samples, sample values are reported in units of dry weight measure, with the exception of tritium analyses. In addition, special analytical problems, and/or any modifications of referenced methods will be noted. The number of significant figures reported in a result will be consistent

with the limits of uncertainty (reported to two significant figures) inherent in the analytical method. Consequently, most analytical results will be reported to no more than two or three significant figures. Where EPA Contract Laboratory Program (CLP) analyses and procedures are required by the client, the significant figure requirements specified in the CLP Statement of Work will be followed by Paragon.

Standard units of reporting for radiochemical analyses will relate to the level of activity of the sample per unit volume or weight. Typical units of reporting for these radioactivity measurements are:

1. Picocuries per gram or picocuries per liter (pCi/g or pCi/L).
2. Disintegrations per minute per gram or Disintegrations per minute per liter (DPM/g or DPM/L)
3. Becquerels per gram or Becquerels per liter (Bq/g or Bq/L)

Where:

$$1 \text{ Curie} = 2.22 * 10^{12} \text{ DPM}$$

and

$$1 \text{ Curie} = 3.7 * 10^{10} \text{ Bq}$$

Standard units of reporting for inorganic analysis will be in terms of units of mass per unit of weight or unit of volume. Typical units of reporting for these inorganic measurements are:

1. Milligrams per liter or micrograms per liter (mg/L or $\mu\text{g/L}$) for metals analysis results for aqueous samples (the latter units are most common), and milligrams per kilogram (mg/Kg) for metals in solid matrices.
2. Wet chemistry parameters (such as hardness, total organic carbon, total organic halides, total cyanide, etc.) are reported in units of milligrams per liter for aqueous matrices and milligrams per kilogram for solid matrices.
3. Miscellaneous parameters (such as pH, specific conductivity, flashpoint, etc.) will have specific reporting units mandated by their respective analysis technique.

Standard units of reporting for organic analysis will be in terms of units of mass per unit of weight or unit of volume. Typical units of reporting for these inorganic measurements are:

1. For total petroleum hydrocarbons (gasoline and/or diesel), usual reported units of measure are milligrams per liter for aqueous matrices and milligrams per kilogram for solid matrices.
2. For all other parameters, the reporting units for these analyses are micrograms per liter or micrograms per kilogram ($\mu\text{g/L}$ or $\mu\text{g/Kg}$).

10.5.1 FACSIMILE REPORTS

For projects which require unusually rapid turnaround of sample analysis results (i.e. results are to be reported in one week or less from sample receipt), the laboratory will normally provide a facsimile to the client, followed by the full data report at a later date. If the analysis results provided by facsimile have undergone the same review/validation processes normally followed for the normal data packages, the FAX report will indicate that the sample analysis results are final, and will be the same values that will be reported in the full data report.

However, if the accelerated turnaround time requirements preclude a full review/validation of the sample data, the report will be marked with a notation that it is "PRELIMINARY DATA" and may change as the review process is completed. Preliminary data is only reported in this fashion with prior agreement by the client.

10.5.2 ELECTRONIC DATA DELIVERABLES

The electronic data deliverables (EDDs) generated by the laboratory will be project-specific, and will be in a format specified by the project client. The laboratory's LIMS can generate a wide variety of formats, and Paragon's Information Services staff can create additional EDD formats as desired. All EDDs will be submitted to the client on 3.5" diskettes, unless a client specifically requests alternative means of transmittal.

The EDD files will be examined to ensure that they are an accurate reflection of the analysis data appearing in the hardcopy reports generated by the laboratory. This will be done with by reviewing of a printout of the EDD, and comparing it to the associated hardcopy reports. This review will be performed either by the laboratory staff that generated the analysis results, the Information Services staff or the Project Manager. During this process, the reviewer will verify that the information in the EDD matches the data reported on the hardcopy, and that the format requirements for the client EDD are met. Only after the completion of this review will the EDD be transmitted to the client.

10.5.3 HARDCOPY DATA PACKAGES

The format and content of a data report is dependent upon project needs, and it is beyond the scope of this document to describe project-specific report requirements. In the absence of client-specified data package deliverables, the following sections describe the items that must be included in all reports.

10.5.3.1 COVER LETTER

The cover letter shall be presented in block letter style and shall include:

1. Date the report was prepared.
2. The Paragon name, address, and telephone number.
3. Client name and address.
4. A tabular presentation of field/client sample ID, Paragon Sample ID, date received, matrix and date collected (presented as an attachment to the cover letter called Sample Cross Reference Table).
5. Discussion of previously submitted or partial reports that pertain to the samples discussed in the current report.
6. Discussion of any procedural anomalies or analytical non-conformances and corrective actions.
7. Signature of the Department Manager or Project Manager.

10.5.3.2 ANALYSIS REPORTS

Analysis reports are presented in tabular format, and consistent significant figures and units of measurement shall be used. In each analysis report, the following information shall be included:

1. Each page of data will be identified with the laboratory name, the client name, project name or number (if one exists).
2. Paragon sample ID and client/field sample ID (if different).
3. Dates that the samples were received, prepared and analyzed.
4. Sample collection dates (if available).
5. Sample matrix.
6. Parameters analyzed, results, units of measurement, and reporting limits.
7. Footnotes or qualifiers referenced to specific data (if required).
8. Explanations or keys to flags and abbreviations used.
9. Surrogate recoveries, where applicable.

10.5.3.3 QUALITY CONTROL REPORT

Each final report will include a quality control report which includes results from spike, duplicate and blank sample analysis. Additional QC information may be prepared to comply with contract or project-specific requirements.

10.6 DATA QUALIFIERS - FLAGGING CODES

Whenever the data quality objectives of this LQAP are not met, the associated sample results must be flagged with the appropriate flagging codes. These codes will be applied only in the event that the laboratory cannot generate (through reanalysis) fully compliant data. Additionally, if sample values fall outside the calibration range of the method (are below the reporting limit) or unresolvable interferences exist in the sample, descriptive flagging codes will be used to indicate the nature of the circumstances affecting sample results.

Data qualifiers shall be added by the laboratory prior to reporting the analysis results.

The laboratory shall apply data qualifying flags to each environmental field sample, based on an evaluation of all available QC information (e.g., ambient blanks, equipment blanks, trip blanks, field duplicates, matrix spike/matrix spike duplicate (MS/MSD) samples, laboratory blanks, laboratory control samples, calibration verification check samples, etc.). Analytical batch comments shall be added to the narrative section of each data report package to explain any non-conformance or other issues. The definitions of the data qualifiers used by Paragon are shown in Table 10-1 at the end of this section.

10.7 DATA STORAGE

All data reports, project files, raw analysis results (initial calibration, continuing calibration, standards verification, qc samples, forms, review checklists, etc.), logbooks, quality assurance records (e.g., retired SOPs, PE results, audit reports, training records, expired LQAPs), and related documentation are retained at the laboratory for a minimum of six months after the completion of a project. During this time, documents may be removed from the laboratory work areas and placed in a temporary storage location on-site. Access to these records is restricted to Paragon employees and for specific project or client records, designated representatives from the client's firm.

After the six-month temporary storage period, project documents will be sent off site to a record storage contractor employed by Paragon. Storage boxes will be filled with laboratory documents, inventoried, and sealed at Paragon. Chain-of-custody seals will be applied to the storage boxes when they are sealed, prior to shipment. The contents of each box will be entered into a electronic tracking system at Paragon that will facilitate retrieval when requested by the client. At the completion of this process, the data storage contractor (Data Management Record Services Inc. - DMRS) will be contacted and will take the storage boxes and place them in secured fire-proof storage areas at their storage facility. Coordination for the storage and retrieval of documents by DMRS is performed by Paragon's QA group.

DMRS maintains an inventory of all boxes from Paragon stored at their facility, and is responsible for the maintenance and protection of those records. Upon request from Paragon, DMRS will retrieve any of Paragon's records and deliver them to the laboratory on the next business day. As of this writing, no provisions have been made to destroy any records generated by the laboratory. Should Paragon destroy any records, written notification will be provided to all clients affected. At a minimum, records will be retained for five years. If a specific contractual requirement, project demand, or government regulation requires that records be maintained for a longer period of time, project files will be marked for longer retention and kept as required.

10.8 PROCEDURES FOR HANDLING UNACCEPTABLE DATA

All QC information will be recorded in the notebooks and printouts in the same format used for sample results. It is the analyst's responsibility to check the QC information against limits for the analysis. When an analysis of a QC sample (blank, spike, check standard, replicate, or similar sample) shows that the analysis of that batch of samples is not in control, the analyst will immediately bring the matter to the attention of the group leader. The group leader will, if necessary, consult with the Laboratory QA Manager and/or the laboratory Project Manager to determine whether the analysis can proceed, or if selected samples should be rerun, or specific corrective action needs to be taken before analyzing additional samples. Out-of-control analyses must be documented. The analyst or group leader will file a "Non-Conformance Report" with the laboratory QA Manager for lab analysis out of control events that require documentation.

If non-compliant data cannot be corrected, the affected results must be flagged as discussed in Section 10.6 above. The determination of which data qualifiers to be applied to the data will be the primary responsibility of the Project Manager, after consultation with the analytical Department Manager and the Laboratory's Quality Assurance Manager.

10.9 RESPONSE TO INQUIRIES

In the event that a client needs additional information to supplement the reports delivered to them, the Project Manager supporting the client's project will be contacted. It will be the project manager's responsibility to determine the client's needs for additional data, how to assemble the information, and to allocate laboratory resources to service the client's request. If necessary, the Project Manager will coordinate with the QA group to retrieve documents stored in the off-site storage facility.

If the results of the client's inquiry indicates that a new or updated report needs to be issued, the Project Manager will be responsible for coordinating and completing this effort. If amended analysis reports are to be sent to the client, the same data review and validation procedures described earlier in this section will be performed. The transmittal correspondence for the updated/revised report will contain all the relevant information as described in Section 10.5.3, and will clearly indicate whether the new report supersedes previous reports, amends them, or contains information that supplements earlier submissions.

Table 10-1 ORGANIC AND INORGANIC DATA QUALIFIERS

Qualifier ¹	Description
J (O)	The analyte was positively identified, the quantitation is an estimation.
U (I, O)	The analyte was analyzed for, but not detected. The associated numerical value is at or below the MDL.
B (O)	The analyte was found in an associated blank, as well as in the sample.
B (I)	The reported value was <RL but >MDL
M (I)	Duplicate injection precision was not met
S (I)	The reported value was determined by the Method of Standard Additions (MSA)
N (I)	Spiked sample recovery not within control limits
E (O)	Concentration exceeds upper level of the calibration range of the instrument
E (I)	The reported value is estimated because of an interference
D (O)	The value reported represents a dilution
A (O)	A tentatively identified compound that is a suspected aldol-condensation product
W (I)	Post-digestion spike for Furnace AA analysis is out of control limits, while sample absorbance is <50% of spike absorbance
I (O)	Surrogate recovery is not reported because of the presence of a chromatographic interferent, or a sample dilution caused surrogate not to be detected
I (I)	Interference by another parameter caused a dilution to be performed, and reporting limit is raised to account for this dilution
* (I)	Duplicate analysis not within control limits
+ (I)	Correlation coefficient for the Method of Standard Additions (MSA) <0.995

¹ Legend: I = Qualifier applies to inorganic analyses.
O = Qualifier applies to organic analyses.

Table 10-2 RADIOCHEMISTRY DATA QUALIFIERS

Qualifier	Footnote	Comments
J	Reported value is greater than the Instrument Detection Limit but less than the Method Detection Limit. The reported result is qualified as an estimation of due to additional uncertainty at this level. See narrative for discussion.	For KPA Tot U Reporting: when analytical result (w/o DL factor) exceeds the IDL but is less than the MDL.
U	Result is less than the sample specific minimum detectable activity.	For use in comparing to Currie MDA. The MDA should always be reported in association with activity and TPU.
B1	Analyte was detected in the associated method blank. The sample result is greater than 5 times the concentration measured in the blank.	Report for samples which show greater than 5 times method blank activity. Narrate. Flag affected result.
B2	Analyte was detected in the associated method blank. The sample result is less than 5 times that measured in the blank. See narrative for discussion.	Reprep or report result with appropriate qualification. Flag affected results.
B3	Analyte was detected in the associated method blank above the MDC but below the requested MDC.	Report result. Flag affected results.
YR	Chemical yield outside default limits. Result rejected. See narrative for discussion.	Result unusable by judgment of spectroscopist. Must narrate. Flag affected result.
Y2	Chemical yield outside default limits. Analyte quantitation is accurate to within report uncertainty.	Chemical yield outside control limits; spectroscopist determines that the result not compromised within stated uncertainty. Additional uncertainty may be estimated and accounted for. Must narrate and document. Flag affected result.
NH	Matrix spike recovery outside default limits (high). See narrative for discussion.	Determine whether cause isolated to one sample or matrix. Reprep or flag affected results.
NL	Matrix spike recovery outside default limits (low). See narrative for discussion.	Determine whether cause isolated to one sample or matrix. Reprep or flag affected results.
SQ	Spectral interference prevents accurate quantitation. See narrative for discussion.	For alpha, beta and gamma spectroscopy techniques. Identification is acceptable but interference compromises quantitation. Flag affected result.
SI	Identification is tentative due to spectral interference. See narrative for further discussion.	For alpha, beta and gamma spectroscopy techniques. Identification is not possible due to spectral interference. Flag affected result.
RQ	Rejected. Result does not meet quality control requirements. See narrative for discussion.	Result unusable for generally unspecified reasons. Must narrate. Flag affected result.

Qualifier	Footnote	Comments
R	Result rejected. See narrative for discussion.	Result does not pass QC or is otherwise deemed by the laboratory as unusable. Flag affected result.
TI	Nuclide identification is tentative. See narrative for discussion.	Tentative ID. Flag affected result.
MI	Sample result is estimated due to matrix interference. See narrative for discussion.	Matrix interference has compromised quantitation. Note in narrative. Flag affected result.
*	Duplicate analysis not within control limits.	DER (or RPD) does not meet criteria. Flag dup and associated sample.
S1	MDC elevated above requested value due to matrix interference from sample solids.	For gross alpha and beta: RMDA not met due to elevated sample solids. Flag affected results.
S2	MDC elevated due to matrix interference from sample solids. Activity detected above achieved MDC.	For gross alpha and beta: RMDA not met due to elevated sample solids but activity detected. Flag affected results.

11. PERFORMANCE AND SYSTEMS AUDITS

11.1 INTERNAL AUDITS

Two types of internal audit procedures will be used to assess and document performance of laboratory staff: systems audits and performance evaluation sample audits. These are performed at specified intervals under the direction of the QA Manager. These audits form one of the bases for corrective action requirements and constitute a permanent record of the conformance of measurement systems to QA requirements.

11.1.1 INTERNAL SYSTEMS AUDITS

This audit is general in nature, and provides an overview of laboratory operations. This type of audit must be performed at least once a month unless an external audit is performed during the same calendar month. The laboratory QA Manager will perform the laboratory system audit in accordance with checklists designed to aid the auditor in ensuring all areas of laboratory operations are reviewed. The scope of these audits may include the examination of the operations of a specific analytical department or may focus on the evaluation of a specific quality-related system as applied throughout the laboratory.

Examples of system-wide elements that may be audited include:

- Standard operating procedures, including system of review, issue, filing, maintenance, training, understanding, documentation of deviations and implementation of SOPs.
- Adherence to standard operating procedures, the LQAP and regulations.
- Personnel and training files, including job descriptions, resumes, documented training and training file maintenance.
- General laboratory safety, including appropriate clothing, waste disposal, health and safety plan review, obvious safety concerns.
- Labeling of reagents, solutions, standards, and associated documentation.
- Equipment and instrumentation documentation, calibration/
- maintenance records, operating manuals.

- Sample handling, storage and disposal including storage locations, security, tracking/chain-of-custody, disposal practices and records, labeling and retention.
- Documentation of sample analysis, methodologies, quality control requirements.
- Documentation of discrepancy reports and corrective action.
- General procedures for data security, review, documentation, reporting and archiving.

When the operations of a specific department are evaluated, a number of functions are reviewed, such as:

- Documentation of technical training and analyst proficiency
- Method detection limit studies
- Internal chain-of-custody documentation
- Nonconformance documentation
- Documentation of standard preparations
- Instrument maintenance documentation
- Standard operating procedures
- Control charts
- Documentation of sample preparation and analysis
- Documentation of data review

In addition, the QA Manager may monitor analyses randomly to assure adherence to approved analytical methods.

Audit results are reported in writing to responsible management for review and corrective action, if necessary. A maximum of two weeks is given to respond to the original report. The original copy of the completed report, with responses, is kept on file in the QA Department. QA personnel follow up by verifying the effectiveness of the implemented corrective action.

11.1.2 PERFORMANCE EVALUATION SAMPLE AUDITS

Paragon has utilized commercial third-party vendors of performance evaluation materials and has used the products of these vendors as single-blind PE samples. These type of performance evaluation samples are purchased in order to demonstrate competency in a newly developed method or to demonstrate effective corrective action. The results of Paragon's use of these commercial PE programs are evaluated in the same manner as the laboratory's participation in the federal PE programs. Deficiencies in the laboratory's performance will be addressed and corrected in the same manner as those used for federal PE programs. The Laboratory Quality Assurance Manager is responsible for coordinating the laboratory's participation in these PE Studies, and will maintain all documentation generated as a result of Paragon's participation in these programs.

11.2 EXTERNAL AUDITS

External audits may be performed by a state or federal agency or client as part of an on-going certification process, or as a result of Paragon's participation in specific programs/projects that require an external laboratory audit to be performed. External laboratory audits generally consist of reviews of analytical capabilities and procedures, chain-of-custody procedures, document control, QA/QC procedures, and laboratory organization. External audits usually include analysis of blind performance evaluation samples.

11.2.1 EXTERNAL SYSTEMS AUDITS

In addition to the internal audits described above, Paragon is regularly audited by the following agencies

1. State of Colorado Department of Health;
2. State of Utah Department of Health;
3. State of California Department of Health Services;
4. State of Arizona Department of Health Services;
5. US Army Corps of Engineers.

11.2.2 PERFORMANCE EVALUATION SAMPLE AUDITS

The results of interlaboratory studies will be evaluated by the QA Manager as part of the performance audits. The interlaboratory performance evaluation programs Paragon is currently participating are as follows:

1. US EPA Water Pollution Study Audit Program (WP).
2. US EPA Water Supply Study Audit Program (WS).
3. State of California Department of Health Services Hazardous Waste (HW) PE Program.
4. Department of Energy (DOE), Office of Environmental Management (OEM), Quality Assessment Program (QAP).
5. EPA National Exposure Research Laboratory (NERL), Characterization Research Division - Las Vegas (CRD-LV), Performance Evaluation Study.
6. Department of Energy (DOE), Idaho Operations Office, Mixed Analyte Performance Evaluation Program (MAPEP).
7. Environmental Resource Associates Proficiency Testing Program (quarterly).

11.3 LABORATORY CERTIFICATIONS

The following licenses, accreditations, and certifications are held by Paragon:

1. State of Arizona Department of Health Services;
2. State of Colorado Department of Health;
3. State of California Department of Health Service;
4. State of Idaho Department of Health and Welfare;
5. State of Kansas Department of Health and Environment;
6. State of Massachusetts Department of Health;
7. State of North Dakota Department of Health and Consolidated Laboratories;
8. State of Oklahoma Department of Environmental Quality;
9. State of South Carolina Department of Health and Environmental Control;
10. State of Tennessee Department of Health;
11. State of Utah Department of Health;
12. State of Washington Department of Ecology.

Paragon has been audited for compliance with and/or performs analyses for the following federal programs:

1. US Air Force Center for Environmental Excellence (AFCEE);
 2. US Army Corps of Engineers (USACE MRD);
 3. US Naval Facilities Engineering Service Center (NFESC);
 4. The Program Manager for the Rocky Mountain Arsenal (PMRMA);
 5. US Department of Energy, Rocky Flats Facility;
- The Los Alamos National Laboratory (on behalf of US DOE).

12. QUALITY ASSURANCE REPORTS TO MANAGEMENT

For day-to-day reporting, a Non-Conformance Report (NCR) is initiated for laboratory QA situations that require immediate attention. The employee that discovers the nonconformance is responsible for initiating the NCR. The Project Manager and QA Manager must approve the corrective action proposed. Client's guidance is solicited by the Project Manager if appropriate. The QA Manager distributes completed copies of the NCR to the Project Manager, Department Manager, and Operations Manager.

Quality Assurance Reports (QAR) to laboratory management are compiled and updated by the QA Manager every six (6) months. These reports summarize a wide range of QA activities. These activities may include, but are not limited to:

Performance Evaluation Studies. Results of both external and internal performance audits are distributed to laboratory line management for review and action, as appropriate. Any required response to deficiencies must be submitted to QA for review. After acceptable corrective action responses are received for all noted deficiencies, a summary of the results with attached responses is distributed.

Agency and Client On-Site Audits. On-site evaluations of laboratory facilities and procedures are conducted by external auditors to ensure conformance to the requirements of their respective programs. These are scheduled and coordinated through the QA Department. The summary evaluation received from the agency reporting findings and/or recommendations and observations are distributed through the QA Department to laboratory line management for review and action, as appropriate. Any required response to deficiencies must be submitted to QA for review. After acceptable corrective action responses are received for all noted deficiencies, a summary of the results with attached responses is distributed.

Technical Systems Audits. An assessment of the laboratory data quality objective for completeness is performed by QA personnel and evaluated based on reported deliverables for data precision, data accuracy, and completeness.

Management Systems Reviews. Thorough and efficient documented laboratory operational procedures and systems facilitate generation of a quality analytical product. Audits and surveillances of selected SOPs are conducted on a periodic basis by QA personnel to verify conformance. Based on these procedural reviews, a report is issued summarizing any findings and recommendations for revision in laboratory procedures to improve the analytical systems.

Employee Orientation and Training Activities. Orientation to Paragon's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Documentation of these activities is kept in individual files with the QA Department.

13. CORRECTIVE ACTIONS

Corrective action is necessary when any measurement system fails to follow this LQAP. Items which may need corrective action range from a minor problem of an analyst failing to sign a data report form to a major problem of an analyst using an improper analytical method. For this reason, corrective action protocols must be flexible.

In general, items needing corrective action fall into two “correction” categories: short-term and long-term; each requiring different action.

Short-term Corrective Actions--These actions consist of minor and major problems which can be corrected immediately. Examples include failure to date or sign a form, and errors in data entry. Corrective action is initiated by verbally calling attention to the problem followed by written notification.

Long-Term Corrective Actions--The actions consist of minor and major problems which require a series of actions to resolve the problem. The actions to be taken are coordinated by the Section Manager or QA Manager, and a Non-Conformance Report (Appendix D) is used to document the action. The report will describe the analysis involved, the data, analyst, the identification of all affected or suspect samples, probable cause, the corrective action measure(s) taken, and the final disposition/resolution of the problem. The report is to be signed by the chemist, the Section Manager and the QA Manager.

A copy of the completed form is retained by the QA Manager, with additional copies placed in the laboratory's raw data file and the project file.

13.1 NON-CONFORMANCE REPORT

Non-Conformance Reports (NCRs) are controlled documents that are administered by Paragon's Quality Assurance Group. These reports are sequentially numbered and tracked on a tracking log. When laboratory staff needs to initiate/complete an NCR, they will obtain a numbered original and enter pertinent information on the NCR tracking log.

The staff member will then complete the form by entering all pertinent information and the final disposition required to adequately address the Non-Conformance. Subsequent review/approval of the NCR will take place, with analysis group supervisory and QA reviews indicated with signatures in the appropriate areas on the form.

After all authorizations/reviews of the NCR have been completed, copies are made for the analytical group's records, and as needed for data package documentation. The original NCR form is returned to the Quality Assurance Group, who will log the receipt of the completed form into the NCR tracking log. The NCRs are then placed in sequential order in archive notebooks in the QA Manager's office for long-term storage/archival.

13.2 RESPONSIBILITIES FOR CORRECTIVE ACTION

When an out-of-control event is recognized, each individual involved with the analysis in question has an interactive role and responsibility.

13.2.1 ANALYST

The initial responsibility to monitor the quality of a function or analytical system lies with the individual performing the task or test. Quality indicators are evaluated against laboratory established or project-specific QA/QC requirements. If the evaluation reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. When an acceptable resolution cannot be met and/or data quality is negatively impacted, the analyst will notify the appropriate manager.

13.2.2 SECTION MANAGER

This staff member must review all analytical and QC data for reasonableness, accuracy and clerical errors. In an out-of-control event, the Section Manager works with the analyst and QA Manager to solve the problem and prevent the reporting of suspect data by stopping work on the analysis in question (if necessary) and insuring that all results that are suspect are repeated (if possible), after the source of the error is determined and remedied.

13.2.3 QUALITY ASSURANCE MANAGER

In the event that an out-of-control situation occurs that is unnoticed at the bench or supervisory level (e.g., a performance failure on a blind QC sample), the QA Manager will notify the Section Manager, help to identify and solve the problem where applicable, insure the work is stopped on the analysis and that no suspect data is reported. The QA Manager must also notify the appropriate Project Manager of the out-of-control situation.

13.2.4 PROJECT MANAGER

This project member must review results, events and their corrective actions for reasonableness and correlation to the project requirements, notify the client if an event affects the quality of the data, and work with the client to develop acceptable salvage alternatives.

13.3 SUMMARY OF CORRECTIVE ACTION PROCEDURES

The corrective action procedures that will be taken by the analytical staff following a failure to meet criteria specified in this LQAP are summarized in the following tables.

Table 13-1 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR GFAA ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Blank (ICB)	$\leq RL^1$ listed in Section 3.0	Rerun fresh blank solution. If still out of control, recalibrate the instrument.
Calibration Curve Correlation Coefficient	≥ 0.995	Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument or narrate why the data are acceptable.
Second source calibration verification standard (ICV)	$\pm 10\%$ of true value	Rerun standard. If still out of control, recalibrate instrument
Continuing calibration verification standard (CCV)	$\pm 10\%$ of true value	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV.
Continuing calibration blank (CCB)	$\leq RL$ listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples run since last acceptable CCB if necessary, or narrate why data are acceptable
Method Blank (MB)	$\leq RL$ listed in Section 3.0	Determine the cause of the blank problem, redigest set, if necessary, or narrate why data are acceptable.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, redigest and reanalyze samples, if necessary, or narrate why data are acceptable.
Sample duplicate (DUP)	See precision acceptance criteria in Section 3.0	If all other QC acceptable, flag data as a matrix homogeneity problem. If there are other QC failures, correct as required by other QC samples.
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	If MS/MSD not within limits, generate and analyze a post-digestion spike. If spike recovery is acceptable, flag data as a matrix homogeneity problem. If post-spike recovery is not acceptable, either dilute samples to remove interference, perform MSA on samples, or narrate why data are acceptable.

¹ RL = Reporting Limit.

Table 13-2 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR CVAA ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Blank (ICB)	\leq RL listed in Section 3.0	Rerun the blank. If still out of control, reprocess and reanalyze the blank.
Calibration Curve Correlation Coefficient	≥ 0.995	Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument or narrate why the data are acceptable.
Calibration verification standard (ICV)	$\pm 10\%$ of true value	Rerun standard. If still out of control, recalibrate instrument
Continuing calibration verification standard (CCV), second source	$\pm 20\%$ of true value	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV.
Continuing calibration blank (CCB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples run since last acceptable CCB if necessary, or narrate why data are acceptable
Method Blank (MB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, redigest set, if necessary, or narrate why data are acceptable.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, redigest and reanalyze samples, if necessary, or narrate why data are acceptable.
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag data as a sample homogeneity problem. If LCS is not in control, corrective action as listed for LCS.

Table 13-3 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR ICAP ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Curve Correlation Coefficient	≥ 0.995	Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument or narrate why the data are acceptable.
Initial calibration verification standard (ICV), second source	$\pm 10\%$ of true value	Rerun standard. If still out of control, recalibrate instrument
Calibration Blank (ICB)	\leq RL listed in Section 3.0	Rerun fresh blank solution. If still out of control, recalibrate the instrument.
Continuing calibration verification standard (CCV)	$\pm 10\%$ of true value	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptance CCV.
Continuing calibration blank (CCB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples run since last acceptable CCB if necessary, or narrate why data are acceptable
Interference Check Standards (ICSA/ICSAB)	$\pm 20\%$ of true value	Rerun check solution. If still out of control, recalibrate instrument, or regenerate interelement correction factors and reanalyze.
Method Blank (MB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, redigest set, if necessary, or narrate why data are acceptable.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, redigest and reanalyze samples, if necessary, or narrate why data are acceptable.
Sample duplicate (DUP)	See precision acceptance criteria in Section 3.0	If all other QC acceptable, flag data as a matrix homogeneity problem. If there are other QC failures, redigest samples and reanalyze.
Serial Dilution	$\pm 10\%$ of undiluted sample value	Note exceedence in narrative
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	Analyze a post-digestion spike. If spike recovery is acceptable, flag data as a matrix homogeneity problem. If post-digestion spike not acceptable, include discussion in narrative.

Table 13-4 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR ION CHROMATOGRAPHY ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Curve Correlation Coefficient	≥ 0.995	Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument or narrate why the data are acceptable.
Initial calibration verification standard (ICV)	$\pm 10\%$ of true value	Rerun standard. If still out of control, recalibrate instrument
Continuing calibration verification standard (CCV)	$\pm 10\%$ of true value	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV.
Continuing calibration blank (CCB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples since last acceptable CCB if necessary, or narrate why data are acceptable
Method Blank (MB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples if necessary, or narrate why data are acceptable.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reanalyze samples if necessary, or narrate why data are acceptable.
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag data as a sample homogeneity problem. If LCS is not in control, corrective action as listed for LCS.

Table 13-5 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR COLORIMETRIC ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Curve Correlation Coefficient	≥ 0.995	Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument or narrate why the data are acceptable.
Initial calibration verification standard (ICV)	$\pm 15\%$ of true value	Rerun standard. If still out of control, recalibrate instrument before sample analysis.
Continuing calibration verification standard (CCV)	$\pm 15\%$ of true value	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV.
Continuing calibration blank (CCB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples since last acceptable CCB if necessary, or narrate why data are acceptable
Method Blank (MB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples if necessary, or narrate why data are acceptable.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reanalyze samples if necessary, or narrate why data are acceptable.
Sample Duplicate (DUP) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag data as a sample homogeneity problem. If LCS is not in control, corrective action as listed for LCS.
Sample matrix spike (MS) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, qualify the data as a sample homogeneity problem. If LCS is not in control, corrective action as listed for LCS.

Table 13-6 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR AUTOANALYZER ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Curve Correlation Coefficient	≥ 0.995	Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument or narrate why the data are acceptable.
Continuing calibration verification standard (CCV)	$\pm 10\%$ of true value	Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV.
Method Blank (MB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples if necessary, or narrate why data are acceptable.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reanalyze samples if necessary, or narrate why data are acceptable.
Sample Duplicate (DUP)	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag data as a sample homogeneity problem. If LCS is not in control, corrective action as listed for LCS.

Table 13-7 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR
TITRIMETRIC ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Method Blank (MB)	\leq RL listed in Section 3.0	Determine the cause of the blank problem, reanalyze samples if necessary, or narrate why data are acceptable.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reanalyze samples if necessary, or narrate why data are acceptable.
Sample Duplicate (DUP)	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag data as a sample homogeneity problem. If LCS is not in control, corrective action as listed for LCS.

Table 13-8 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR GC/MS VOLATILES ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
BFB instrument tune	Per method requirements	Retune instrument until within criteria.
Initial Calibration	System Performance Check (SPC) and Calibration Check (CCC) criteria are met.	Rerun calibration standards. If still outside criteria, prepare new standards and recalibrate.
One-point calibration	System Performance Check (SPC) and Calibration Check (CCC) criteria are met.	Rerun calibration standard. If still out of control, generate new initial calibration curve.
Internal Standards (IS)	50% to 200% of IS responses of calibration standard	Reanalyze once. If same results generated, matrix effect has been verified & flag data. If acceptable results obtained, report reanalysis data.
Method Blank (MB)	\leq RL listed in Section 3.0 except for MeCl ₂ & acetone ($\leq 5 \times$ RL)	Reanalyze blank. If acceptance criteria still not met, reanalyze samples or narrate why data are acceptable. Flag sample data as appropriate.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reprepare and reanalyze samples, if necessary, or narrate why data are acceptable.
Surrogate	See acceptance limits in Section 10.8.7	Reanalyze once. If same results generated, matrix effect has been verified & flag data. If acceptable results obtained, report reanalysis data.
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag data as a sample homogeneity problem. If LCS is not in control, corrective action as listed for LCS.

Table 13-9 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR GC/MS SEMI-VOLATILES ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
DFTPP instrument tune	Per method requirements	Retune instrument until within criteria.
Initial Calibration	System Performance Check (SPC) and Calibration Check (CCC) criteria are met.	Rerun calibration standards. If still outside criteria, prepare new standards and recalibrate.
One-point calibration	System Performance Check (SPC) and Calibration Check (CCC) criteria are met.	Rerun calibration standard. If still out of control, generate new initial calibration curve.
Internal Standards (IS)	50% to 200% of IS responses of calibration standard	Reanalyze once. If same results generated, matrix effect has been verified & flag data. If acceptable results obtained, report reanalysis data.
Method Blank (MB)	≤ RL listed in Section 3.0 except for phthalates (≤ 5 x RL)	Reextract and reanalyze samples if holding times are not expired. If acceptance criteria not met, flag sample data as appropriate.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reextract and reanalyze samples if holding times are not expired. If acceptance criteria can not be met, flag data as appropriate.
Surrogate	See acceptance limits in Section 10.8.7	Reanalyze once. If same results generated, matrix effect has been verified & flag data. If acceptable results obtained, report reanalysis data.
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag the data appropriately. If LCS is not in control, corrective action as listed for LCS.

Table 13-10 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR GC SEMI-VOLATILES ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Curve Correlation Coefficient	$\geq .995$	Rerun calibration standards. If still outside criteria, prepare new standards and recalibrate.
Continuing Calibration Verification Standard (ICV)	$\pm 15\%$ of target values when calculated from initial multi-point calibration curve	Rerun calibration standard. If still out of control, generate new initial calibration curve.
Degradation Check (Pesticides only)	$\pm 15\%$ degradation max for endrin and DDT individually (8081); $\pm 20\%$ degradation max for endrin and DDT individually (8080)	Reanalyze degradation check. If still out of control, trim column, replace injection liner, etc., and recalibrate and reanalyze all samples.
Continuing Calibration Check Standard (CCV)	$\pm 15\%$ of target values when calculated from initial multi-point calibration curve	Rerun check standard. If still out of control, stop analysis, correct problem, recalibrate, and reanalyze all samples run since last compliant CCV.
Method Blank (MB)	\leq RL listed in Section 3.0	Reextract and reanalyze samples if holding times are not expired. If acceptance criteria not met, flag sample data as appropriate.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reextract and reanalyze samples if holding times are not expired. If acceptance criteria can not be met, flag data as appropriate.
Surrogate (if applicable)	See acceptance criteria in Section 10.8.7	Reanalyze once. If same results generated, matrix effect has been verified & flag data. If acceptable results obtained, report reanalysis data.
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag the data appropriately. If LCS is not in control, corrective action as listed for LCS.

Table 13-11 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR GC VOLATILES ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Curve Correlation Coefficient	$\geq .995$	Rerun calibration standards. If still outside criteria, prepare new standards and recalibrate.
Continuing Calibration Verification Standard (ICV)	$\pm 15\%$ of target values when calculated from initial multi-point calibration curve	Rerun calibration standard. If still out of control, generate new initial calibration curve.
Continuing Calibration Check Standard (CCV)	$\pm 15\%$ of target values when calculated from initial multi-point calibration curve	Rerun check standard. If still out of control, stop analysis, correct problem, recalibrate, and reanalyze all samples run since last compliant CCV.
Method Blank (MB)	\leq RL listed in Section 3.0	Reextract and reanalyze samples if holding times are not expired. If acceptance criteria not met, flag sample data as appropriate.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reprepare and reanalyze samples if necessary. If acceptance criteria can not be met, flag data as appropriate and/or narrate why data are acceptable.
Surrogate	See acceptance criteria in Section 10.8.7	Reanalyze once. If same results generated, matrix effect has been verified & flag data. If acceptable results obtained, report reanalysis data.
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag the data appropriately. If LCS is not in control, corrective action as listed for LCS.

Table 13-12 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR HPLC ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Calibration Curve Correlation Coefficient	$\geq .995$	Rerun calibration standards. If still outside criteria, prepare new standards and recalibrate.
Continuing Calibration Verification Standard (ICV)	$\pm 15\%$ of target values when calculated from initial multi-point calibration curve	Rerun calibration standard. If still out of control, generate new initial calibration curve.
Continuing Calibration Check Standard (CCV)	$\pm 15\%$ of target values when calculated from initial multi-point calibration curve	Rerun check standard. If still out of control, stop analysis, correct problem, recalibrate, and reanalyze all samples run since last compliant CCV.
Method Blank (MB)	\leq RL listed in Section 3.0	Reextract and reanalyze samples if holding times are not expired. If acceptance criteria not met, flag sample data as appropriate.
Laboratory Control Sample (LCS)	See acceptance criteria in Section 3.0	Determine and correct problem, reextract and reanalyze samples if holding times are not expired. If acceptance criteria can not be met, flag data as appropriate.
Surrogate (if applicable)	See acceptance criteria in Section 10.8.7	Reanalyze once. If same results generated, matrix effect has been verified & flag data. If acceptable results obtained, report reanalysis data.
Sample matrix spike & sample matrix spike duplicate (MS/MSD) [if required]	See acceptance criteria in Section 3.0	If LCS is within acceptance criteria, flag the data appropriately. If LCS is not in control, corrective action as listed for LCS.

Table 13-13 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR GAS FLOW PROPORTIONAL ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Plateau	Run quarterly, following major maintenance or as indicated by change in instrument performance	Stop analysis; run plateau, reset operating voltage if slope at old operating voltage exceeds acceptable limit; or narrate why condition is acceptable
Operating Voltage	Slope < 2.5 % / 100 volts at operating voltage	Reset operating voltage according to plateau determination, reset discriminators, recalibrate; Determine and correct problem; or Narrate why condition is acceptable
Discriminator Optimization	Beta to alpha cross talk <1%	Optimize crosstalk settings and recalibrate; Determine and correct problem; or Narrate why condition is acceptable
Configuration	Check gas flow, cables, software settings, etc.	Determine and correct problem; or Narrate why condition is acceptable
Test Specific Initial Efficiency	Run yearly, after major maintenance or as indicated by change in instrument performance: verifies at +/-10% of expected	Reject outliers, remeasure; Reprepare sources and/or recalibrate instrument, verify calibration; Determine and correct problem; or Narrate why condition is acceptable
Test Specific Initial Self-Absorption / Crosstalk Calibration Curves	Run yearly, after major maintenance or as indicated by change in instrument performance: efficiency +/-15% of fitted values; crosstalk +/- 25% of fitted values.	Reject Outliers, remeasure; Reprepare sources and/or recalibrate instrument, verify calibration; or Narrate why condition is acceptable
Daily Response Check	Within +/- 10% tolerance or +/- 3 sigma of historical performance.	Check P-10 and source integrity, repeat check; Tag detector off-line; Repeat check following day; Determine and correct problem, run plateau/set discriminator, reestablish limits, recalibrate; or Narrate why condition is acceptable
Weekly 1000 minute background check	Current and acceptable (alpha < 0.5 cpm, beta < 3 cpm or +/- 3 sigma of historical performance)	Replace planchet, check P-10, recount; Tag detector off-line; Repeat check following day; Determine and correct problem, reestablish limits; or Narrate why condition is acceptable
Daily 60 minute background check	+/- 3 sigma of historical performance	Replace with clean planchet, check P-10, recount; Repeat check; Tag detector off-line; Repeat check following day; Determine and correct problem, reestablish limits; or Narrate why condition is acceptable
Sample Residue	Gross alpha <100 mg, gross beta <200 mg, otherwise within calibration range	Reprep overweight sources, redistribute or reprep poorly distributed sources; or Qualify or narrate why condition is acceptable
Method blank	One per batch; Activity less than MDC, RMDC or 1/5 associated sample activity	Reprep affected batch or samples; Determine and correct problem; or qualify or narrate why condition is acceptable
Lab Control Sample (LCS)	One per batch; See acceptance criteria in Section 3.0	Reprep; or Recalibrate and recount; or Qualify or narrate why condition is acceptable
Duplicate Sample	One per batch or greater than every 10 samples: 2 sigma DER<1.42, 3 sigma DER<1.42	Narrate 2 sigma failure when > 2x MDC; Reprep and/or recount affected samples for 3 sigma failure of >80% of nuclides; Determine and correct problem; or Qualify or narrate why condition is acceptable
Chemical yield	40-110%, where applicable	Reprep; Notify client, or Qualify or narrate why condition is acceptable

Table 13-14 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR GAMMA/X-RAY SPECTROSCOPY ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Configuration	Check bias, gain, etc, weekly	Adjust, verify calibrations
Initial Energy and Shape Calibration	Fitted values verify within 1 keV of observed	Check source position, detector configuration, recount and reprocess; Determine and correct problem; or Narrate why condition is acceptable
Initial Efficiency Calibration	Fitted values verify within +/- 10% of observed	Check source position, detector configuration, recount and reprocess; Determine and correct problem; or Narrate why condition is acceptable
Daily Response Check	+/- 10% tolerance or +/- 3 sigma of historical performance for all long-lived nuclides.	Check source position, detector configuration, recount and reprocess; Determine and correct problem; or Narrate why condition is acceptable
Monthly 120000 sec. background calibration	Satisfactory to meets sensitivity requirements or +/- 3 sigma of historical performance	Check shield operation, clean cave, repeat check; Tag detector off-line; Determine and correct problem, reestablish limits; or Narrate why condition is acceptable
Weekly 60000 sec background check	Satisfactory to meets sensitivity requirements or +/- 3 sigma of historical performance	Check shield operation, clean cave, repeat check; Tag detector off-line; Determine and correct problem, reestablish limits; or Narrate why condition is acceptable
Method blank	One per batch or 20 samples; Activity less than MDC, RMDC or 1/5 associated sample activity	Recount; Reprep and/or recount affected batch or samples; Determine and correct problem; or Qualify or narrate why condition is acceptable
Lab Control Sample (LCS)	One per batch in sample geometry; supplied in count room; See acceptance criteria in Section 3.0	Recount; Reprep and/or recount affected batch or samples; Determine and correct problem; or Qualify or narrate why condition is acceptable
Duplicate Sample	One per batch or greater than every 10 samples: 2 sigma DER<1.42, 3 sigma DER<1.42 for 80% of nuclides	Narrate 2 sigma failure for nuclides > than 2 X MDC; Reprep and/or recount affected samples for 3 sigma failure of >80% of nuclides; Determine and correct problem; or Qualify or narrate why condition is acceptable
Chemical yield	40-110%, where applicable	Reprep; or Qualify or narrate why condition is acceptable

Table 13-15 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR ALPHA SPECTROSCOPY ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Configuration	Check bias, energy range, pump, etc, weekly	Adjust, repair.
Weekly Energy Calibration and Check	Slope within expected range	Check source integrity, detector configuration, recount and reprocess; Tag detector off-line; Determine and correct problem; or Narrate why condition is acceptable
Weekly Efficiency Calibration and Check	Fitted values verify within +/- 20% of expected	Check source position and integrity, detector configuration, recount and reprocess; Tag detector off-line; Determine and correct problem; or Narrate why condition is acceptable
Weekly 60000 sec. background calibration	Satisfactory to meets sensitivity requirements	Replace bkg filter, clean chamber and detector, repeat check; Tag detector off-line; Determine and correct problem, reestablish limits; or Narrate why condition is acceptable
Method blank	One per batch or 20 samples; Activity less than MDC, Required MDC or 1/5 associated sample activity	Recount; Reprep and/or recount affected batch or samples; Determine and correct problem; or Qualify or narrate why condition is acceptable
Lab Control Sample (LCS)	One per batch; See acceptance criteria in Section 3.0	Recount; Reprep and/or recount affected batch or samples; Determine and correct problem; or Qualify or narrate why condition is acceptable
Duplicate Sample	One per batch or greater than every 10 samples: 2 sigma DER<1.42, 3 sigma DER<1.42	Narrate 2 sigma failure for nuclides > than 2 X MDC; Reprep and/or recount affected samples for 3 sigma failure; Determine and correct problem; or Qualify or narrate why condition is acceptable
Chemical yield	20-110%	Reprep; or Qualify or narrate why condition is acceptable
Regions of Interest	Properly set, tailing does not compromise quantitation	Adjust ROI's to fit identified peaks; For tailing, cleanup or reprep and recount affected samples, Consult with supervisor of Technical Manager; Correct for tailing; Qualify or narrate why condition is acceptable
Spectral Interferences	Interfering activity does not compromise quantitation	Recount; Cleanup, reprep and/or recount affected samples; Consult with supervisor or technical manager; Determine and correct problem; or Qualify or narrate why condition is acceptable
Tracer Solution	Current and traceable	Replace, recertify

Table 13-16 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR LIQUID SCINTILLATION ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Configuration	Check bias, energy range, pump, software settings, etc. weekly	Adjust, repair.
Quarterly normalization	Current, daily checks verify stability	Check source integrity, detector configuration, recount and reprocess; Tag instrument off-line; Determine and correct problem; or Narrate why condition is acceptable
Daily Response Check	Fitted values verify within +/- 10% of expected or +/- 3 sigma of historical performance	Check source integrity, detector configuration, recount and reprocess; Tag detector off-line; Determine and correct problem; or Narrate why condition is acceptable
Yearly Efficiency Calibration	Fitted values verify within +/- 10% of expected	Check source integrity, detector configuration, recount and reprocess; Tag detector off-line; Determine and correct problem; or Narrate why condition is acceptable
Daily background check	Satisfactory to meets sensitivity requirements or +/- 3 sigma of historical performance	Clean source, repeat check; Tag detector off-line; Determine and correct problem, reestablish limits; or Narrate why condition is acceptable
Method blank	One per batch or 20 samples; Activity less than MDC, Required MDC or 1/5 associated sample activity	Recount; Reprep and/or recount affected batch or samples; Determine and correct problem; or Qualify or narrate why condition is acceptable
Lab Control Sample (LCS)	One per batch supplied in count room; See acceptance criteria in Section 3.0	Recount; Reprep and/or recount affected batch or samples; Determine and correct problem; or Qualify or narrate why condition is acceptable
Duplicate Sample	One per batch or greater than every 10 samples: 2 sigma DER<1.42, 3 sigma DER<1.42	Narrate 2 sigma failure for nuclides > than 2 X MDC; Reprep and/or recount affected samples for 3 sigma failure; Determine and correct problem; or Qualify or narrate why condition is acceptable
Chemical yield	40-110%, where applicable	Reprep; or Qualify or narrate why condition is acceptable
Reagent Blank	Within criteria	Reprep, remeasure, Qualify or narrate why condition is acceptable

Table 13-17 SUMMARY OF CORRECTIVE ACTION PROCEDURES FOR KINETIC PHOSPHORESCENCE ANALYSES

Quality Control	Acceptance Criteria	Corrective Action
Configuration	Check, software settings, etc. weekly	Adjust.
Reference Cell Intensity	>10 cts/pulse	Renew solution; Renew Dye in Dye Laser; Replace cartridge; Align optics; Determine and correct problem; or Narrate why condition is acceptable
Daily Background Calibration	CCV and CCB acceptable	Repeat calibration, Clean cuvette and remeasure; Determine and correct problem; or Narrate why condition is acceptable
Calibration	$R^2 > 0.995$	Recalibrate; Determine and correct problem; or Narrate why condition is acceptable
IDL/MDL/RL	Quarterly	Perform study, narrate
Continuing calibration verification	+/- 15 %	Recalibrate; Determine and correct problem; or Narrate why condition is acceptable
Continuing calibration blank	< RL	Recalibrate; Determine and correct problem; or Narrate why condition is acceptable
R^2	>0.95 where uranium present	Dilute, remeasure; Qualify or narrate why condition is acceptable
Lifetime	200 - 350 us where uranium present	Dilute, remeasure; Qualify or narrate why condition is acceptable
Method blank	one per batch or 20 samples; Activity less than RL , RDL or 1/5 associated sample concentration	Reprep and/or recount affected batch or samples; Determine and correct problem; or Qualify or narrate why condition is acceptable
Lab Control Sample (LCS)	one per batch; See acceptance criteria in Section 3.0	Recount; Reprep and/or recount affected batch or samples; Determine and correct problem; or Qualify or narrate why condition is acceptable
Duplicate Sample	one per batch or every 10 samples. See acceptance criteria in Section 3.0	Reprep affected batch or Determine and correct problem; or Qualify or narrate why condition is acceptable
Matrix Spike	one per batch and matrix type; See acceptance criteria in Section 3.0	Qualify or narrate why condition is acceptable

14. PERSONNEL TRAINING

Qualifications of personnel are based upon education and experience. The selection of well-qualified personnel is an important step in the operation of Paragon. In order to maintain staff qualification and provide for personnel advancement within the laboratory, Paragon follows a formal documented program of orientation and training.

14.1 ORIENTATION

Each new regular employee receives a three part orientation: a human resources orientation, a safety orientation, and a department orientation. The human resources orientation involves matters of immediate personal concern such as benefits, salary, and company policies. The safety orientation is an in-depth examination of the Paragon Chemical Hygiene Plan and safety program, which are consistent with the requirements of OSHA's Hazard Communication Program (29 CFR 1910.1200), Paragon's Radiation Safety Plan, and the Colorado Department of Public Health's Rules and Regulations pertaining to Radiation Control. The new employee's Department Manager provides the employee with a basic understanding of the role of the laboratory within the structure of Paragon and the basic elements of that individual's position within the laboratory. The training of a new employee concentrates on his/her scientific background and work experience to provide the employee with a level of competence so that the individual will be able to function within the defined responsibilities of his/her position immediately.

Temporary employees receive the same orientation as regular staff with the exception of the human resources orientation.

14.2 ANALYTICAL TRAINING

Analysts/technicians shall be qualified to perform specific analytical procedures and methods. The qualification process, at a minimum, is described in the Standard Operating Procedure - "Training and Technical Review" and typically consists of background/theory, a documented process of on the job training, and a demonstration of proficiency. Additional training techniques utilized include:

- On-the-job training
- Lectures
- Programmed learning
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Participation in check-sample or proficiency sample programs

Department Managers shall be responsible for providing documentation of training and proficiency for each employee under their supervision. The files shall also include examples demonstrating performance of passing QC samples. The QA department shall maintain a file for each technical employee.

14.2.1 ANALYTICAL METHOD TRAINING

New analytical staff members are trained by the analytical group leaders under the following guidelines:

1. The new employee is taken through (observes) an analytical procedure in which the analytical method is demonstrated by trained personnel. Job requirements are outlined and quality control measurements are defined. A copy of the method is given to the employee for them to review prior to beginning the analysis.
2. The procedure is performed (on QC samples only) by the trainee who will work under the supervision of the analytical group leader or an analyst proficient in that method. Results of sample preparation and/or analysis are evaluated and problems or corrective actions discussed.
3. The trainee is then required to perform the procedure on laboratory blanks and standard matrix spikes (LCS samples). The results of these samples are reviewed by the analytical department leader. If the blank and spike recovery data meets existing quality control criteria for that method, the technician or analyst is deemed to have demonstrated proficiency and is allowed to work on actual client samples. If values

are outside the current acceptance limits, steps 2 and 3 are repeated until the trainee can consistently meet the acceptance criteria for the method. After the certification process has been successfully completed, the analytical department leader will forward the documentation to the QA group for inclusion into the employee's training file.

All analysts and technicians are monitored on a continual basis to evaluate performance. Should out of control situations which cannot be readily rectified appear, the laboratory manager can, at his discretion, "de-certify" personnel and require the individual to be retrained according to above steps 2 and 3.

14.2.2 PROFICIENCY DEMONSTRATION

Paragon's analytical staff is continually challenged to demonstrate their proficiency in the methods and processes they perform. Other sections of this LQAP describe the routine procedures that are followed by the laboratory during the day-to-day processing of samples that demonstrate the laboratory is generating analysis data that meets all analytical requirements. However, there are other, non-routine procedures that are performed by the laboratory's staff that demonstrates their proficiency in their assigned tasks. The following sections describes these non-routine proficiency demonstration procedures

14.2.2.1 METHOD DETECTION LIMITS

Most analytical methods require the periodic regeneration of method detection limit data, and this process has been fully described in earlier sections of this LQAP. The generation of acceptable detection limits by the laboratory requires a thorough understanding of the total analytical process, and is a rigorous test of the proficiency of the analytical staff who perform this analysis. With the permission of the analytical department manager and the Quality Assurance Manager, an analyst's or technician's performance in a method detection limit study (that generates method detection limit values that are consistent with past performance) may be used to demonstrate proficiency in the method. This information may be used in lieu of other demonstrations of proficiency, except when a

regulatory promulgated method explicitly requires specific procedures to be followed for the initial demonstration of proficiency.

14.2.2.2 PERFORMANCE EVALUATION SAMPLES

As described in an earlier section of this LQAP (titled “Performance and Systems Audits”), Paragon participates in several performance evaluation programs. These programs typically submit single-blind samples to the laboratory for analysis and return a summary of performance to Paragon after results have been furnished to the sponsoring agency.

The QA Manager distributes Paragon’s results to each department for review. For results within the defined acceptance limits, no action is required. If results are outside acceptance limits, then the appropriate Department Supervisor conducts an investigation to determine the root cause of the error. The Department Supervisor summarizes the findings and corrective action in a report to the QA Manager. The QA Manager summarizes all results for the sponsoring agency.

The successful participation in these programs by laboratory staff are rigorous tests of the staff’s ability to perform their analytical procedures. Paragon considers 90% to be a minimum acceptable score for each analytical procedure. Records of their successful participation in these programs will be placed in the analytical staff’s training records, and may be used to show that they have been adequately trained in the methods they have performed. This information may be used in lieu of other demonstrations of proficiency, except when a regulatory promulgated method explicitly requires specific procedures to be followed for the initial demonstration of proficiency.

14.2.2.2 INITIAL DEMONSTRATION OF PROFICIENCY

A number of promulgated methods performed by Paragon contain sections that explicitly state the procedures that are to be followed to verify the capability of the laboratory in performing the method, prior to the analysis of actual field samples. Some of these

methods require that laboratory staff generate initial proficiency demonstration data showing that they can meet specific acceptance criteria for precision and accuracy. This initial precision and recovery evaluation (IPR) will be performed initially by any laboratory staff as they first begin to perform one of these methods.

The IPR will be performed by preparing a set of four replicate standard matrix spikes (blank spikes) at a concentration in the middle of the reporting range for each parameter. For single-component analytes, all target analytes must be tested in the IPR study. Where there are multi-component parameters reported by the method (such as PCB's), the analytical department leader will determine if it is appropriate to test a subset of the total number of parameters reported by the method (e.g., perform an IPR study using PCBs 1016 and 1260 only). Once these replicate samples are created, they will be processed through the entire analysis method, and results generated for all analytes present in the samples. Analyte recoveries and precision data (standard deviations) will be calculated and compared to Paragon historical data or to criteria appearing in the promulgated method.

If the data generated by the laboratory staff meet the historical or promulgated criteria, the analytical staff who participated in the study are deemed to have passed the proficiency demonstration, and are judged to be capable of processing field samples. If an evaluation of the IPR data indicates that acceptance criteria has not been met by the staff, additional IPR studies must be performed until acceptable results have been generated for all target analytes.

With the completion of the IPR study, the analytical department manager will collect the data generated and transmit it to the Quality Assurance Manager. After review of this information by the QA Manager, the records will be placed in the training file of the employees who participated in the study.

As long as subsequent analytical effort by the analysts or technicians shows that they have maintained their proficiency, there will be no need to perform another IPR study for a given method. However, if performance data from routine analyses shows a degradation in the skills or capabilities of the laboratory staff performing a method, another IPR study may be mandated by the laboratory QA Manager.

14.3 TRAINING RECORDS

Training records for all analytical staff members will be maintained by the Paragon Quality Assurance Department. Training files may contain (but are not limited to) the following information:

1. Records of academic training pertinent to the employee's work assignment.
2. Summaries of any training seminars attended while employed at Paragon.
3. Any test results for examinations taken at Paragon.
4. Records of any Health & Safety instruction received while at Paragon.
5. Results of proficiency demonstrations as described in Section 14.2.2 of this LQAP.
6. If available, a current resume of the employee.

15. LABORATORY SAFETY

15.1 HEALTH AND SAFETY TRAINING

The goal of Health and Safety (H&S) training is to ensure that the laboratory personnel have adequate knowledge to safely perform their assigned duties. This training is presented by the laboratory's H&S Officer.

Health & Safety training is provided to each new employee as soon as possible after beginning work. The components of this course include, but are not limited to:

- Laboratory safety and hazard communication videotapes with computer based exams.
- An introduction to the Health and Safety Manual, including a discussion of required procedures and protocols.
- Information regarding the Hazard Communication Program, including the use of Material Safety Data Sheets (MSDS).
- Proper procedures for material handling, storage of chemicals, and use of personal protective clothing.
- Emergency procedures for chemical spills, contact, or exposure and other potential emergency situations.
- Procedures for disposal of wastes including excess or spent reagents, chemical standards, analysis samples or miscellaneous materials.
- Equipment handling and associated safety procedures and precautions.
- An explanation of the Medical Surveillance Program, which includes an annual physical examination for all employees engaged in laboratory activities.
- Chemical-Specific Hazard Communication training is provided as needed. This may be due to the addition of a new analytical procedure, modification of a method which requires the use of an additional chemical or anticipated receipt of samples potentially containing a known hazard.

15.2 RADIOCHEMISTRY HEALTH AND SAFETY

In general, all sample shipments received by Paragon from the field will be screened for surface exposure rates in the Sample Receipt area. Radiation survey meters are permanently located in this area for use by the Sample Custodian. If screening with the radiation survey equipment indicates that samples in the shipment have a higher external radiation exposure rate than expected from previous information received from the client, or from Paragon's previous knowledge of the site, the Sample Custodian will immediately contact the Health & Safety Officer (HSO) and request guidance for handling of the samples. The Safety Officer will use his/her judgment to determine if gross alpha/beta screening will be required for the incoming samples or not.

For many programs, field sampling teams and their engineering firms will generate radioactive material screening information in order to determine shipping classification for all sample shipments. If this information is provided to Paragon, it may not be necessary to screen the received samples for gross alpha/beta/gamma content by proportional counter analysis, depending on the HSO's confidence in the data. However, if no screening information is provided by the client for a sample shipment, and the surface radiation exposure survey at the laboratory indicates that unsuspected radioactive material may be present in the samples, Paragon will perform a gross alpha/beta/gamma screen by gas flow proportional counting.

15.3 LABORATORY SAFETY

Sample receiving areas and laboratories are equipped with suitable hoods, respirators, personal protective equipment and eye wear, gloves, barrier creams and or other measures to prevent or minimize staff contact with hazardous substances. Safety equipment such as eyewash stations, drench showers, spill adsorbents and neutralizers, fire extinguishers, first aid materials, and breathing oxygen are available.

As a matter of policy, Paragon shall not accept known initiator explosives, known dioxin-contaminated materials or unusual biohazard materials except in cases where the laboratory

has been designed to safely handle high hazard samples. Paragon is licensed by the Colorado Department of Health to process and handle radioactive materials according to the specifications of the Colorado Rules and Regulations Pertaining to Radiation Control. Paragon may accept nitroaromatics and nitroamines providing that the client makes provisions for disposal of samples with a positive explosive identification.

15.4 HEALTH AND SAFETY MANAGER

A laboratory staff member is designated as Health & Safety Manager by the Laboratory Manager. The Health & Safety Manager prepares and maintains safety-related SOPs, conducts safety and occupational health orientation, training and review sessions as required, and maintains up to date familiarity with safety and occupational health issues pertinent to the laboratory.

16. LABORATORY WASTE DISPOSAL

16.1 WASTE IDENTIFICATION

Paragon Analytics Inc. (PAI) utilizes process knowledge plus both chemical and radiochemical analyses to identify and characterize our waste streams. The analytical data is first used to classify the material as Non Hazardous/Non Radioactive, RCRA, TSCA, Low Level Radioactive , or Mixed Hazardous and Radioactive Wastes. The analytical data on the material is then reviewed to determine the appropriate waste profile to dispose of the material in.

16.2 WASTE STORAGE

Paragon is classified as a small quantity generator, and generates between 100 kg and 1000 kg of waste per month. Because of this rate of waste generation, waste materials created at the laboratory may accumulate on site for a maximum of nine months, depending upon the location of the Temporary Storage and Disposal Facility (TSDF).

The wastes generated by Paragon Analytics will initially be stored in Satellite Accumulation Areas (SAA) in the generating lab. The SAA containers are to be labeled with the waste identity, chemical and physical hazards, and spill clean up data. The SAA containers will be selected by the Health and Safety Department to provide compatibility, containment, easy handling, and to minimize the number of containers utilized.

The waste containers in the waste accumulation areas will be handled in the following manner. All containers used will meet United States Department of Transportation (USDOT) standards as per 49 CFR Parts 173, 178, & 179. The containers will be labeled with a preprinted hazardous waste label with accumulation start date, DOT Hazardous Material Warning Label , and orientation labels. The waste containers will be stored in such a manner to allow easy access to all containers for servicing or for emergency personnel in the event of an accident. Furthermore, all containers with liquids shall be

provided with a spill containment system that meets the requirements of Colorado 6 CCR 1007-3 Part 264.175.

16.3 WASTE DISPOSAL

RCRA Hazardous Wastes are disposed of by Paragon by using various hazardous waste disposal options. The most common disposal option employed at Paragon is destructive incineration at an approved and permitted hazardous waste incinerator. Most of the purely hazardous and non radioactive waste are disposed of via incineration at a Chemical Waste Management (CWM) facility. Mixed radioactive and hazardous wastes may be handled in a variety of methods depending upon composition and available disposal options. Disposal of Low Level Radioactive Waste (LLRW) that has not originated from U.S. Department of Energy sites will be disposed of at the Benton County Washington Low Level Radioactive Waste Disposal Site as mandated by the Rocky Mountain Low Level Radioactive Waste Board (RMLLRWB). The disposal of any purely Low Level Radioactive Waste from DOE sites will be accomplished at the Barnwell Low Level Radioactive Waste Disposal Site in South Carolina. This is due to the fact that the RMLLRWB prohibits disposal of any material originating from the Department of Energy at the Benton County Washington Low Level Radioactive Waste Disposal Site. The following table discusses the disposition of each waste stream on an individual basis.

Table 16-1 Paragon Analytics Analytical Process Waste Disposal Summary

Waste Name	Waste Type	Disposal Method	Waste Disposal Subcontractor	Disposal Frequency
Aqueous Lab Waste	RCRA	Destructive Incineration	CWM	As Needed
Halogenated Organic Waste	RCRA	Destructive Incineration	CWM	As Needed
NonHalogenated Organic Waste	RCRA	Destructive Incineration	CWM	As Needed
Contaminated Soils & Solids	RCRA	Destructive Incineration	CWM	As Needed
PCB Extracts	RCRA/TSCA	TSCA/RCRA Destructive Incineration	CWM	As Needed
Ion Exchange Resin	RCRA	Destructive Incineration	CWM	As Needed
Radioactive Solids	LLRW Only	Burial at Barnwell LLRW Disposal Site	EMC or Bionomics	As Needed
Radioactive Solids from DOE Sites	LLRW Only	Burial at Benton Co. LLRW Disposal Site	EMC or Bionomics	As Needed
Aqueous Radioactive Solutions	LLRW Only	Solidification & Burial at Barnwell LLRW Disposal Site	EMC or Bionomics	As Needed
Aqueous Radioactive Solutions from DOE Sites	LLRW Only	Solidification & Burial at Benton Co. LLRW Disposal Site	EMC or Bionomics	As Needed
Liquid Scint. Counting Wastes(Non Hazardous Cocktails) With Other Than H-3 & C-14	LLRW Only	Incineration	EMC & Permafix or DSSI	As Needed

Table 16-1 Paragon Analytics Analytical Process Waste Disposal Summary

Waste Name	Waste Type	Disposal Method	Waste Disposal Subcontractor	Disposal Frequency
H-3 & C-14 Liquid Scintillation Counting Wastes(Non Hazardous Cocktails)	LLRW Only	Incineration	EMC & Permafix	As Needed
Mixed Hazardous Radioactive Solids	RCRA/LLRW	Encapsulation To Pass TCLP & Burial at Barnwell LLRW Disposal Site	Permafix, EMC, or Bionomics	As Needed
Halogenated Organic Waste	RCRA/LLRW	Destructive Incineration	Permafix, DSSI, &/or Bionomics	As Needed
Non Halogenated Organic Waste	RCRA/LLRW	Destructive Incineration	Permafix, DSSI, &/or Bionomics	As Needed
Ion Exchange Resin	RCRA/LLRW	Dewater, Encapsulate, and Burial at Barnwell LLRW Disposal Site	Scientific Ecology Group, Bionomics	As Needed

17. PROCUREMENT CONTROL

17.1 RECEIPT/VERIFICATION OF STANDARDS

In this section, the term standard shall apply to any analyte solution of known concentration which is traceable to a certified reference material. This includes calibration standards, spiking solutions and laboratory control samples.

Upon receipt, all purchased standard reference materials (neat and stock solutions) are recorded into section-specific standard logbooks. Standard logbook entries include Paragon unique ID, name of the neat compound or solution, manufacturer, manufacturer's lot number, certified purity, expiration date and storage location. Subsequent preparations of stock, intermediate, and working solutions are also documented in the standard logbooks. These entries must include all discrete measurements made during preparation, parent materials, solvent(s) and a Paragon ID number.

All primary reference standard and standard solutions are purchased from reliable commercial sources. Standards traceable to NIST are preferred; however, ASTM or equivalent specifications are acceptable. Certification records of all standards received are retained.

Second source reference standards and standard solutions are purchased from a different supplier than the primary standard. If a second supplier is not available, the second source standard can be prepared from a different lot number of the same composition from the same supplier. For rare or unusual materials for which an alternate lot is not available (and only one vendor supplies the material), a second source verification solution can be prepared by an analyst who is different from the one who prepared the original stock solution. These two solutions can then be compared against one another.

Newly prepared standard solutions (surrogate, internal, calibration, spiking) are verified against another known standard prepared from another source prior to utilization. The verification data is maintained on file in the respective area.

The maximum deviation allowed between two sources of reference material for GC analysis methods is 15 %. The maximum deviation allowed between two sources of reference material for GC/MS analysis methods is 25 %. The maximum deviation allowed between two sources of reference material for inorganic analysis methods is 10%. The maximum deviation allowed between two sources of reference material for radiochemical analysis methods is 10 %.

17.2 RECEIPT/VERIFICATION OF SOLVENTS AND ACIDS

The verification procedure for organic solvents involves taking an initial volume of solvent and concentrating it to a reduced final volume. The initial volume used for this procedure and its final volume vary depending upon solvent. Not all solvents are screened due to initial solvent quality or application (eg., technical grade solvents used for rinsing glassware will not be screened for contaminants, acids used solely to soak inorganic glassware for cleaning will not be tested).

Acids are processed through the same procedures for which they will be used during routine analysis, only without a sample present in the process. In effect, a method blank will be generated and analyzed using the acids under test, and the results of this process evaluated.

After initial screening, the reagent quality is either confirmed or denied. If the solvent quality is found to be unacceptable, it shall not be used for production activities. A new solvent lot shall be ordered and the screening process repeated until an acceptable lot is identified. The records generated by the laboratory from this process will be kept on file in the departments that performed the analysis.

17.3 RECEIPT OF GENERAL REAGENTS

Most reagents that are used in the analysis of organic and inorganic methods are initially investigated for purity and possible contaminants. These reagents vary in usage, depending upon the analysis parameter, and sample integrity is greatly influenced by the interaction of components inherent in the reagents. Therefore, the need for overall monitoring of reagents is vital. Reagents stored in the laboratory areas must have a date recorded on their label. The date may be either the date the material was received from the vendor and unpacked from their shipping container, or the date at which the container was first opened. Table 17-1 indicates the general handling and storage procedures followed by Paragon for its analytical supplies and reagents.

Table 17-1 REAGENT STORAGE

Reagent	Method of Storage
Solvents	Stored in original containers in designated storage areas until required for use. As needed, laboratory personnel transfer solvents from storage areas to ready-use storage cabinets in the laboratory area. Note: opened bottles of methanol used for volatile organic analyses are stored in the GC-volatiles and GC/MS-volatiles analysis areas.
Inorganic Acids	Shipping containers are transferred upon receipt to laboratory. Individual bottles are removed from shipping containers and placed in ready-use storage cabinets which are designed for storage of corrosive acids.
Organic Acids	Upon receipt, acids are distributed directly to the analytical department. These acids are stored in designated areas for ready use or in cabinets designed for storage of corrosive acids.
Caustics	Upon receipt, caustics are distributed directly to the analytical department. Caustic containers are stored in the department's cabinets designated for caustic reagent storage only.
Other Reagents	Stored in designated cabinets or refrigerators in each department. Standards that require storage at 4 °C or below freezing are stored in each department's refrigerators or freezers (respectively) designated for standards only.