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August 27, 2010

Michelle Mullin
Project Manager
USEPA Region 5
77 West Jackson Boulevard
Chicago, IL 60604-3590

Subject: **Quality Assurance Project Plan (QAPP)**
RCRA 3008(h) Consent Order (RCRA-05-2010-0012)
Former Tecumseh Products Company Site, Tecumseh, Michigan

Dear Ms. Mullin:

RMT, Inc. (RMT) on behalf of Tecumseh Products Company (TPC) is submitting two CDs containing the Quality Assurance Project Plan (QAPP) in support of the investigation work currently being performed by TPC according to RCRA 3008(h) Consent Order (RCRA-05-2010-0012).

Also attached per your request is a CD containing a pdf version of the Current Conditions Report previously submitted to USEPA.

If you have any questions regarding the attached documents, please contact me at graham.crockford@rmtinc.com, or (734) 971-7080, ext. 7122.

Sincerely,

RMT, Inc.

Graham Crockford
Project Manager

Attachments:
Quality Assurance Project Plan (QAPP) – 2 CD PDF versions
Current Conditions Report (CCR) – 1 CD PDF version

cc: Jason Smith, Tecumseh Products Company
Douglas McClure, Conlin, McKenney & Philbrick, P.C.
Central Files

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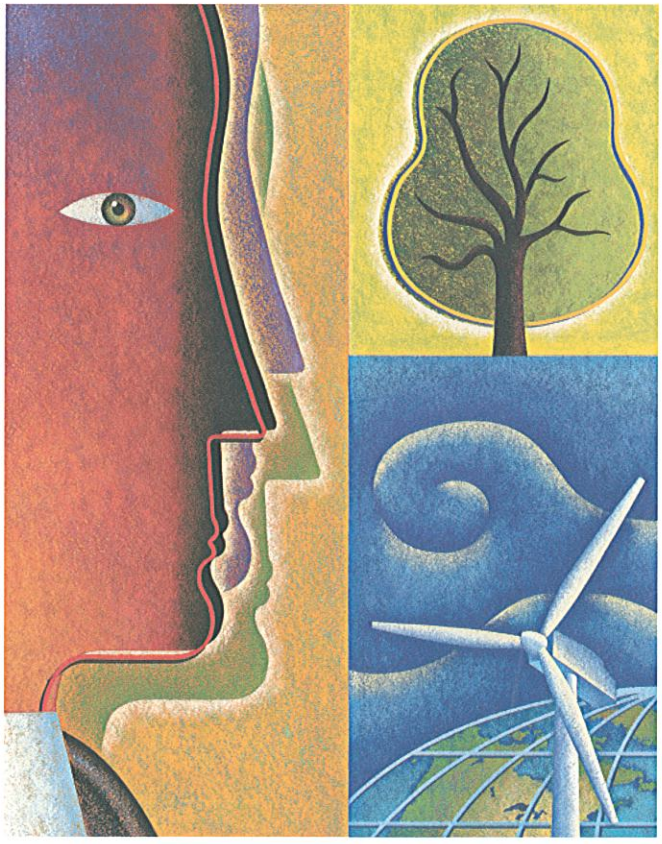
Quality Assurance Project Plan for RCRA Consent Order RCRA-05-2010-0012

Former Tecumseh Products Company
Site MID 005-049-440
Tecumseh, Michigan

August 2010



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Quality Assurance Project Plan for RCRA Consent Order RCRA-05-2010-0012

Former Tecumseh Products Company
Site MID 005-049-440

Tecumseh, Michigan

August 2010

Prepared For
Tecumseh Products Company

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- Michelle Mullin – USEPA Region 5 Project Manager
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Section 1
Project Description

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific Quality Assurance/Quality Control (QA/QC) procedures associated with the Remedial Investigation (RI) and Presumptive Corrective Measures (CM) for the former Tecumseh Products Company (TPC) Site located in Tecumseh, Michigan. This QAPP was prepared by RMT, Inc. (RMT) on behalf of TPC in response to the Resource Conservation and Recovery Act (RCRA) Consent Order (RCRA-05-2010-0012) for MID-005-049-440. Specific protocols for investigation methods, sampling, sample handling and storage, Chain-of-Custody, and laboratory and field analyses will be described. All QA/QC procedures will be structured in accordance with applicable technical standards, The United States Environmental Protection Agency's (USEPA's) requirements, regulations, guidance, and technical standards. This QAPP has been prepared in accordance with the USEPA Region 5 QAPP policy as presented in *U.S. EPA RCRA QAPP Instructions*, and other relevant guidance documents, including *The Use of Field Methods to Support RFI Streamlining*, U.S. EPA, Region 5 Memorandum, June 20, 1997**.**

1.1 Introduction

The purpose of the QAPP is to provide QA procedures for sampling, analysis, and data evaluation for the scope of work detailed in the various workplans. This QAPP includes a description of the procedures that will ensure acquisition of reliable and high quality data, and that will meet the data quality objectives of the project. This QAPP describes procedures that pertain specifically to soil sampling, monitoring well installation, groundwater sampling, surface water sampling, active soil gas monitoring point installation, soil gas sampling, indoor air sampling, passive soil gas monitoring point installation, and passive soil gas sampling. This QAPP is designed to generally meet the requirements of the RCRA program.

This QAPP is intended to supplement various workplans developed to meet the objectives of the RI and Presumptive CM. These workplans will detail the purpose of the work to be performed and the associated sampling and analysis requirements. This QAPP, together with the various workplans that may be developed throughout the project, comprise the Sampling and Analysis Plan for this site. These workplans may be informal in nature, and in accordance with the Consent Order, USEPA approval is not needed prior to implementation of the proposed work.

1.1.1 Overall Project Objectives and Decision Statements

The primary purpose of the RI is to gather sufficient information to quantify and mitigate current risk to human health and the environment. Overall objectives of the data collection will be as follows:

- Verify and further define the nature and extent of constituents of concern (COCs) in previously identified on-site and off-site areas (soil, groundwater, surface water, soil gas, and indoor air matrices). Data quality must be sufficient to allow comparison with established screening levels. The target screening levels for all project parameters are summarized in Tables 1 through 4.
- Determine the nature and extent of COCs in the previously unidentified areas, if any. Laboratory data will be compared to target screening levels.
- Determine the nature and characteristics of the hydrogeologic units at the subject property.
- Collect sufficient data to design, evaluate and/or optimize, as appropriate, any Presumptive CM.
- Collect sufficient data for the affected media to prepare a human health baseline risk assessment, to prepare a preliminary ecological risk assessment, and/or to prepare a feasibility study for CM, as needed.

The Decision Statements for the RI and Presumptive CM are as follows:

- What is the nature and extent of select environmental constituents in onsite or offsite soil, surface water, groundwater, soil gas, and indoor air media that present unacceptable risks, which would therefore warrant remedial action?
- If present, how can unacceptable current exposures to affected media and continued releases to the environment be appropriately mitigated?
- Are Presumptive CM, if any, effective in mitigating current unacceptable exposures and/or continued release of COCs into the environment?

Associated specific objectives for field and laboratory data collection are described in Section 3 of this QAPP.

1.1.2 Project Status / Phase

TPC and RMT will utilize an integrated and phased approach to the RI and Presumptive CMs. During the RI, data collection will be conducted in phases. Decisions regarding the necessity for additional phases of investigation will be made by comparing RI results to target screening levels. The field investigations may include the following activities:

- Groundwater sampling at existing wells;
- Installation of new monitoring wells;
- Water supply well sampling;
- Storm sewer sampling;
- *In situ* permeability testing of aquifer materials;
- Surface water sampling;
- Active soil gas sampling at existing sample locations;
- Installation of new soil gas sample points;
- Passive soil gas sampling;
- Indoor air sampling;
- On-site surface soil and subsurface soil sampling;
- Pilot testing of various potential remedial options; and
- Required monitoring of treatment systems, if any.

Samples will be analyzed for COCs identified in the workplan(s). At a minimum RI samples will be analyzed for the project specific list of chlorinated volatile organic compounds (CVOCs):

- 1,1-Dichlorethane (1,1-DCA);
- 1,2-Dichlorethane (1,2-DCA);
- 1,1-Dichlorethene (1,1-DCE);
- cis-1,2-Dichlorethene (cis-DCE);
- trans-1,2-Dichlorethene (trans-DCE);
- Tetrachloethene (PCE);
- 1,1,1-Trichloroethane (TCA);
- Trichlorethene (TCE); and
- Vinyl chloride.

A limited number of samples may also be analyzed for physical and/or chemical parameters.

Data and recommendations from the various phases of the RI will be prepared and submitted to USEPA for review in a timely manner, typically as part of a quarterly progress report. The rationale and scope of RI and Presumptive CM activities will be

discussed with the USEPA. However, in accordance with the Consent Order, further investigation and/or presumptive CM may be implemented to mitigate risk to human health and the environment, as appropriate, without prior USEPA approval. As required by the Consent Order the following documents will be submitted to USEPA for review and approval:

- Environmental Indicators (EI) Report – Human Exposures – due 9/29/2011
- EI Report – Groundwater Stabilized – due 9/29/2012
- RI Report – due 9/29/2012
- Final CM Proposal – due 3/29/2013

1.1.3 QAPP Preparation Guidelines

This QAPP has been prepared in general accordance with the *U.S. EPA Region 5 RCRA QAPP Policy*.

1.2 Site Description

This section presents history and background information on the former TPC site in Tecumseh, Michigan and the environmental issues that precipitated the Consent Order.

1.2.1 Location

The former TPC manufacturing site in Tecumseh, Michigan is an inactive manufacturing facility, which was primarily used for the production and reconditioning of compressors and condensing units for refrigeration and air conditioning units. The former TPC manufacturing site is located in Section 34, Township 5 South, Range 4 East, Tecumseh, Lenawee County, Michigan (Figure 1). The site is also known as the property located at 100 East Patterson Street. The study area includes the off-site areas immediately surrounding the site and extends in the direction of groundwater flow (east/northeast) to the River Raisin.

1.2.2 Facility / Site Size and Borders

The approximately 53-acre site is bordered on the north by East Patterson Street, on the west by a railroad right-of way that runs adjacent to Evans Street, on the east by Maumee Street, and on the south by a series of industrial/commercial properties including a fire station, a garage, a bank, and a furniture warehouse. The former TPC property includes an expanse of interconnected buildings/building additions that occupy approximately 750,000 square feet (Figure 2).

1.2.3 Natural and Manmade Features

The subject property is occupied primarily by a large manufacturing building that is approximately 750,000 square feet; approximately 80,000 square feet of which is used for office space and for research and development activities. Prior to June 2008, the main building on the property was primarily used for manufacturing and warehouse space. The majority of this space is currently unoccupied; however, a portion of the building along the northwest side of the building, Area H and Area J on Figure 3, remains in use by TPC. The space is used for administrative offices and to house research and development activities. A description of current and historical uses of the site is provided in greater detail in Section 1.3 of this QAPP. The remainder of the facility was used for manufacturing and warehousing, and is currently unoccupied. Four additional buildings reside on the subject property; Building R was used for wastewater treatment, Building Q was used for chemical storage, Building L was used for vehicle maintenance, and Building X-1 was used for recycling. A gravel and asphalt parking lot occupies the area east of the main building. A gravel driveway extends along the south and east sides of the main building. The developed area (Parcel 325-0241-00) is bordered by a fence. South of the main building is a grassy, undeveloped area (Parcel 325-0250-00).

1.2.4 Topography

The site is roughly rectangular and consists of approximately 53 acres. The site topography is flat with a gentle slope (approximately 1-percent slope) to the east. East of the site the surface elevation drops steeply into a wetland area/flood plain adjacent to the River Raisin. The River Raisin is located about one-half of a mile east of the site.

1.2.5 Local Geology and Hydrogeology

The site geology generally consists of a surficial clay interval ranging from 3 to 7 feet thick, underlain by unconsolidated fine to coarse sand and gravel. A second clay layer is continuous beneath the site. Local water well logs indicate that bedrock is 150 to 200 feet deep at the site.

Groundwater flow at the former TPC site and surrounding study area is generally east toward the River Raisin. A mean horizontal hydraulic gradient of 0.001 was measured across the former TPC property. East of the site, in proximity to the change in surface elevation, the horizontal hydraulic gradient increases. East/southeast of the site, the presence of discontinuous gravel and/or sand with gravel units that are more conductive than the bulk of the sand aquifer facilitates the decrease in static water elevation. Vertical groundwater movement is impeded by the continuous clay layer underlying the gravel deposit. Site geology and hydrogeology is described in greater detail in the

Current Conditions Report (CCR) submitted to USEPA on September 21, 2009 and the July 2010 Technical Memorandum titled "Summary of Groundwater Investigation Activities – March 2010 through June 2010: Former Tecumseh Products Company Site, Tecumseh, Michigan," which was submitted to USEPA as part of the Second Quarter 2010 Progress Report.

1.2.6 Land Use

Generally, land use in the vicinity of the subject property consists of light industry, commercial operations, and residential properties. The site is zoned for industrial use and the surrounding area is zoned for mixed residential-commercial use. Near the River Raisin there is an undeveloped wooded wetland/floodplain area with areas of standing water. The City of Tecumseh wastewater treatment plant is located northeast of the site, at the end of Cummins Street.

The City of Tecumseh owns and operates a well field FE9 [redacted] FE9. This well field, and the associated wellhead protection area, is hydraulically upgradient of the site. Although, city water is available throughout the developed portion of the study area, five private supply wells are present within the area.

1.2.7 Ecological Communities and Habitats

Portions of the former TPC site and study area have terrestrial communities comprised of wooded and grassland flora and fauna. Downgradient of the study area, the River Raisin and adjacent wetland/floodplain has aquatic communities comprised of lentic, lotic, wetland and temporary ponds.

1.3 Site / Facility History

1.3.1 General History

The TPC site consisted of farmland (undeveloped woodlands/farmland) until it was first developed for industrial use in the late 1800s and early 1900s. Prior to TPC's acquisition of the site in 1934, portions of the property had been occupied by an iron foundry, a fence company, and other manufacturing operations. Since 1934, the site has been occupied by various divisions of TPC. Historical documents indicate that prior to June 2008, the uses of the site have not changed significantly since 1934, other than changes in some product lines, several episodes of facility expansion, and an increasing level of development.

FE9 = FOIA EXEMPTION 9, GEOLOGIC INFORMATION

The TPC site is occupied by a series of interconnected buildings/building additions that occupy approximately 750,000 square feet (main building). There are other buildings on site, but they are significantly smaller in size, and were typically not utilized for manufacturing operations. Letter designations, *i.e.*, Area K, Building Q, etc., for each building/building addition are shown on Figure 3.

The oldest portions of the main building, referred to as Area B and Area H (Figure 3), are located in the northern portion of the site were constructed around 1908; subsequent building expansions and additions have connected these two areas and grown the main building to the south and east. Areas H, J, and Z in the northwestern portion of the building have historically housed the TPC offices and corporate headquarters and TPC research and development (Engineering Department). The rest of the main building was used primarily for the manufacture and storage of TPC products. The first products manufactured by TPC included automotive parts, refrigeration systems, small tools, and toys. By June 2008, when manufacturing operation ceased at the site, TPC operations focused on the production and reconditioning of compressors and condensing units for refrigeration and air conditioning units. Historically these operations included the use of TCE and 1,1,1-TCA for parts degreasing operations.

In December 2009 the property was transferred to Tecumseh Bakery, LLC and its holding company Consolidated Biscuit Company (CBC). The purchase agreement includes a lease agreement which allows TPC to continue to occupy the engineering area. Approximately 30 TPC employees currently occupy the leased office/engineering portions of the building (Areas H and J). The balance of the site, including Area K which is included in lease as a TPC storage area, remains unoccupied.

In April 2010, when CBC was sold to a Chicago investment group, Tecumseh Bakery, LLC became the sole owner of the site, and plans to convert the facility into a bakery were abandoned. At present, TPC is unaware of any plans to occupy or sell the facility.

See Section 3.0 of the CCR for a more complete description of historic manufacturing operations including waste management, environmental permits, and chemical storage.

1.3.2 Past Data Collection Activities

In 2008, a Phase I Environmental Site Assessment (ESA) was conducted by Atwell-Hicks, LLC as part of the sale of the facility and associated property to CBC. The Phase I ESA Report recommended that a Phase II Subsurface Investigation be conducted to address the identified recognized environmental conditions (RECs). A Phase II ESA was performed by ATC Environmental Consultants (ATC) on behalf of CBC between

December 2008 and January 2009. A copy of the Draft Limited Phase II ESA Report was provided to TPC in February 2009. The Phase II ESA Report was finalized on September 4, 2009.

TPC retained RMT to investigate soil and groundwater conditions at the site and surrounding area. Between February 2009 and September 2009, TPC performed on-site and off-site investigations to define the extent of the CVOCs in soil and groundwater. In September 2009, RMT submitted a CCR to the USEPA and the Michigan Department of Environmental Quality (MDEQ, now the Michigan Department of Natural Resources and Environment [MDNRE]). The CCR described and summarized the physical setting of the site, the historical operations, sampling data, potentially complete exposure pathways, and voluntary remedial activities undertaken by TPC.

Between November and December 2009, 12 additional monitoring wells were installed and a complete round of groundwater samples was collected in an attempt to address the remaining data gaps related to the off-site migration of VOCs. The findings of these investigation activities were submitted to USEPA on February 12, 2010 in a Technical Memorandum titled "Status Update – Characterization of Volatile Organic Compounds in Groundwater, Former Tecumseh Products Company Site, Tecumseh, Michigan."

In March 2010, RMT initiated a supplemental investigation to address the potential data gaps related to the off-site migration of VOCs in groundwater. Eleven additional monitoring wells were installed and a complete round of groundwater samples was collected between March 2010 and April 2010. Regular quarterly monitoring was conducted again in May 2010 and one additional well was installed and sampled in June 2010. The findings of these investigation activities are described in more detail in a Technical Memorandum titled "Summary of Groundwater Investigation Activities – March 2010 through June 2010: Former Tecumseh Products Company Site, Tecumseh, Michigan," which was submitted to USEPA with the Second Quarter 2010 Progress Report on July 15, 2010.

Concurrent with the off-site groundwater investigation, RMT conducted an on-site evaluation of the volatilization to indoor air migration pathway. In October 2009, RMT prepared and implemented a Workplan to investigate sub-slab soil gas at 18 locations throughout the manufacturing building. In January 2010, RMT collected a second round of sub-slab soil gas samples and indoor air samples at 5 locations in Building Area P. The ventilation system in Building Area P was not operational during sample collection. In February 2010, RMT retested indoor air in Building P with the building ventilated and mixed. In March 2010, RMT sampled indoor air (IA-09 through IA-17, and IA-19)

adjacent to the ten sub-slab monitoring points in the secondary use area and re-sampled five of the sub-slab monitoring points. The results of the P-Building investigation are summarized in a Technical Memorandum titled "Investigation and Mitigation Strategy for Indoor Air - P-Building: Former Tecumseh Products Company (TPC) Manufacturing Facility," and the results of the secondary use area investigation are summarized in a Technical Memorandum titled "Investigation and Mitigation Strategy for Indoor Air – Secondary Use Area Former Tecumseh Products Company (TPC) Manufacturing Facility Tecumseh, Michigan."

Between March 2010 and May 2010, RMT conducted a preliminary evaluation of off-site soil gas. In March 2010, 14 soil gas sample points were installed around the site perimeter and adjacent to residential properties within the study area. A complete round of samples was collected between March 31 and April 2, 2010. A re-sample event was conducted in May 2010. These investigation activities are summarized in a Technical Memorandum titled "Summary of Off-Site Soil Gas Investigation Activities – March through April 2010: Former Tecumseh Products Company Site, Tecumseh, Michigan," which was submitted to USEPA with the Second Quarter 2010 Progress Report on July 15, 2010.

In July 2010, RMT conducted a passive soil gas survey in the approximately 250,000 square foot area described in the CCR as the northern source area. The goal of this investigation was to locate, discrete source areas, if any, for further investigation and/or treatment. The results of this investigation are qualitative and will be used to assist with the development of future RI workplan(s) and presumptive CM.

1.3.3 Current Status

TPC is focused on complying with the USEPA Region V Consent Order (RCRA-05-2010-0012) for the site (MID-005-049-440) which is dated February 29, 2010. The future RI and Presumptive CM will focus on the project objectives outlined in Subsection 1.1.1 of this QAPP. The current status of RI and Presumptive CM activities are described in the following documents:

- Current Conditions Report dated September 2009;
- Technical Memorandum titled "Investigation and Mitigation Strategy for Indoor Air - P-Building: Former Tecumseh Products Company (TPC) Manufacturing Facility," dated March 9, 2010;
- Technical Memorandum titled "Investigation and Mitigation Strategy for Indoor Air – Secondary Use Area Former Tecumseh Products Company (TPC) Manufacturing Facility Tecumseh, Michigan," dated June 17, 2010;

- Technical Memorandum titled "Summary of Off-Site Soil Gas Investigation Activities – March through April 2010: Former Tecumseh Products Company Site, Tecumseh, Michigan," which was submitted to USEPA with the Second Quarter 2010 Progress Report on July 15, 2010; and
- Technical Memorandum titled "Summary of Groundwater Investigation Activities – March 2010 through June 2010: Former Tecumseh Products Company Site, Tecumseh, Michigan," which was submitted to USEPA with the Second Quarter 2010 Progress Report on July 15, 2010.

1.4 Project Objectives and Intended Data Usages

For this project, it will be necessary to gather sufficient information to define on-site source areas, to evaluate the nature and extent of releases from the source areas, and also to determine whether unreasonable risks to human or the environment are associated with the areas. This could include evaluation of the impact of releases on human health and the environment both within and beyond the facility property boundary, if applicable. The data collection activity will specifically address the following concerns:

- To define the horizontal and vertical area over which COCs exceed target screening levels;
- To determine soil physical parameters and their effect on contamination migration;
- To determine whether potentially unacceptable risk to human health or the environment is present;
- If potential unacceptable risk is present, to perform a human health risk assessment and/or design and implement presumptive CM to eliminate the potential risk;
- To evaluate the effectiveness of various remediation strategies or presumptive CM to control or abate sources, to control contaminant migration, and to abate the risk represented to human health or the environment by site constituents.

Future workplans will define the specific project objectives and intended data usages for the proposed work, including a field sampling plan.

1.4.1 Project Target Parameters List

Project target parameters were selected on the basis of historical use in TPC manufacturing operations and the presence of constituents in previously collected samples. Soil and groundwater samples may be analyzed for volatile organic compounds (VOCs). In addition, a limited number of soil samples may also be analyzed for cation exchange capacity (CEC), Atterburg limits, percent moisture, grain size distribution, and/or total organic carbon (TOC) to determine soil physical parameters and their effect on contamination migration. Similarly a limited number of groundwater

samples may be analyzed for monitoring natural attenuation (MNA) parameters: chloride, ferrous iron, nitrogen as nitrate, sulfate, TOC, etc.

Soil gas and indoor air samples may be analyzed for a project specific list of CVOCs. In addition, active soil gas samples will be analyzed for a tracer gas, such as isopropanol or helium.

Previous investigation, specifically the Phase II ESA performed by ATC indicates that semi volatile organic compounds (SVOCs), polynuclear aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCBs) and metals are not COCs. Since pesticides and herbicides were not used in any facility operations, TPC does not anticipate sampling for these parameters. Tables 1 through 4 show the analytical method and reporting limit for each target analyte. Table 5 summarizes the laboratory methods and sample collection and preservation requirements for each parameter.

The exact analytical suite for each sample will vary by area and the rationale for selecting the analytical parameters for each area will be provided in the various workplans. Select samples may be analyzed for an expanded suite of either chemical or physical parameters as warranted by the specifics of the investigation.

1.4.2 Field Parameters

Soil samples collected in and around potential source areas will be screened with a Photo Ionization Detector (PID) equipped with the appropriate 11.7 volt lamp. The principal use of the PID will be as a Health and Safety screening device to monitor VOC levels in the breathing zone of field staff. The PID may be used to semi-quantitatively screen soil samples for the presence of VOCs. The procedure to screen samples is included as Appendix A. Groundwater field parameters include turbidity, specific conductance, temperature and pH in order to ensure stabilization is achieved and to ensure a representative groundwater sample is collected and analyzed. Other parameters such as reduction-oxidation potential (redox) and dissolved oxygen may be measured in the field.

1.4.3 Laboratory Analytical Methods

The laboratory analytical methods selected to quantify the project target parameters are presented in Table 5. The analytical methods were selected to achieve reporting limits that will enable comparison with appropriate screening levels.

1.4.4 Use of Historic Data

The TPC site has been the subject of extensive environmental investigations and actions since 2008. These previous investigations generated data on subsurface soil and groundwater quality that will be used to help design the future workplans, to select appropriate sampling locations, and to define the target parameter list as described in Subsection 1.4.1 of this QAPP. With the exception of the Phase II ESA performed by ATC, RI activities have been conducted by RMT. The sampling techniques, field methods, analytical methods used by RMT during previous investigation activities are generally consistent with the procedures and methods described in this QAPP.

The usability of historic data in the Corrective Action will be evaluated in accordance with the Region 5 Memorandum Concerning the Use of Historic Data, and will be reviewed for the components listed below before it will be applied:

- Identification of specific dates, locations, and depths of samples in all media;
- Sampling techniques utilized, including well construction information;
- Sample collection, preservation, and transportation practices;
- Identification of all constituents for which the samples were analyzed;
- QC samples and results;
- Laboratory acceptability (including any audits, certifications, *etc.*);
- Analytical method documentation;
- Original analytical laboratory-submitted data package;
- Data package QC report;
- Data reporting (including reporting limits, treatment of non-detects, *etc.*);

Phase II ESA results that are used will be reported as such and used in conjunction with recent data. The Phase II ESA is described in more detail in the ESA Report.

1.4.5 Aquifer and Fate and Transport Modeling

Information collected from aquifer testing, including slug tests, pilot program tests, and large-scale pump tests may be collected and applied in developing a conceptual groundwater flow model. The specific methods used in these field programs will be detailed in the various workplans.

1.5 Sampling Locations

Historical and current sampling locations are shown on Figure 2 and Figure 3. Future investigation activities may include new sampling locations. If new sampling locations are proposed, the applicable workplan will include a description of the proposed sample locations and depths. However, depending on the nature of encountered field conditions such as underground or overhead utilities, sampling locations may be changed. The person who shall be responsible for making such decisions will be the RMT Project Manager and Technical Coordinator whose responsibilities are described in Subsection 2.2.2 of this QAPP.

1.5.1 Rationale of Selected Sampling Locations

All soil, surface water, groundwater, soil gas, and air sampling locations will be selected on the basis of historical site information, Phase I Environmental Site Assessments, site walkovers, and additional data collected from site records and personnel. Rationale for existing sampling locations is provided in previous workplans and/or the reports which summarize the RI activities. Since no sediments are present on the property, RMT does not anticipate sampling this matrix. In general new sample locations and sampling intervals will be designed to provide data adequate to characterize the magnitude and extent of COCs in the affected media necessary to support a risk assessment and evaluate potential remedial options. The full, detailed rationale for selecting the sampling locations will be described in the various workplans, however, RMT may make modifications in the field based on PID results, visual/olfactory senses, accessibility, and drilling limitations.

1.6 Project Schedule

RI and Presumptive CM activities will be completed in a timely manner so that, in accordance with the Consent Order, the following documents can be submitted to USEPA for review and approval on or before the required submittal date:

- Environmental Indicators (EI) Report – Human Exposures – due 9/29/2011;
- EI Report – Groundwater Stabilized – due 9/29/2012;
- RI Report – due 9/29/2012; and
- Final CM Proposal – due 3/29/2013.

1.7 Data Quality Objectives

Sample data will be collected to meet definitive data quality objectives (DQOs). Trimatrix Laboratories (Trimatrix) of Grand Rapids, Michigan will perform the laboratory analyses for soil, surface water, and groundwater samples. Pace Analytical Laboratories (Pace) of

Minneapolis, MN will analyze active soil gas and indoor air samples. Beacon Environmental Services (Beacon) will analyze passive soil gas samples. If the workplan objectives require rapid turnaround, soil and groundwater samples may be analyzed by a mobile laboratory operated by Environmental Chemistry Consulting Services, Inc., (ECCS). Appendix B contains the Quality Assurance Manual for each of these laboratories.

The analyses of samples collected during RI and Presumptive CM activities will include QA/QC procedures and documentation as described in the applicable Laboratory Quality Assurance Manual. This information will be used to evaluate the presence of COCs in the sampled media. The DQO process is a series of planning steps based on scientific methods that are designed to ensure that the type, quality, and quantity of environmental data used in decision-making are appropriate for the intended application.

DQO's are qualitative and quantitative statements derived from the outputs of each step of the DQO process that clarify the investigative objectives; define the type of data to be collected; and specify the conditions from which to collect the data. The DQO's are then used to develop a scientific and resource-effective data collection design.

The DQO process allows decision-makers to define their data requirements and specify how different types of data will be used in the investigative process before any data are collected.

- **Level 1 and 2 DQO's - Screening and Field Analysis**: These data are obtained using portable instruments that can provide real-time qualitative (screening) and semi-quantitative (field analysis) data to assist in the optimization of sample quality, guide sample acquisition efforts, and in some instances, provide health and safety support. These data are generated by less precise analytical methods and with less rigorous sample preparation. Sample preparation steps may be restricted to simple procedures, such as dilution, instead of elaborate preparation. Field screening may provide analyte identification and quantification, although the quantification may be relatively imprecise. A portion of field and screening data may be confirmed using analytical methods and QA/QC procedures and criteria associated with definitive data. Field and screening data without associated confirmation data are not considered to be data of known quality. Most screening and field instruments will require calibration as described in this QAPP and will be used for:
  - Groundwater sampling at existing wells;
  - field-measured parameters (pH, temperature, specific conductance, and turbidity),
  - depth to groundwater measurements,
  - field-measured soil vapors and ambient air quality (PID), and
  - health and safety monitoring.

- **Level 3 DQO's – Engineering**: These data, if collected, provide an intermediate level of data quality for site characterization. These may include mobile laboratory–generated data, or quick-turnaround laboratory data without full QC documentation.
- **Level 4 DQO's - Confirmative Data**: These data are generated following prescribed analytical methods, by the certified laboratory. Data are analyte-specific, with confirmation of the analyte's identity and concentration. Methods produce tangible raw data (*e.g.*, instrument printouts) in the form of paper printouts or computer-generated electronic files. Data may be generated at the site or at an off-site location as long as QA/QC requirements are satisfied. For the data to be definitive, either analytical or total measurement error or precision of the analytical method must be determined. This provides the highest level of data quality. These analyses require SW-846 analytical and data validation procedures in accordance with USEPA-recognized protocol.

Section 2
Project Organization and Responsibility

TPC has retained RMT to plan and perform field investigations, and to evaluate the data collected from environmental sampling programs at the site. The RMT project team is composed of engineers, hydrogeologists, scientists, laboratory personnel, and support staff. This multi-disciplinary project team provides the mechanism for completing the project in a systematic, comprehensive, and timely manner.

The RMT project manager will provide overall direction to the project team to ensure that the work is carried out on time, within budget, and in a technically appropriate manner.

Senior members of RMT's staff will act as technical consultants, who will assist the project manager in the overall scoping, technical quality, and administration of the work. The Quality Assurance (QA) Manager or designee, independent of staff responsible for preparing project outputs, will review all outputs before distribution. The Project Manager and QA Manager will work together to ensure that data quality objectives are met.

The contract laboratory will perform chemical and physical analyses for the project. Senior laboratory staff will also be available to assist in data validation and data management.

2.1 Project Organization Chart

Figure 4 illustrates the project organization and interaction among the various parties.

2.2 Management Responsibilities

The project will be performed under the overall supervision of RMT. The RMT office and field personnel will share quality management responsibilities for the field activities. In general, typical responsibilities for the various personnel include the following:

2.2.1 Project Manager

The project manager is responsible for meeting the overall project objectives and for identifying major project issues; acts as liaison between RMT, TPC, and the USEPA; provides QC review and conformance, project plans, and ongoing review for logic and reasonableness of interim results; coordinates the activities of the QA reviewer and other quality control (QC) data reviewers to provide objective oversight; and approves and signs reports.

2.2.2 Technical Coordinator

The technical coordinator provides technical supervision, guidance, and review for daily on-site operations; is responsible for conformance with the project plans, schedule, and procedures on-site; performs or designates daily review and signing of notes and logs; and performs ongoing coordination with the RMT project manager on the work progress, interim results, and problem identification and resolution.

2.3 Quality Assurance Responsibilities

2.3.1 Quality Assurance Manager

The quality assurance manager provides overall independent QA oversight for the project and follows the project closely to audit QA/QC procedures and to ensure conformance with QA objectives.

2.3.2 QC data reviewers

QC reviewers provide discipline-specific reviews of portions of the project workplans, progress, problems, and outputs and is called upon systematically for reviews or called upon by the project manager for specific reviews. QC reviewers include RMT staff responsible for providing data validation, data assessment, and internal performance and system audits.

2.4 Laboratory Responsibilities

2.4.1 Laboratory Project Managers

The laboratory project managers will report directly to the RMT technical coordinator and be responsible for ensuring that all laboratory services are available when needed and for overseeing production and final review of all analytical reports. Specifically, the laboratory project managers will assume responsibility for the following:

- Coordinating laboratory analyses,
- Supervising in-house Chain-of-Custody,
- Scheduling sample analyses,
- Overseeing data review,
- Overseeing preparation of analytical reports, and
- Approving final analytical reports prior to submission to RMT.

2.4.2 Laboratory Quality Assurance Officers

The laboratory QA officers have the overall responsibility for data after it leaves the laboratory. The laboratory QA officers will be independent of the laboratory but will communicate data issues through the appropriate laboratory project manager. In addition, the laboratory QA officer will:

- Oversee laboratory QA,
- Oversee QA/QC documentation,
- Determine whether to implement laboratory corrective actions, if required,
- Define appropriate laboratory QA procedures,
- Prepare laboratory SOPs, and
- Sign the title page of the QAPP.

Final responsibility for project quality rests with the RMT Project Manager. The appropriate laboratory Project Manager and QA Officer, prior to release of all data to TPC, will provide independent QA.

2.5 Field Responsibilities

2.5.1 RMT Field Coordinator

The RMT Field Coordinator will guide the field personnel in achieving a thorough understanding of the QAPP and their respective roles relative to one another within the established project framework. The Field Coordinator will also act as the site Health and Safety Representative (HSR).

The Field Coordinator is also responsible for the day-to-day activities of contractor field personnel. In this capacity, the Field Coordinator is responsible for the quality assurance of daily project activities and the maintenance of the QAPP. Further responsibilities include the review of field notebooks, driller's logs, and other field-related documentation.

2.5.2 RMT Field Personnel

These environmental staff will be responsible for measuring and recording field parameters; installing monitoring points, collecting, labeling, and transporting samples; and conducting in-field measurements, in accordance with the workplan and QAPP. RMT field personnel will be drawn from RMT's pool of corporate resources and will report to the Field Coordinator.

2.6 Special Training Requirements and Certification

All RMT personnel will be fully trained and current in OSHA Hazwoper 40-hour training and first aid training. Only fully trained and/or certified personnel will use specialized field equipment or construction equipment, such as a backhoe.

2.7 Distribution List

The following people will receive a copy of this QAPP.

- Graham Crockford - RMT Project Manager
- Michelle Mullin – USEPA Region 5 Project Manager
- Jason Smith – TPC Corporate Environmental Director
- Lynn Dennison - TPC Counsel
- Douglas McClure – Conlin, McKenney & Philbrick, PC (TPC outside counsel)
- Stacy Metz - RMT Technical Coordinator
- Thomas Stolzenburg – RMT QA Manager
- David Kreeger - RMT Field Coordinator
- Terry Hertz/Mark Bailey – RMT Data Validation
- Alan Debus - USEPA Region 5 Regional QA Manager
- Jennifer Rice – Trimatrix Laboratories Project Manager
- Melanie Ollila – Pace Analytical Laboratories QA Officer
- Harry O'Neill – Beacon Environmental Services Project Manager
- Michael Linskens– ECCS QA Officer

In the event of significant modifications to any portion of the QAPP, the RMT project manager will issue replacement pages and distribute them to each of the QAPP recipients. The replacement pages will contain the date of the modification in the header at the top of the page to enable readers to easily identify them from the original pages. A table will be included with this distribution that will contain a chronology of the updates and modifications to the QAPP.

Section 3
Quality Assurance Objectives for Measurement Data

The overall quality assurance (QA) objective is to develop and implement procedures for field sampling, Chain-of-Custody, laboratory analysis, and reporting that will provide results such that the data may be deemed valid for the stated purpose. Specific procedures for sampling, Chain-of-Custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control (QC), audit, preventive maintenance of field equipment, and corrective action are described in other sections of this report. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability.

3.1 Precision

3.1.1 Definition

Precision is a measure of the degree to which two or more measurements of the same parameter are in agreement. It can be used to evaluate random error in a data set.

3.1.2 Field Precision Objectives

Field precision will be assessed through the collection of one field duplicate for every 20 or fewer investigative samples of a similar media (a minimum of 5 percent by medium).

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) and relative standard deviations (RSD) for three or more replicate samples. The equations to be used for precision in this project can be found in Section 12 of this QAPP. Precision control limits are provided in the Laboratory QA Manuals (Appendix B).

Laboratory precision shall be assessed through the analysis of a sample/sample duplicate pair and field duplicate pairs. For soil and groundwater matrices, laboratory precision shall be also assessed through the analysis of matrix spike/matrix spike duplicate (MS/MSD) samples. Note that each of the parameters of concern listed in Tables 1 and 2 of this QAPP are included in method spiking solutions for MS and MS/MSD analyses.

3.2 Accuracy

3.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. It can be used to evaluate the presence of bias or non-random error in a data set.

3.2.2 Field Accuracy Objectives

For volatile organic compounds (VOCs) in water, accuracy in the field will be accomplished through the use of field blanks. One volatile organic analysis (VOA) trip blank consisting of distilled deionized ultra pure water will be included along with each shipment of aqueous VOA samples. For soil gas samples, field accuracy will be assessed through the use of a tracer. If a qualitative tracer such as isopropanol is used, sample results may be subject to corrective measures if the detected concentration of qualitative tracer exceeds 10,000 ppbv. If a quantitative tracer such as helium is used, sample results may be subject to corrective measures if the detected concentration of the quantitative tracer exceeds 10-percent by volume. Field accuracy will also be insured by adherence to all sample handling, preservation and holding times, which are summarized by the matrix in Table 5.

The QC effort for field specific conductivity, pH, temperature, and turbidity measurements will include twice daily calibration of the instrument using standard solutions of known value according to the procedures specified in the instrument operating manual. The QC level of effort for the field measurement of pH consists of pre-measurement calibration and a post-measurement verification using two standard reference solutions each time as appropriate to the sample pH. During calibration, the instrument is adjusted until the proper measurement is obtained. If the instrument cannot be calibrated, it will be replaced. Specific conductance calibration is obtained through one point calibration with a standard. This instrument will also be replaced if it will not calibrate.

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of MS/MSD, standard reference materials (SRM), laboratory control samples (LCS) and surrogate compounds, and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 12 of this QAPP and the Laboratory SOPs which are included in Appendix C.

Note that all parameters of concern included in Tables 1 through 4 of this QAPP are included in method spiking solutions for the LCS and MS/MSD samples (where applicable). Also, in the case of sampling for VOCs in soil, use of the appropriate sampling techniques, as described in Subsection 4.4.1, will ensure data that is both accurate and representative of on-site conditions.

3.3 Completeness

3.3.1 Definition

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

3.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 12 of this QAPP. The field completeness objective for this project will be 90 percent or greater.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 12 of this QAPP. The laboratory completeness objective for this project, with respect to critical measurement parameters will be 95 percent or greater.

3.4 Representativeness

3.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the Workplan(s) is followed and that proper sampling techniques are used.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Using the proper analytical procedures ensures representativeness in the laboratory, appropriate methods, meeting sample holding times and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of facility conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the RCRA program. The rationale of the sampling network is discussed in detail in the Current Conditions Report (CCR) and the workplan(s).

3.5 Decision Rule

3.5.1 Definition

A decision rule is a statement that allows for a course of action or non-action to be taken, based on assumptions made to draw out and test its logical or empirical consequences.

3.5.2 Decision Rule Objectives

The decision rule objectives address the following:

- Define statistical parameter(s) characterizing the population (*e.g.*, mean, maximum, and percentile) and incorporate the scale of decision-making (*e.g.*, residential lot size). Typically, samples shall be collected discretely without compositing to provide greater indication of locally contaminated zones and hot spots. Discreet sampling will be used for site characterization. In latter phases of the project, such as following excavation of contaminated soil, composited samples may used in decision making.
- Identify action level(s) (*e.g.*, Soil Screening Level; Maximum Contaminant Level for drinking water; Ecological Data Quality Levels (EDQL) or a reference-based standard).

Develop "if/then" statements defining conditions that would cause the decision maker to choose among alternative actions (*e.g.*, remediation or no remediation). The screening levels specified in the Tables 1 through 4 of this QAPP will be used as preliminary decision levels *i.e.*, to determine if further investigation, risk assessment, and/or

remediation may be necessary. Preliminary decision rules for this facility are stated as follows:

- Analytical data from soil samples and passive soil gas samples will be used to identify potential source areas. If a source area is suspected based on historical operations, existing soil and groundwater data, qualitative passive soil gas survey data, and/or other lines of evidence, then that source area will be targeted for additional investigation and/or presumptive CM.
- In order to determine the extent of a COCs in groundwater, groundwater samples shall be taken from existing groundwater monitoring wells according to procedures specified in Section 4.5 of this QAPP. Groundwater and surface data will be used for the following activities:
  - To map the extent of the target parameters (Subsection 1.4.1) above the screening levels listed in Table 2;
  - To conduct groundwater modeling in order to determine whether contaminant migration is stable;
  - To define source areas which may be subject to CM;
  - To evaluate presumptive CM which target groundwater, if any; and
  - To conduct risk assessment activities, as needed.
- For soil gas samples, if the detected concentration of the tracer exceeds the designated maximum concentration (10,000 ppbv for a qualitative tracer such as isopropanol or 10-percent by volume for a quantitative tracer such as helium), samples affected by elevated concentrations of tracer will be subject to corrective measures such recollection of the affected samples.
- In order to determine the existence or extent of COC in soil gas, soil gas samples may be taken from existing soil gas monitoring points according to procedures specified in Section 4.8 of this QAPP. If COCs listed in Table 3 are identified in soil gas above the screening levels, then generated data for that COC may be subjected to a the following activities:
  - To map the extent of the target parameters (Subsection 1.4.1) above the screening levels listed in Table 3;
  - To conduct location specific Johnson-Ettinger Modeling/risk assessment; and/or
  - To identify properties for additional investigation and/or presumptive CM.
- If COCs listed in Table 4 are detected above decision levels in indoor air, then additional investigation will be performed and/or Presumptive CM initiated, as appropriate.

The primary purpose of the ongoing remedial investigation (RI) is to gather sufficient information to quantify and mitigate risk. As the RI progresses, additional decision rules may be developed and included in the appropriate workplan(s). Decision rules for CM may be dependent on remediation goals which have not yet been developed.

3.6 Comparability

3.6.1 Definition

Comparability is an expression of the confidence with which one data set can be compared to another.

3.6.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the Field Sampling sections of the QAPP and workplans are followed and that proper sampling techniques are used.

3.6.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented in the QAPP. Comparability is also dependent on similar QA objectives.

3.7 Sensitivity

3.7.1 Measures to Ensure Sensitivity of Field Data

Sensitivity in the field is ensured by adherence to manufacturers' specifications when using field equipment to measure pH, specific conductance, turbidity, temperature, organic vapors, and water levels.

3.7.2 Measures to Ensure Sensitivity of Laboratory Data

The fundamental QA objective with respect to the sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. For laboratory analyses, the sensitivity required will be the laboratory reporting limits. Tables 1 through 4 compare the reporting limits of selected compounds to the lowest applicable screening levels. For soil and groundwater Michigan Part 201 criteria were used to define relevant and appropriate screening levels. For soil gas and indoor air, appropriate screening levels were calculated based on current chemical toxicity data.

Complete tables of the Part 201 criteria for soil and groundwater, and the calculated risk-based criteria for soil gas and indoor air are included in Appendices D and E.

Laboratory reporting limits are included in Tables 1 through 4. With the exception of 1,2-dibromo-3-chloropropane and 1,2-dibromoethane, these reporting limits are less than the lowest applicable screening levels. Based on historical operations and existing data, bromated VOCs are not expected to be COCs. Therefore the listed reporting limits for 1,2-dibromo-3-chloropropane and 1,2-dibromoethane are considered acceptable for RI purposes, and an alternative method/laboratory are not necessary. The achievement of the listed reporting limits depends on instrument sensitivity and matrix effects. Therefore, monitoring the instrument sensitivity is important to ensure data quality through constant instrument performance. The instrument sensitivity will be monitored through the analysis of a method blank, a calibration check sample, and laboratory control samples.

3.8 Level of Quality Control Effort

The level of QC effort provided by the laboratory will be equivalent to the applicable level of QC specified under SW-846 and the laboratory Standard Operating Procedures (SOPs) in accordance with certification for the parameters to be tested, and as specified in the laboratory QA manual (Appendix B). Field blank, trip blank, method blank, field duplicate, laboratory duplicate, LCS, standard reference materials (SRM),and MS/MSD samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

- Field and trip blanks consisting of distilled water will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field-sampling program.
- Field blank samples are analyzed to check for procedural contamination at the facility that may cause sample contamination.
- Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage.
- Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures.
- Duplicate samples are analyzed to check for sampling and analytical reproducibility.
- MS/MSDs provide information about the effect of the sample matrix on the digestion and measurement methodology. Depending on site-specific circumstances, one MS/MSD should be collected for every 20 or fewer investigative samples of a given matrix. MS/MSD samples are designated/collected for organic analyses of soil and groundwater.

The general level of the QC effort will be one field duplicate and one field blank for every 20 or fewer investigative samples. One trip blank consisting of distilled deionized ultra pure water will be included along with each shipment of aqueous VOC samples. The frequency of duplicate and field blank samples to be collected is listed in Table 6 of this QAPP. QC Sampling procedures are specified in Section 4.13 of this QAPP.

The QC effort for field specific conductivity, pH, temperature, and turbidity measurements will include twice daily calibration of the instrument using standard solutions of known value according to the procedures specified in the instrument operating manual. Calibration of pH is obtained through two-point calibration with standards of pH 7 and pH 4 or 10. During calibration, the instrument is adjusted until the proper measurement is obtained. If the instrument cannot be calibrated, it will be replaced. Specific conductance calibration is obtained through one point calibration with a standard. This instrument will also be replaced if it will not calibrate.

Section 4
Sampling Procedures

The sampling procedures used in to conduct remedial investigation (RI) activities and presumptive corrective measures (CM) will be consistent for the objectives of this project. This Section provides field standard operating procedures (SOPs) for activities that have or are expected to be performed as part of the RI and Presumptive CM activities. Deviations from these SOPs will be described in the associated workplan and/or summary of field activates.

4.1 Soil Boring Procedures and Protocols

Soil boring procedures for surface and shallow soil borings will be accomplished and documented in accordance with the procedures detailed below.

4.1.1 Soil Boring Procedures

The soil borings will be drilled using 4.25–inch inside diameter (I.D.) hollow-stemmed augers, Roto-sonic™ techniques, hydraulic direct-push techniques, or manual methods (*e.g.*, a slide hammer or hand auger). Geoprobe® direct-push technology or equivalent may also be used at appropriate locations to advance soil borings. The Geoprobe® system mounted on a pickup truck or all-terrain vehicle uses hydraulic percussion to advance a narrow-diameter (2-inch nominal) drill tool. This drilling technique can be used to collect soil or groundwater samples or to install narrow-diameter wells.

The soil borings will be sampled using a split barrel sampling device or an acrylic liner. Soil sampling from Geoprobe® or equivalent borings will be accomplished using either a Macro-core® or Large-bore® soil sampler, or equivalent. Both are piston samplers equipped with an acrylic liner to collect soil samples from discrete intervals.

Soil samples from hand augers will be collected directly from the borehole unless adequate sample material cannot be obtained. In this case, the sample material will be collected from the auger itself. The core will be opened upon retrieval, and the on-site geologist will log and describe the samples using the Unified Soil Classification System (USCS) methods. The field sample description will include the USCS field classification.

Soil borings that are not converted into monitoring wells will be sealed/plugged. Bentonite chips or a bentonite slurry will be used to seal the boring to ground surface. The slurry will be placed using a tremie from the bottom up. Any asphalt or concrete that is disturbed will be patched with the appropriate material. A description of the

specific drilling techniques to be used will be included in the workplans. In general the drilling/soil sampling technique will be selected from available options given the following considerations:

- Any physical limitations (*e.g.* overhead clearance) of the area to be sampled;
- The nature of the soil;
- The expected potential for cross contamination;
- The amount and type of soil to be sampled;
- The project data quality objectives (DQOs); and
- Cost.

4.1.2 Boring Identification

Each soil boring installed is part of the RI or Presumptive CMs will be identified as follows: AA-xx-y. The AA variable will be filled with a code indicating the type of completion as listed below:

- "B" means perimeter or of-site soil boring location;
- "NS" means northern source area soil boring location;
- "SS" means southern source are soil boring location;

The variable xx will be filled in with the number of the boring. Borings will be numbered consecutively. The "y" suffix will be used to indicate unique boring parameters *e.g.*, "b" will indicate a borings installed in a utility corridor.

4.2 Monitoring Well Installation Procedures

Monitoring well installation and development procedures for temporary, shallow, and deep monitoring wells will be accomplished and documented in accordance with the procedures detailed below.

4.2.1 Temporary Well Installation Procedures

Temporary wells may be installed at the site to aid in selecting locations of permanent monitoring wells. The temporary wells will consist of either 1-inch–diameter, schedule 40 PVC monitoring wells or ScreenPoint® temporary stainless-steel screens and will be placed in Geoprobe®, or equivalent, boreholes to allow for groundwater sampling. These temporary well points may include a filter pack, placed around the well screen, and a bentonite seal, placed above the well screen to isolate the well. Water level

measurements and groundwater samples will be taken from the temporary wells in accordance with the procedures presented in Subsection 4.5.6.

4.2.2 Shallow Monitoring Well Installation Procedures

Soil borings will be converted to monitoring wells at the locations detailed in the workplans. Monitoring well casings and screens will arrive at the site in the original factory packaging and will remain in the packaging until the casing and screen materials are installed in the borehole. Permanent monitoring wells will typically be constructed of 2-inch Schedule 40 PVC flush-threaded riser pipe and equipped with a 5-foot screen.

After the screen and well casing are lowered into the borehole, the filter sand will be backfilled around the well screen and casing to a depth of approximately 2 feet above the top of the screen. Approximately 2 feet of bentonite chips will be used to seal the annular space above the sand pack. Additional bentonite chips or a bentonite slurry will be used to seal the remainder of the annular space above the sand pack, to approximately 1 foot below the ground surface. The slurry will be placed using a tremie from the bottom up. The remaining borehole annulus will be grouted with cement/bentonite grout to land surface.

Monitoring wells will be protected with a locking flush-mount or aboveground steel protective cover. The aboveground protective covers will be cemented in place around the PVC riser to stick up from the ground surface approximately 2 to 3 feet. The flush-mount protective covers will be installed flush with the ground surface, with the PVC riser finished below ground. The protective covers will be locked and clearly labeled for identification purposes. Monitoring well construction details will be recorded at the time of installation on well construction diagrams. Deviations from this protocol may be required to implement specific workplans. In that event details of the well installation practices will be included in the workplans.

4.2.3 Deep Monitoring Well Installation Procedures

For monitoring wells located in areas where contaminated soils could be dragged down into other, unaffected units, the following procedures will be used to install the wells. The wells will be constructed by double-cased methodology using either Roto-Sonic™ or hollow stem auger methods. An initial pilot hole will be advanced and logged using 4 ¼ –inch I.D. augers or casing to determine the depth of the affected unit or in the case of affected clay-rich sediments, to the bottom of the affected soil. Fourteen-inch O.D. augers or casing will then be used to advance a borehole into the unit of interest. An 8-inch I.D. steel casing will then be set to the depth of the borehole. The borehole

annulus will then be pressure grouted with a cement-bentonite grout to the land surface, and allowed to set for a minimum of 24 hours prior to continuing the well. The borehole will then be advanced using 4 ¼ – inch I.D. augers or casing in conjunction with 5 foot continuous split-barrel sampling through the affected zone to the base of the lower unit. Monitoring wells will be constructed with a 2 – inch Schedule 40 riser equipped with a 5-foot well screen. After the screen and well casing are lowered into the borehole, the filter sand will be backfilled around the well screen and casing to a depth of approximately 2 feet above the top of the screen. A bentonite slurry will be used to seal the entire annular space above the sand pack, to approximately 3 feet below the ground surface. The remaining borehole annulus will be grouted with cement/bentonite grout to land surface.

Monitoring wells will be protected with a locking flush-mount or aboveground steel protective cover. The aboveground protective covers will be cemented in place around the PVC riser to stick up from the ground surface approximately 2 to 3 feet. The flush-mount protective covers will be installed flush with the ground surface, with the PVC riser finished below ground. The protective covers will be locked and clearly labeled for identification purposes. Monitoring well construction details will be recorded at the time of installation on well construction diagrams.

4.2.4 Monitoring Well Development

Monitoring wells will be developed after the grout and well seal material has cured. Curing time will typically be 24 hours following well installation, although for shallow wells, where grout and well seal material is placed above the water table, well development may be performed immediately following well installation.

The development will remove fine particles around the well screen and filter pack to improve hydraulic communication between the well and the surrounding aquifer. Development will be accomplished using a pump or bailer to surge and purge the well. Development will be complete when the purge water turbidity measurements are less than 10 Nephelometric Turbidity Units (NTUs) as a goal. However, for wells that are screened in silt- or clay-rich units, this may be impractical. Development of these wells will be complete when the purge water is relatively clear (visibly) and free from suspended solids, or after approximately five well volumes have been evacuated. Development notes will be recorded on the well construction diagrams.

4.2.5 Well Identification

Each permanent well installed during the RI or Presumptive CM activities will be identified as follows: MW-xx-y. The variable xx will be filled in with the number of the well. Wells will be numbered consecutively, or in the event that the new wells are nested with a preexisting well(s), the number of the pre-existing well will be applied to the new well. The "y" suffix will indicate monitoring well depth: "S" indicates a shallow water table well and "D" indicates a deep well screened at or near the top of the lower clay unit.

4.3 Active Soil Gas Sampling Point Installation Procedures

Procedures for sub-slab and deep monitoring active sub-slab soil gas and deep soil gas monitoring points will be accomplished and documented in accordance with the procedures detailed below.

4.3.1 Sub-Slab Soil Gas Sampling Point Installation Procedures

Each sampling point will be constructed using a Geoprobe® or similar equipment. A one to three – inch diameter hole will be drilled through the concrete slab, and the hole will extend approximately 0.5 foot below the slab into the granular fill. Sampling points will be constructed with a stainless steel monitoring implant (Geoprobe® Vapor Implant AT8617S, or equivalent) equipped with a 6-inch screened interval and Teflon tubing. The screened interval will be set in the granular fill immediately below the slab. The monitoring point screen will be backfilled using glass beads, to approximately 0.5 foot below the ground surface. The remaining annulus will be sealed with concrete grout.

Sampling points will be protected with a flush-mount protective cover. The flush-mount protective covers will be installed flush with the ground surface. The protective covers will be clearly labeled for identification purposes. The monitoring points will be allowed to equilibrate for a minimum of 12 hours prior to sample collection.

4.3.2 Deep Soil Gas Sampling Point Installation Procedures

Soil borings will be converted to deep soil gas sampling points at the locations detailed in the workplans. Each sampling point will be constructed in a 2.5-inch-diameter borehole installed using a Geoprobe® as described above. Sampling points will be constructed with a stainless steel monitoring implant (Geoprobe® Vapor Implant AT8617S, or equivalent) equipped with a 6-inch screened interval and Teflon tubing. Unless otherwise specified in the workplan, the screened interval will be set to a maximum depth of 8.0 to 8.5 feet below ground surface. If saturated conditions are

encountered in the pilot boring, the screened interval will be set such that the bottom of the screen is 1 foot above the groundwater table. The screened interval will be set to a minimum depth of 5.0 to 5.5 feet below ground surface. The sampling point location specified in the workplan may be moved or abandoned if saturated conditions are encountered at a depth less than 6.5 feet below ground surface.

The suspended monitoring point screen will then be backfilled using glass beads, and the remaining annulus will be sealed with bentonite chips, to approximately 1 foot below the ground surface. The remaining borehole annulus will be grouted with cement grout to land surface. A sufficient length of Teflon tubing (at least 1 foot) will be left above the concrete grout seal so that the sampling apparatus could be connected directly to the sample point without a tubing extension.

Sampling points will be protected with a flush-mount protective cover. The flush-mount protective covers will be installed flush with the ground surface, with the excess Teflon tubing coiled below ground in the sample point vault. The protective covers will be bolted closed and clearly labeled for identification purposes. Sample point construction details will be recorded at the time of installation on well construction diagrams. The monitoring points will be allowed to equilibrate for a minimum of 12 hours prior to sample collection.

Deviations from this protocol may be required to implement specific workplans. In that event, details of the well installation practices will be included in the workplans.

4.3.3 Sample Point Identification

Each monitoring point installed during the RI or Presumptive CM activities will be identified as follows: AA-xx-y. The AA variable will be filled with a code indicating the type of completion as listed below:

- "SG" means deep soil gas sampling location; and
- "SV" means sub-slab soil gas sampling location.

The variable xx will be filled in with the number of the boring. Borings will be numbered consecutively. The "y" suffix will be used as needed to indicate unique boring parameters as defined in the appropriate workplan or investigation summary report *e.g.*, "b" may indicate a sample locations installed in a utility corridor.

4.4 Soil Sampling Procedures and Protocols

4.4.1 On-Site Soil Sampling Procedures

As on-site soil boring/wells are installed, soil samples which are collected using a split barrel sampling device or acrylic liner for soil classification purposes will also be subject to field screening with a photo-ionization detector (PID) as described in the SOP in Appendix A. This field screening will primarily be conducted for health and safety purposes. If the workplan includes collection of soil samples for laboratory analysis, soil samples will be split lengthwise and collected simultaneously in an airtight zipping bag for PID analysis and in sample containers for laboratory analysis.

Samples being submitted to an off-site laboratory for volatile organic compound (VOC) analysis will be collected and preserved in the field using either EPA Method 5035, Encore samplers or O2SI sample kits with methanol preservation. Samples collected for immediate on-site analysis may not be field preserved and will be collected in accordance with laboratory protocol, e.g. using laboratory provided lock-in-load samplers. Table 1 summarizes the type and number of containers needed for sample collection associated with this QAPP. Appendix F presents procedures for methanol preservation of soil samples. Field procedures for sample documentation, handling, storage, shipment, preservation, and Chain-of-Custody will be conducted in accordance with the procedures outlined in Subsection 4.13.

Soil sampling locations and sampling intervals will be consistent with the workplan and will be selected on the basis of historical site knowledge and the specific target of each soil boring. Modifications may be made in the field on the basis of PID results, visual/olfactory senses, accessibility, and drilling limitations.

4.4.2 Soil Sample Identification

Each soil sample collected part of the RI or Presumptive CMs will be identified as follows: AA-xx-y (zz-zz'), where AA-XX-y represents the soil boring or monitoring well location and (zz-zz') represents the sample depth interval.

4.5 Groundwater Sampling Procedures and Protocols

Standard field sampling protocol will be followed for all groundwater sampling events. Sampling personnel will be familiar with procedures and requirements of the QAPP, and the samplers will have a copy of the current sampling procedures in their possession, readily available for reference during groundwater sampling events.

4.5.1 Groundwater Sampling Order

To minimize cross-contamination, cleaner wells (lower detections of VOCs) will be sampled prior to wells with greater levels of VOCs. In general, upgradient wells and off-site wells at the perimeter of the furthest extent of VOCs will be sampled first, followed in order by upgradient on-site wells, off-site downgradient affected wells, on-site downgradient wells and source area wells.

4.5.2 Static Water Elevation

To determine the static water elevation (SWE), the static water level (SWL) will be measured prior to purging and sampling at each groundwater sampling location. On-site static water level measurements will be obtained at each location on the first day of the sampling event, prior to groundwater sample collection. The measurement will be obtained prior to purging the groundwater monitoring well. Each well will have a permanent reference point on the top of the well casing, designated top-of-casing (TOC), from which water level measurements will be taken. The reference point will be surveyed to the nearest 0.01 feet, and referenced to National Geodetic Vertical Datum (NGVD) or a local datum.

The measurement will be taken using an electronic water level meter with an accuracy of ± 0.01 feet. The meter will be decontaminated prior to each measurement. Minimum contact of the tape and probe/sounder and the water in the well is required to decrease the potential for cross contamination. Disposable gloves of appropriate materials will be used while determining the SWL.

Prior to collecting the measurement, field personnel will verify the location of the measuring point on the PVC casing. The probe will be slowly lowered into the well until the sounder beeps or the light becomes illuminated. In wells where caps are not vented, the cap will be removed and the water level will be allowed to equilibrate. Once the water level is equilibrated, the measurement will be read from the tape to the nearest 0.01-foot increment and recorded on the field notes. This measurement is the SWL as measured in feet below the TOC measuring point.

The SWE will then be calculated using the following equation:

SWE = TOC - SWL,

where

SWE = static water elevation (ft NGVD),
TOC = top of casing elevation (ft NGVD), and
SWL = static water level, depth to water below TOC (ft).

4.5.3 Field Readings

Physically or chemically unstable analytes will be measured in the field. Field measurements for turbidity, temperature, pH, specific conductance, reduction-oxidation (redox) potential, and dissolved oxygen (if appropriate) will be collected as required at each sampling point using the appropriate field probe or meter during purging and sampling procedures. A Hydrolab Quanta multimeter, or equivalent (*e.g.*, QED MP-20, YSI Series 6000), capable of measuring turbidity, pH, temperature, and specific conductance will be utilized.

Operation and maintenance instructions and procedures will comply with manufacturers' specifications. Field instrument calibration procedures will be performed a minimum of twice a day. Field readings will be documented in accordance with standard RMT field sampling forms.

Examples of field data sheets, meter calibration logs, and field note forms are given in Appendix G. A log of meter calibrations and checks should be maintained during each sampling event. The calibration and checks should be performed two times a day, following the procedures that are specified in the meter manual.

4.5.4 Purging Procedures

RMT will use an appropriate pump to obtain samples from the piezometers and monitoring wells using the following procedures:

- By utilizing the difference in the depth to water and the total depth of the well, along with the length of the well screen, the bladder pump or intake of the polyethylene/ polypropylene tubing will be placed in the well at the center of the screened interval, or at the center of the water column if the groundwater elevation intersects the well screen.
- For the bladder pump, connect the control box and air supply to the pump reel.

- Locate the air compressor/controller downwind of the well being sampled (bladder pump sampling only).
- Hook the pump discharge tubing to the flow-through cell containing pre-calibrated instruments.
- Purge at an initial rate of 0.5 liter or less per minute. Reduce flow rate if water is very turbid or water level is being rapidly drawn down. Check drawdown every 1 to 5 minutes to ensure drawdown does not exceed 1 foot/until drawdown stabilizes. If flow is reduced to lowest possible rate (~0.05 to 0.1 liters per minute) and drawdown continues, continue with purging at lowest rate.
- Once an appropriate purge rate is achieved, continue purging until stabilization is achieved. Stabilization to be complete when temperature change is ≤ 0.5°C, conductivity change is ≤ 10 percent, pH is ± 0.1 pH unit, and turbidity change is ± 10 percent (or less than 10 NTUs) between three successive 3-5 minute purging intervals.
- Field measurements and readings will be conducted utilizing a flow-through cell as detailed above. A Hydrolab Quanta multimeter (or a comparable alternative) and a flow cell system will be used to collect these readings.
- In addition, to providing basic groundwater quality characterization information, groundwater sample may also be field-analyzed for redox potential and dissolved oxygen. Redox potential and dissolved oxygen will be measured using the flow-through cell and the Hydrolab Quanta multimeter (or a comparable alternative).
- After stability is reached, disconnect the flow-through cell from the pump outlet line and fill all of the sampling bottles for the appropriate parameters in the appropriate manner as described below
- Well purging information will be recorded on the sampling form(s) included in Appendix G.

4.5.5 Groundwater Sampling Procedures

After purging the appropriate volume based upon the measured stabilized readings, the wells will be sampled utilizing the following procedures:

- Samples will be placed in new or pre-cleaned bottles provided by the laboratory. Those samples requiring the use of field preservatives will be placed into the pre-preserved bottles.
- Whenever possible, sample locations from relatively "clean" to relatively "dirty."
- Take precautions to ensure that the sampling equipment does not come in contact with the sample bottles, the purge container, or any potentially contaminated surface. Vehicle engines are shut off during purging and sampling.

- Obtain and record final readings of the field parameters when sampling.
- Prepare bottles by writing the date, the sampler's name, and the time of day in the sampler section.
- Fill bottles according to the following sample bottle filling sequence:
  - VOCs;
  - Ferrous Iron; and
  - Inorganic monitored natural attenuation (MNA) parameters.
- When sampling for parameters requiring filtration, insert an in-line filtration cartridge/capsule into the end of the pump discharge tubing, or pressurized bailer. Fill the pre-preserved bottles with water discharged from the in-line filtration cartridge.
- Conduct the procedures for sample documentation, handling, storage, shipment, preservation, and Chain-of-Custody in accordance with the procedures outlined in Subsection 4.13.
- When sampling is complete, place the dedicated polytubing or bladder pump tubing back in the well and move on to the next location. Typically it is not necessary to decontaminate the flow-through cell between sampling points.

Because formation conditions at some wells may not recharge a well quickly enough to keep up with even very low purge rates, a well or piezometer may be purged dry before obtaining stabilization. If this occurs, samples will be collected using the same pump and dedicated tubing after the well has recharged sufficiently to allow for the sample containers to be filled.

A new pair of protective gloves will be used at each well. The gloves will be changed if they become contaminated or damaged during purging or sample collection activities at any given well.

4.5.6 Borehole Groundwater Sampling

Groundwater samples may be collected directly from boreholes. At these locations, a Geoprobe® groundwater sampling tool (Screen-Point-15, Screen-Point, Perma-Screen, or Mill-Slot), or equivalent, will be positioned in the borehole at the selected sampling interval. Groundwater purging and sample collection will be completed using a mini-bailer; dedicated Tygon and high-density polyethylene tubing with a peristaltic pump; or a foot valve–equipped positive displacement pump.

Water level measurements will be collected in accordance with Subsection 4.5.2 of this QAPP. At each sampling point, the well will be developed by purging prior to sample collection. A minimum of 1 liter of water will be purged from each well point. Should the well points purge dry before 1 liter is evacuated, purging will be discontinued, the water level will be allowed to recover, and the sample will be collected.

4.5.7 Field Filtering

Field filtration is to be performed in the field during sample collection because even short delays can change water chemistry and affect the measured concentrations of select parameters such as metals. The field filtering will be accomplished using a 0.45-micron in-line filtration cartridge/capsule, which is inserted into the end of the pump discharge tubing. Filtered samples will be collected into pre-preserved sample bottles as water exits the filter. The filters will be changed between each sampling point.

4.6 Surface Water Sampling

If surface water sampling is necessary, it will be conducted in the following manner. Field personnel will access surface water sampling locations either from the banks of the water body, or by using a boat or canoe. Wherever possible, surface water will be collected approximately three feet from the shore by dipping the appropriate sample container into the water. Unpreserved sample containers will be slowly lowered into the water so as not to disturb the bottom sediment and allowed to fill. For samples requiring preservation, water from a clean unpreserved sample container will be poured slowly into the required pre-preserved sample containers.

Surface water samples require no field filtration, but due to seasonal rainfall and stream flow conditions, differences may exist in the amount of suspended solids in the water samples. Field personnel will document sample collection procedures on field data forms. Documentation will include color, odor and a visual assessment of sample turbidity. Field procedures for sample documentation, handling, storage, shipment, preservation, and Chain-of-Custody will be conducted in accordance with the procedures outlined in Subsection 4.13.

4.7 Storm Sewer Sampling

If storm sewer water sampling is necessary, it will be conducted in the following manner. Field personnel will access the storm sewer either though existing manholes or at outfall locations. Prior to sample collection, a PID will be used to qualitatively determine whether VOCs are present in the atmosphere of the storm sewer. Storm water will be collected by dipping the appropriate sample container into the water. Unpreserved sample containers will be slowly lowered into the water so as not to disturb the bottom sediment and allowed to fill. For samples

requiring preservation, water from a clean unpreserved sample container will be poured slowly into the required pre-preserved sample containers.

Storm water samples require no field filtration, but due to seasonal rainfall and stream flow conditions, differences may exist in the amount of suspended solids in the water samples. Field personnel will document sample collection procedures on field data forms. Documentation will include color, odor and a visual assessment of sample turbidity. Field procedures for sample documentation, handling, storage, shipment, preservation, and Chain-of-Custody will be conducted in accordance with the procedures outlined in Subsection 4.13.

4.8 Active Soil Gas Sampling

Standard field sampling protocol will be followed for all active soil gas sampling events. Sampling personnel will be familiar with procedures and requirements of the QAPP, and the samplers will have a copy of the current sampling procedures in their possession, readily available for reference during soil gas sampling events. Active soil gas sampling procedures are consistent with the procedures outlined in MNDRE Remediation and Redevelopment Division Operation Memorandum Number 4 (Op. Memo 4). These methods are summarized below.

- Each sampling system will consist of a laboratory supplied certified clean 1-liter SUMMA® canister and a dedicated flow controller or critical orifice. The laboratory will set each flow controller to maintain a sampling rate of approximately 100 mL per minute. Each canister will be evacuated to a nominal 26 to 30 inches of mercury (in. Hg), sealed, and shipped to the field under Chain-of-Custody documentation;
- Fresh 0.25-inch I.D. Masterflex tubing and a Teflon tubing extension, if needed, will be used to connect to the soil gas sample probe and the peristaltic pump;
- Assemble the sampling apparatus according to the instructions provided by analytical laboratory (Appendix H). The SUMMA® canister is connected to a dedicated flow controller by tightening the Swagelock® connection. A 6-inch section of ¼-inch Teflon tubing is then connected to the flow controller using a Swagelock nut and ferrule. The tubing is fitted with a moisture filter (female luer lock fitting). An approximately 3-inch section of Masterflex tubing, having an inner diameter of ¼ inch, is then connected to the moisture filter (male slip luer fitting). All connections are checked and tightened as appropriate;
- The sample information, including the canister number, flow controller number, initial sample canister vacuum is recorded on RMT standard soil gas sampling forms (Appendix G);
- Typically, future soil gas sampling will be conducted using a quantitative tracer, such as helium, using the following procedures:
  - Connect fresh tubing to the tank of tracer gas,

  - Cover the sample probe with a clear plastic bag and weight the bag around the perimeter of the sample probe to ensure that the tracer gas remains in the atmosphere around the sample,
  - Cut a small hole in the plastic bag and insert the tracer gas tubing,
  - Cut another small hole in the bag to connect the sample probe to the peristaltic pump,
  - Seal both holes in the plastic bag with tape,
  - Open the valve on the tracer gas to enrich the atmosphere around the sampling apparatus,
  - Using a portable gas meter, measure and record the concentration of tracer gas in the off-gas from the sample probe during sample probe purging as described below,
  - If the tracer gas concentration is greater than 10-percent, perform field corrective measures (adjusting/tightening fittings, using clay to improve the seal around the sample probe tubing, *etc.*) until the tracer gas concentration is less than 10-percent. If the concentration remains above 10-percent, report the problem to the Technical Coordinator and move to the next sample location. Do NOT collect a sample;

- The peristaltic pump will be used to purge the vapor probe for approximately 5 minutes, removing approximately 3 volumes (2 liters) of vapor to ensure that the sample will be representative of the soil gas and not the stagnant vapor in the probe;
- Once purging is complete, a PID will be used to screen the off-gas from the sampling location;
- Once the PID reading is complete, the sample apparatus is connected directly to the sample point or to the Teflon tubing extension by fitting the Masterflex tubing over the Teflon tubing;
- The valve on the sample canister is then opened to begin the collection of the soil gas sample for laboratory analysis. The initial canister vacuum and start time are recorded on the field form;
- Historically isopropyl alcohol has been used as a tracer. Future soil gas sampling will be conducted using a quantitative tracer as described above, unless otherwise indicated in the sampling workplan. If using a qualitative tracer, such as isopropyl alcohol, use the following procedures:
  - After sample collection has begun, apply the tracer to a paper towel,
  - Place the towel next to the sample point,
  - Cover the sample apparatus with a fresh clear plastic bag to ensure that the tracer gas remains in the atmosphere around the sample, and

  - Request laboratory analysis of the applies tracer gas on the Chain-of-Custody form;

- Monitor vacuum gage during sample collection, a rapid drop in canister pressure indicates a leak in the sample collection apparatus. If a leak is detected in the field, the affected sample may be subject to field corrective measures;
- After approximately 10 min, the dedicated flow controller on the sample canister is closed, and the final vacuum reading and sample stop time are recorded; and
- Field procedures for sample documentation, handling, storage, shipment, preservation, and Chain-of-Custody will be conducted in accordance with the procedures outlined in Subsection 4.13. Field personnel will document sample collection procedures on field data forms or field notebooks.

4.9 Air Sampling

Standard field sampling protocol will be followed for all indoor air sampling events. Sampling personnel will be familiar with procedures and requirements of the QAPP, and the samplers will have a copy of the current sampling procedures in their possession, readily available for reference during soil gas sampling events. These methods are summarized below.

- Each sampling system will consist of a laboratory supplied cleaned and certified 6-liter SUMMA® canisters and a dedicated flow controller or critical orifice. Each canister will be evacuated to a nominal 26 to 30 inches of mercury (in. Hg), sealed, and shipped to the field under Chain-of-Custody documentation;
- The sampling apparatus is assembled according to the instructions provided by analytical laboratory (Appendix H). The SUMMA® canister is connected to a dedicated flow controller by tightening the Swagelock® connection. A 6-inch section of ¼-inch Teflon tubing is then connected to the flow controller using a Swagelock nut and ferrule. All connections will be checked and tightened as appropriate;
- The sample information, including the canister number, flow controller number, initial sample canister vacuum is recorded on RMT standard soil gas sampling forms (Appendix G);
- The valve on the sample canister is then opened to begin the collection of the indoor air sample for laboratory analysis. The initial canister vacuum and start time is recorded;
- After the designated sample collection time (8-hours) has elapsed, the dedicated flow controller on the sample canister is closed, and the final vacuum reading and sample stop time are recorded.
- Field procedures for sample documentation, handling, storage, shipment, preservation, and Chain-of-Custody will be conducted in accordance with the procedures outlined in Subsection 4.13. Field personnel will document sample collection procedures on field data forms or field notebooks.

4.10 Passive Soil Gas Monitoring Procedures

All passive soil gas monitoring points will be temporary. Procedures for the installation, sampling and abandonment of passive soil gas monitoring points will be accomplished and documented in accordance with the procedures detailed below.

4.10.1 Passive Soil Gas Sample Point Installation Procedures

To install each passive soil gas (PSG) sampler, a 1¼ inch-to 1½ inch-diameter hole is made to an approximate depth of 12 inches using a hammer drill. A ½ inch-diameter drill bit is then used to extend a smaller diameter hole to a two- to three-foot depth. A one-foot long, one-inch diameter sanitized metal pip is lowered into the sample hole. Any portion of the pipe remaining above grade is cut flush with the ground surface, using a pipe cutter. A tapping dowel and hammer are used to push the pipe into the base of the drilled hole. Once the sample point has been constructed, a PID measurement is taken from the hole and then the laboratory provided BeSure Sampler is installed in the hole using the following procedures:

- Unwind the retrieval wire from the sampler vial.
- Holding the capped end of the vial in one hand, pull the wire tight (to straighten it) with the other hand.
- Remove the solid cap on the sampler vial and replace it with a sampling cap (a one-hole cap with a screen meshing insert). Place the solid cap in the clean cap storage container.
- Lower the sampler, open-end down, into the metal pipe approximately four inches so that the retrieval wire sticks out of the hole.
- Bend the end of the wire over the top of the pipe so that the coil of wire hangs over the top and outside of the pipe.
- Plug the top of the hole with a wad of aluminum foil. Using the tapping dowel, push down the aluminum foil so it forms a seal on the metal pipe and rests approximately ¼ inch below the surface.
- Cover the hole to grade with a ¼ inch thick concrete patch.
- Clearly mark and label the sample location and number with a paint pen.
- Record sample information, including sample number, grid location, date and time of placement on RMT standard passive soil gas sampling forms (Appendix G);
- Leave the sampler in place for approximately seven days.

4.10.2 Passive Soil Gas Sample Point Abandonment Procedures

After approximately seven days, use the following procedures to remove each PSG sampler and abandon the sample point:

- Use a small chisel and hammer to remove the concrete patch and expose the aluminum foil.
- Remove the aluminum-foil plug, using the scratch awl, as necessary.
- Use the retrieval wire to remove the sampler from the hole.
- Holding the sampler upright, clean the sides of the vial with a clean towel (especially close to the sampling cap).
- Remove and discard the sampling cap, cut all the wire from the vial with the wire cutters, and clean the vial threads completely with a clean gauze cloth.
- Firmly screw one of the solid caps from the cap storage container on the sampler vial and clean the vial completely with the gauze cloth.
- With a ballpoint pen record the sample number, corresponding to the sample location, on the cap's label. Do not use a permanent marker.
- Record the date and time of retrieval on RMT the standard passive soil gas sampling forms (Appendix G);
- Place the sealed and labeled sampler vial in a smaller 3" x 4" plastic sampler bag. Then place the individually bagged and labeled sampler into the larger bag labeled "Return Shipment Bag." Each return shipment bag should be returned with approximately 40 samplers.
- Included one trip blank with each return shipment bag.
- After all samples have been retrieved, verify that the caps on each sampler are sealed tightly and that the seals on the sampler bags are closed. Verify that all Samplers are stored in the Return Shipment Bag, which contains an adsorbent pak. Seal the Return Shipment Bag and place it in the upper tray of the Field Kit, and place the provided tools and materials in the lower compartment of the Field Kit.
- Use concrete to fill and patch sampling holes.

4.10.3 Sample Point Identification

Each monitoring point installed during the RI or Presumptive CM activities will be identified as follows: PSG-xxx-y. The variable xxx will be filled in with the number of the monitoring point. Monitoring points will be numbered consecutively, or in the event that the new points are installed adjacent to existing sample location, the number

of the pre-existing well will be applied to the new well. The "y" suffix will indicate the sample event as described in the workplan.

4.11 Aquifer Testing

Aquifer testing, including slug tests pilot program tests, and large scale pump tests will be conducted as described in the appropriate workplan.

4.12 Aquifer and Fate Transport Modeling

Information collected from aquifer testing, including slug tests, pilot program tests, and large-scale pump tests may be collected and applied in developing a conceptual groundwater flow model. Groundwater modeling will be completed using a program, such as MODFLOW (McDonald and Harbaugh, 1988) along with the particle tracking post-processing model MODPATH. In addition, this groundwater model may be used in conjunction with another solute transport modeling package (MT3D). These modeling programs will be applied in accordance with ASTM D5447-04 *Standard Guide for Application of a Ground-Water Flow Model to a Site Specific Problem.*

4.13 Field QC Sampling Collection/Preparation and Identification Procedures

Field QC sample collection will include blind field duplicates, field equipment blanks, trip blanks and matrix spike and matrix spike duplicate samples (MS/MSD). Theses quality control samples will be analyzed for the same COCs as the associated samples, blank and duplicate samples may not be analyzed for other parameters, e.g. MNA parameters. To provide a check on cross-contamination, one equipment blank will be collected after sampling and decontamination at one of the contaminated wells included in the monitoring program. Trip blanks will only be analyzed for VOCs.

Field quality control samples are collected to assess the quality of the analytical data and to evaluate sampling and analytical reproducibility (precision). Field quality control samples will consist of duplicate samples, field blanks, and trip blanks. Acceptance criteria for these samples will be in accordance with the current version of the laboratory QA manual. The collection frequency for the various QC samples is summarized in Table 6.

4.13.1 Blind Field Duplicates

Blind field duplicate samples will be collected one per 20 samples or less per similar media. Soil and water field duplicates will be prepared by splitting a single sample between two separate containers. Soil gas field duplicates will be collected either by collected two samples concurrently using a metal "T" to split the sample into separate

SUMMA® canisters or by collecting two samples consecutively at the duplicate sample location. Air and passive soil gas sample field duplicates will be collected concurrently at the duplicate sample location. The results will be used to evaluate sampling and analytical reproducibility (precision). Points where duplicate samples are to be collected will be selected by the field personnel to provide a range of expected contamination concentrations in the field, and will be submitted as blind duplicates to the laboratory.

The first three characters "DUP," followed by a 2-digit number to be assigned in the sequence collected, will identify field duplicate samples. Therefore, the first duplicate sample will be identified as "DUP-01," and the second duplicate sample will be identified as "DUP-02."

4.13.2 Field Equipment Blanks

In order to provide a check on cross-contamination, one equipment blank will be collected after sampling and decontamination at one of the contaminated wells included in the monitoring program. Field equipment blanks will be collected for groundwater samples only, and will be analyzed to assess procedural errors in sampling and equipment decontamination, if necessary. An equipment blank will be collected using the same equipment as the samples, *e.g.* a pump, bailer, or Geoprobe® groundwater sampling tool. The blank will be analyzed for the same parameters as the samples from the wells.

Field blank identifiers will consist of the letters "FB" followed by a 2-digit number assigned in sequence similar to duplicate and trip blank numbers. Therefore, the first two field blanks used will be labeled as "FB-01" and "FB-02," respectively.

4.13.3 Trip Blanks

Trip blanks will be analyzed to assess the possible cross-contamination of aqueous samples resulting from diffusion of VOCs through the septa during sample shipment, as well as possible contamination from fugitive VOCs in the atmosphere at the site. Trip blanks are prepared in the laboratory. Trip blank samples consist of 40-mL vials that are filled with deionized water in the laboratory prior to going to the field. Trip blanks will accompany the VOC water sample bottles from the laboratory to the field and will be returned with the VOC samples to the laboratory. A separate trip blank will be included in every shipping container that includes water, soil, and/or PSG samples intended for VOC analysis.

Trip blank identifiers will consist of the letters "TB" followed by a 2-digit number assigned in sequence similar to duplicate and field blank numbers. Therefore, the first two trip blanks used will be labeled as "TB-01" and "TB-02," respectively.

4.13.4 Matrix Spike and Matrix Spike Duplicate Samples

MS/MSD samples will be analyzed for soil and water matrices to provide information about the effect of the sample matrix on the digestion and measurement methodology. MS/MSD samples are laboratory rather than field quality control samples.

Points where MS/MSD samples are to be collected will be selected by the field personnel and with the sampling location identified. The MS/MSD sample will be collected using the same equipment as the samples, *e.g*. a pump, bailer, or Geoprobe® groundwater sampling tool and collected immediately after the primary VOC sample is collected. Typically MS/MSD samples will be collected at a rate of 1 per 20 water samples. If only one or a few samples are collected, field personnel may not collect a MS/MSD sample. However, laboratory batch MS/MSD are analyzed, as required, at a rate of one per 20 samples or less per similar media will be included in the analytical report regardless of whether the MS/MSD sample was collected at the site.

Assigning the name of the sampling point with the suffix "MS/MSD" will identify MS/MSD samples. Therefore, a MS/MSD sample collected from "MW-01"will be identified as "MW-01 MS/MSD".

4.13.5 Obtaining Contaminant Free Sample Containers

Pre-cleaned sample containers for all soil, groundwater, soil gas, and air samples will be obtained from analytical laboratories such as TriMatrix Laboratories (Trimatrix), Pace Analytical Laboratories (Pace) or sample bottle suppliers such as I-Chem Research, Inc., in New Castle, Delaware, O2SI in Charleston, South Carolina and Daniel Scientific in Simpsonville, South Carolina. The preparation of sample bottles (*e.g.,* preservative added) will be documented.

4.13.6 Sample Preservation Methods/Containers/Holding Times

The approved contract laboratory will provide the sample preservation methods, containers, and holding times for the parameters to be analyzed. Sample preservation requirements are presented in Table 5. Upon collection, the soil and water samples will be placed within a cooler containing sufficient ice sealed in plastic bags to cool and maintain each sample's temperature at or near 4 degrees Celsius (°C) through receipt by

the laboratory. The temperature in the cooler will be measured when they are received at the laboratory.

4.13.7 Sample Bottle Labels

Each sample bottle will be labeled so that the analytical laboratory has the following information:

- Site identification;
- Sampling date and time;
- Sample identification or location;
- Sampling crew; and
- Sample preservation method.

4.13.8 Method of Transport

The method of transport used should be one that will ensure that the samples will be delivered to the laboratory overnight, such as Federal Express or transported by the laboratory courier.

4.13.9 Transport Container and Packaging

Soil and Groundwater Samples

Soil and groundwater samples will be transported in a cooler. Soil and groundwater sample packaging procedures are as follows:

- Place cushioning (bubble pack) in the bottom of the cooler.
- Place cushioning material around all glass bottles.
- Place small bags of ice throughout the cooler or place loose ice around and over the top of the samples. Place the completed Chain-of-Custody Record in a Ziploc® bag (or equivalent watertight plastic bag), and tape it to the inside cover of the cooler.
- Tape shut the drain on the cooler, and wrap tape around the cooler completely in two locations.

Soil and groundwater samples will be transported in a cooler of sufficient size to maintain an internal temperature at or below 4°C through receipt of the sample at the laboratory. Ice sealed in a plastic bag will be placed in the cooler to cool and maintain the samples' temperature at 4°C. Upon receipt of the

coolers at the contract laboratory, the temperature in the cooler will be recorded on the Chain-of-Custody Record or sample log. In the event that the internal cooler temperature exceeds the recorded groundwater sampling temperature, a data qualifier will be placed in the report table and corrective measures in sample cooling procedures will be made to ensure that subsequent sampling events meet the temperature performance standard.

Soil Gas and Indoor Air Samples

Soil gas and indoor samples will be transported in a cardboard box, or similar container. Soil gas and indoor air sample packaging procedures are as follows:

- Place cushioning (bubble pack) in the bottom of the box.
- Place cushioning material around each SUMMA canister and regulator.
- Place additional cushioning material in the box, as needed to fill the remaining space.
- Place the completed Chain-of-Custody Record at the top of the box.
- Tape the box shut, and wrap tape around the box completely in two locations.

4.13.10 Sample Chain-of-Custody

The possession of samples must be traceable from the time of collection through the use of Chain-of-Custody procedures. Specific Chain-of-Custody Records must accompany all sample shipping containers to document the transfer of the shipping containers and samples from the field collection point to the laboratory that is receiving the samples for analysis. The procedures to be implemented are as follows:

- Identify and label each sample in the field.
- Complete Chain-of-Custody Records in the field, indicating sample identification, the number of containers filled, the sampling date, the sampling time, and the sample collector's name.
- Pack the shipping containers with the samples, the Chain-of-Custody Records, and the ice packs, as appropriate. Each set of containers to be shipped together is assigned a Chain-of-Custody Record, which travels with the sample containers.
- Seal and ship the containers to the appropriate laboratory. Common carriers or intermediate individuals will be identified on the Chain-of-Custody Record, and copies of all bills-of-lading will be retained.

– Receive and check the shipping containers in the laboratory for broken seals or damaged sample containers.

If an error is discovered on a sample Chain-of-Custody Record, the person who made the error should correct it when possible. No erroneous material is to be erased. Rather, a single line shall be drawn through mistakes. The date and the initials of the person who is making the correction will be written beside the correction. This procedure applies to words or figures that are inserted or added to a previously recorded statement.

If a Chain-of-Custody Record is damaged in shipment, the field technician will prepare a written statement detailing the pertinent information, including how the sample(s) was collected. The statement should include information such as field log entries regarding the sample.

4.13.11 Laboratory Sample Processing Time

The laboratory will process the samples using a standard turnaround time (TAT) of approximately 14 days.

4.14 Sampling Equipment Decontamination

4.14.1 Soil Boring Equipment Decontamination

Hollow-stem auger and Roto-sonic™ drilling equipment will be steam-cleaned prior to arrival at the site and between each soil boring. All reusable Geoprobe® drilling equipment, sampling apparatus, and excavation equipment in contact with site soils (*e.g.* rods, temporary well screens, trowels, spoons, knives, backhoe bucket, etc.) will be properly decontaminated using a solution of laboratory-grade soap such as Alconox, rinsed with deionized water, and allowed to air dry between each sample location.

4.14.2 Groundwater Sampling Equipment Decontamination

Static water level meters will be rinsed with deionized water between monitoring wells.

Monitoring wells sampled with dedicated bladder pumps or a peristaltic pump with dedicated tubing will not require decontamination. However, if a single bladder pump is used to sample more than one monitoring well, that bladder pump will be decontaminated before use in another monitoring well. The bladder pump used will be decontaminated by dismantling the pump and disposing of the used bladder. The pump, screen and o-rings are then placed it in a container (large enough to allow the

entire length of the pump to be submerged) with laboratory-grade soap and water. The pump is then scrubbed with soapy water. Following this, the pump will be rinsed in a separate clean container of distilled water.

4.15 Investigation Derived Waste Management

Soil cuttings derived from soil boring and monitoring well installation activities will be stockpiled in either DOT 55-gallon drums or roll-off containers. These soils will be stored onsite pending soil analytical data and will be disposed of once the field investigation activities are complete. All decontamination and purge waters will be properly contained and temporarily stored on-site until they can be properly shipped and disposed of at an off-site treatment location. All sampling gloves, Tyvek, and other PPE will be double-bagged and disposed of in a municipal waste dispenser.

Section 5
Custody Procedures

Custody is one of several factors that are necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including originals of all laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if:

- the item is in the actual possession of a person;
- the item is in the view of the person after being in actual possession of the person;
- the item was in actual physical possession but is locked up to prevent tampering; or
- the item is in a designated and identified secure area.

5.1 Field Custody Procedures

5.1.1 Field Notes and Documentation

Field notes will provide the means of recording the data collection activities that are performed. Forms that will be utilized during sample events are included as Appendix G. These forms should be completed in the field as the data are being obtained. Entries into the notes will contain a variety of information. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory.

Note takers should always keep the goal of the field assignment and the intended use of the notes foremost in their mind. The notes should be complete and understandable enough so that someone not associated with the actual field project can use them for the intended purpose without the need to question the note taker or other members of the field crew about the correct interpretation of the notes. There should also be an awareness of what the notes or information might possibly be used for purposes beyond the primary purpose of the field investigation. Field staff should make a point of questioning the project manager or technical coordinator if they are unclear as to the purpose of the field investigation and the level of detail required in the field notes.

At the beginning of each day, the date, the start time, the weather conditions, the names of all sampling team members present, the level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, the field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field notes.

Equipment used to make measurements will be identified, along with the date of calibration, the time of sampling, the sample description, the depth at which the sample was collected, and the volume and the number of containers. The sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description. Measurements made and samples collected will be recorded in the field notes. All entries will be made in blue or black ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark and initialed. Entries into field notes will be legibly written in indelible ink and will provide a clear record of all field activities. The field notes will also be used to document the samples that are collected, the sample date and time, the sample identification, general site observations, the problems encountered, and any other information that may be relevant.

5.1.2 Field Chain-of-Custody Procedures

- The field sampler is personally responsible for the care and the custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.
- Bottles will be labeled with sample numbers and locations using waterproof ink, unless prohibited by weather conditions. For example, a field note entry would explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.
- The Technical Coordinator will review field activities to determine whether proper custody procedures were followed during the fieldwork and to decide if additional samples are required.

5.1.3 Transfer of Custody of Shipment Procedures

The possession of samples must be traceable from the time of collection through the use of Chain-of-Custody Record procedures. Specific Chain-of-Custody Records must accompany sample-shipping containers to document the transfer of the shipping containers and the samples from the field to the laboratory receiving the samples for analysis. The procedures to be implemented are as follows:

- Properly identify and label each sample in the field with indelible, waterproof ink.
- Complete the Chain-of-Custody Records in the field, indicating the sample identification, the containers filled, the sampling date, the sampling time, the sample collector's name, and the sample preservation method, if applicable. This information should also be noted on the field logs.
- Repack the shipping containers with samples, Chain-of-Custody Records, and ice packs, as appropriate. Each set of sample containers to be shipped together in a single shipping container is assigned a Chain-of-Custody Record, which travels with the shipping container.
- Seal and ship containers to the appropriate laboratory. Common carriers or intermediate individuals shall be identified on the Chain-of-Custody Record, and copies of bills-of-lading will be retained.
- Receive and check the shipping containers in the laboratory for broken seals or damaged sample containers. If no problems are noted, the samples are logged into the laboratory, and the Chain-of-Custody Record is completed. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.
- Include a copy of the Chain-of-Custody Records with the analytical data.

While filling out the Chain-of-Custody Record, it is important to write legibly. Errors are to be corrected by first drawing a single line through the incorrect information and then entering the correct information. All corrections are to be initialed and dated by the person making the correction. This procedure applies to words or figures that are inserted or added to a previously recorded statement.

Completed Chain-of-Custody Records will be placed in a plastic bag, sealed, and taped to the inside cover of the shipping container. Ice will be added to the sample container as appropriate. The shipping container will then be sealed, dated, and shipped to the laboratory using an overnight delivery service or courier.

A separate sample receipt is prepared whenever samples are split with a government agency. The receipt is marked to indicate with whom the samples are being split. The person who is relinquishing the samples to the agency should request the agency representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted on the receipt and in the field notes.

If a Chain-of-Custody Record is lost in shipment, a written statement will be prepared by the person who collected the samples listing the samples that were recorded on the

lost form and describing when and how the samples were collected. The statement should include information such as field notes regarding the sample. This statement is submitted to the RMT project manager for further action, as necessary.

5.2 Laboratory Custody Procedures

Laboratory custody procedures for sample receiving and log-in; sample storage and numbering; tracking during sample preparation and analysis; and storage of data are described in the laboratory QA Manuals, provided in Appendix B of this QAPP. Examples of laboratory Chain-of-Custody traffic reports along with instructions for completion are included in Appendix I of this QAPP.

5.3 Final Evidence Files Custody Procedures

RMT is the custodian of the evidence files and maintains the contents of the evidence files, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews. The respondent and their contractor will comply with Section IX of the consent order regarding record preservation.

Section 6
Calibration Procedures and Frequency

This section describes the procedures for determining working concentration ranges for specific analytes and for maintaining the accuracy of the instruments and measuring equipment that are used for conducting field tests and laboratory analyses. These instruments and equipment should be calibrated prior to each use or on a periodic basis.

6.1 Field Equipment Calibration

Instruments and equipment used to gather and generate or to measure environmental data will be calibrated with sufficient frequency and in such a manner that the accuracy and reproducibility of the results are consistent with the manufacturers' specifications.

Equipment to be used during the field sampling will be examined to confirm that it is in good operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that maintenance requirements are being observed. A spare pH electrode and a non-mercury thermometer will be sent to sampling locations where pH and temperature measurements are required, including those locations where a specific conductivity probe/thermometer is required. Personnel using methods of investigation that are standard to the engineering and environmental science professions will carry out field measurements and observations. Field activities will be documented on the field logs to provide a record for subsequent evaluation of data precision, accuracy, and completeness. An electronic water level indicator will be the primary method of measuring groundwater levels. The meter has a coated steel cable with a sensor that activates a buzzer and/or light on the probe when it comes in contact with water. The cable is marked with permanent 0.01-foot increments.

Field instruments include equipment capable of measuring turbidity, pH, temperature, specific conductance, and organic vapors. The following field meters and equipment may be utilized:

- Hydrolab Quanta multimeter, or equivalent (*e.g.*, QED MP-20, YSI Series 6000), capable of measuring turbidity, pH, temperature, specific conductance, reduction-oxidation (redox) potential, and dissolved oxygen, and
- Mini Rae 2000 photo-ionization detector, or equivalent (*e.g.*, Thermo Scientific OVM 580B) equipped with an 11.7 eV lamp.

Operation and maintenance instructions and procedures for the field equipment will follow manufacturers' specifications. Field instrument calibration procedures will be performed daily. Field readings will be documented utilizing the field forms included in Appendix G.

Physically or chemically unstable analytes will be measured in the field. Field measurements for turbidity, temperature, pH, and specific conductivity will be collected as required at each sampling point using the appropriate field probe or meter during purging and sampling procedures.

6.2 Laboratory Instrument Calibration

Calibration is required to demonstrate that instruments that are used to perform quantitative chemical analysis are operating properly. Correct operation is important in meeting sensitivity and in establishing detection limits. There are two types of calibration: (1) operational calibration, which is performed prior to instrument usage (*i.e.*, standard curves); or (2) periodic calibration, which is performed at prescribed intervals.

6.2.1 Calibration Program

A formal calibration program will control instruments and equipment that measure a quantity. Development and implementation of the calibration program will be the responsibility of the contract laboratory. These programs are described in detail in the laboratory quality assurance (QA) manuals and the laboratory standard operating procedures (SOPs) found in Appendices B and C.

Calibration Procedures

Recognized procedures (USEPA, ASTM, and manufacturers' instructions) will be used when available. Written calibration procedures will include the reference materials to be used, the calibration technique, the acceptable performance limits, the frequency, and the documentation.

Equipment Identification

Equipment that is subject to calibration will be labeled with a unique number.

Calibration Frequency

Frequency will be determined by manufacturers' recommendations, agency requirements, equipment type, instrument stability, method type, and prior experience.

Calibration Reference Standards

Physical standards (weights, certified thermometers) will be traceable to nationally recognized standards (*e.g.*, National Institute of Standards and

Technology [NIST]), which are at least four to ten times as accurate as the equipment requirements. Chemical reference standards will be NIST Standard Reference Materials (SRMs), standards provided by the USEPA, or vendor-certified materials that are traceable to these standards.

Calibration Failure

Equipment that fails calibration will be removed from service or tagged to indicate that it is out of calibration. The equipment will be repaired and recalibrated before reuse.

Calibration Records

Calibration records will be maintained for each piece of equipment that requires calibration. This information will include instrument name and number, calibration frequency and acceptance limits, date of calibration, calibration instructions, identity of person performing the calibration, calibration data, and records of any failures or repairs. Records for each instrument will be maintained in a separate folder. Group supervisors will maintain a calendar listing the dates of calibration for instruments that require periodic calibration in their laboratory.

For instruments that are calibrated on an operational basis, calibration generally consists of determining instrument response against compounds of known composition and concentration or the preparation of a standard response curve of the same compound at different concentrations.

6.2.2 Operational Calibration

Operational calibration usually involves measuring a standard response or preparing a standard calibration curve. Operational calibrations for the major pieces of equipment are the responsibility of the contract laboratory.

Section 7
Analytical Procedures

Groundwater, drinking water, surface water, storm water, and soil samples collected during field sampling activities at the TPC site, to be analyzed at a fixed laboratory, will be analyzed by:

TriMatrix Laboratories
5560 Corporate Exchange Ct., S.E.
Grand Rapids, Michigan 49512
616-975-4500
616-942-7463 Fax

Active soil gas and indoor air samples collected during field sampling activities at the TPC site will be analyzed by:

Pace Analytical Services (Minneapolis)
1700 Elm Street SE
Minneapolis, MN 55414
612-607-1700
912-607-6444 Fax

Passive soil gas samples collected during field sampling activities at the TPC site will be analyzed by:

Beacon Environmental Services
323 Williams Street
Suite D
Bel Air, MD 21014
410-838-8780

If workplan objectives require a rapid-turn-around time, a mobile laboratory may be used as an alternative to TriMatrix Laboratories for analysis of groundwater, surface water, storm water, or soil samples collected during field sampling activities at the TPC site. The mobile laboratory will be provided by:

Environmental Chemistry Consulting Services, Inc. (ECCS)
2525 Advance Road
Madison, WI 53718
608-221-8700
608-221-4889 Fax

The quality assurance (QA) manuals for TriMatrix Laboratories, Pace Analytical Laboratories, Beacon Environmental Services, and ECCS are included as Appendix B, and method specific standard operating procedures (SOPs) for each laboratory are provided in Appendix C.

7.1 Field Analytical Procedures

To ensure that the analytical data gathered in the field are both valid and unbiased, field samplers will be take the following steps:

- Become familiar with how each piece of equipment is used;
- Ensure that operating manuals will accompany each piece of equipment to the field;
- Carry out preventive maintenance programs;
- Take some spare components into the field in case of equipment failure or damage;
- Calibrate instruments on a daily basis;
- Document readings and calibrations; and
- Perform daily quality control (QC) checks of field notes.

The accuracy, sensitivity, and precision of the field analytical techniques for measuring water levels, temperature, specific conductivity, and pH are dependent upon the specifications for the instruments used, as well as on the QC techniques employed during their use.

7.2 Laboratory Analytical Procedures

7.2.1 List of Target Compounds and Detection Limits

Laboratory analytical methods for individual constituents will be consistent with those methods for which the laboratory is specifically approved. Analytical methods and sample preservation requirements are summarized in Table 5. Laboratory analyses for each sample utilize standard methods that are appropriate for the impacts expected. Typically SW-846 methods will be used. Method 8260B will be used for VOCs in water and soil samples. Method 8260C will be used for VOCs in passive soil gas samples. USEPA Method TO-15 will be used for VOCs in active soil gas and indoor air samples, and USEPA Method 524.2 will be used for drinking water samples. These analytical methods are subject to change as new EPA approved methods become available.

7.2.2 List of Associated Quality Control Samples

Table 6 summarizes the number and nature of QC sample requirements for the analysis of specific analyte groups. QC samples are also discussed in Sections 4.13 and 8.2 of this QAPP.

Section 8
Internal Quality Control Checks

8.1 Field Quality Control

Quality control (QC) procedures for pH, specific conductance, and temperature measurements are limited to checking the reproducibility of the measurement by obtaining multiple readings of a single sample or standard and by calibrating the instrument. The instruments are calibrated daily and checked periodically throughout the day in the calibration standards. If an instrument reading is off by 10 percent of the calibration standard, it is recalibrated. Instruments that fail to calibrate properly will be replaced.

The following quality control samples will be collected or prepared in the field for submittal to the analytical laboratories:

- Blind field duplicates
- Field equipment blanks
- Trip blanks.

Assessment of field sampling precision and bias will be made through the collection of field duplicates, equipment blanks, and trip blanks in accordance with the applicable procedures described in Section 4.13 of this QAPP. For soil gas samples, a tracer will also be used to help assess field accuracy.

8.2 Laboratory Measurements

The analytical laboratories use two types of quality assurance to ensure the production of analytical data of known and documented usable quality: program quality assurance and analytical method quality control.

The outside laboratories have written quality assurance/quality control (QA/QC) program, which provides rules and guidelines for ensuring the reliability and validity of the work conducted at the laboratory. Compliance with the QA/QC program is coordinated and monitored by the laboratory QA officer, who is independent of the operating departments.

Laboratory procedures are documented in writing, which are edited and controlled by the laboratory QA officer. Internal quality control procedures for analytical services will be conducted in accordance with standard operating procedures and the individual method requirements in a manner consistent with the appropriate analytical methods. These

specifications include the types of audits required (sample spikes, reference samples, controls, blanks, the specific calibration check standards, and laboratory duplicate analysis), the frequency of each audit, the compounds to be used for sample spikes, and the quality control acceptance criteria for these audits.

As described in the applicable laboratory QA Manual, the following internal QC checks may be conducted:

- An identification of all stages in the sampling and analytical process where the QC activity will occur
- Spikes
  - Matrix spikes
  - Laboratory control spikes (low-level organics and other parameters)
  - Surrogates and internal standards
- Standard reference materials
- Blanks (field, trip, instrument, storage, and method)
- Mass tuning for mass spectral analyses
- Confirmation with second column for gas chromatographic (GC) analyses
- Control charts
- Calibration standards
- Proficiency testing of analysts
- Sample duplicates.

The laboratory will document, in the data package provided, that both initial and ongoing instrument and analytical QC functions have been met. The laboratory will reanalyze any samples analyzed in nonconformance with the QC criteria, if it is deemed necessary, if sufficient sample volume is available, and if holding times allow for reanalysis. The quality control acceptance criteria and spike concentrations are specified in the analytical methods.

8.2.1 Laboratory Internal Quality Control Samples

The types of samples used as internal QC samples by the laboratories vary with each method. Relevant laboratory standard operating procedures (SOPs) are provided in Appendix C.

8.2.2 Method Blank Analyses

A method blank is a volume of deionized water or a sample of purified soil that is carried through the entire organic analytical procedure to verify that interference caused by contaminants in the solvents, reagents, glassware, etc. are known and minimized. A method blank will be analyzed with each group of samples. Ideally, a VOC method blank will be below the MDL for the compounds of interest, except for common laboratory solvents.

8.2.3 Duplicate Sample Analyses

Duplicate analyses will be used to calculate the precision (relative percent difference) of an analysis.

8.2.4 Check Standard Analyses (Calibration Verification)

Because standards and calibration curves are subject to change, a midpoint standard or check standard is frequently analyzed with each group of samples to verify the curve, and in some cases, to serve as the entire calibration. This value will be recorded in the instrument calibration log whenever it is performed.

8.2.5 Surrogate Standard Analyses

For GC/mass spectroscopy analysis, all samples and blanks will be fortified with surrogate spiking compounds before purging or extraction to monitor sample preparation and analysis. Recoveries will meet EPA acceptance criteria. At least one method blank will meet EPA criteria, or the samples will be re-extracted, or a nonconformance report will be filed.

8.2.6 Laboratory Matrix Spike and Matrix Duplicate Analyses

To evaluate the effect of the sample matrix upon analytical methodology, a separate aliquot sample will be spiked with the analyte of interest and will be analyzed with the sample. If the percent recovery falls outside established limits, the sample data will be carefully evaluated to determine what remedial action is required.

Section 9
Data Reduction, Validation and Reporting

9.1 Data reduction

All data generated through field activities, or by the laboratory operation shall be reduced and validated prior to reporting. The laboratory shall not disseminate data until it has been subjected to these procedures that are summarized in subsections below.

9.1.1 Field Data Reduction Procedures

Raw data from field measurements and sample collection activities will be appropriately recorded in the field notes. If the data are to be used in the project reports, they will be reduced for summarizing, and the method of reduction will be documented in the report. With the exception of the temperature correction for specific conductance, if necessary, and borehole volume calculations, no calculation will be involved in field data reduction. Only direct-reading instrumentation will be employed in the field. The RMT Technical Coordinator, or designee, will proofread all forms and notebooks to determine if the field crew has made any transcription errors.

9.1.2 Laboratory Data Reduction Procedures

The designated analytical laboratories will perform in-house analytical data reduction and review under the direction of the laboratory QA officer. Collectively the laboratory project manager and the laboratory QA officer will be responsible for assessing data quality and for advising of any data that were rated "preliminary" or "unacceptable" or that had other notations that would caution the data user of possible unreliability. Data reduction, validation, and reporting will be conducted as specified in the applicable Laboratory QA Manual (Appendix B):

- Raw data produced by the analyst is turned over to the respective area supervisor.
- The area supervisor reviews the data for attainment of QC criteria as outlined in SW-846 or other applicable protocols.
- Upon acceptance of the raw data by the area supervisor, a report is generated and sent to the laboratory project manager.
- The laboratory QA officer completes a thorough audit of reports at a frequency of 1 in 10.

- The QA officer and area supervisors decide whether any sample re-analysis is required.
- Final reports are generated and signed by the laboratory project manager. The laboratory package is presented in the same order in which the samples were analyzed.

The QA Manual specifies the data reduction reporting procedures.

9.2 Data Validation

9.2.1 Procedures Used to Validate Field Data

Procedures to validate field data will primarily include checking for transcription errors and reviewing field forms, on the part of field crewmembers. Review of field notes is the responsibility of the Technical Coordinator or designee. The data reviewer will review field notes and field Chain-of-Custody Records to determine if procedures have been followed.

9.2.2 Procedures Used to Validate Laboratory Data

RMT will perform data validation as required by the data quality objectives. Typically soil, groundwater, surface water, storm water, active soil gas and indoor air sample data will be validated, whereas passive soil gas sample data and select rapid turn-around field screening data will not typically be validated. The designated data validator(s) will conduct a review of the spike, duplicate, and blank results provided by the laboratory as well as verify that the sample holding times were met. Additional QC information set forth in the following bullets will be reviewed to check for appropriate matrix performance using the specified analytical methods.

The procedures used to evaluate data may include the following items:

- Checking technical holding times for inorganic and organic analyses.
- Reviewing data for blanks, surrogate spikes, matrix spikes/matrix spike duplicates (organics), laboratory duplicates (inorganic), and laboratory control samples.
- Reviewing internal standard areas and retention times (RT), as appropriate.
- Determining field precision from blind field duplicate data.
- Checking the completeness of the data package to determine if samples and analyses required by the QAPP were processed, if the procedures specified in the QAPP were implemented, and if deliverables specified in the QAPP are included.

- Identifying any questionable data and data omissions and interacting with the laboratory to correct data deficiencies. This is the responsibility of the data reviewer.
- Deciding whether to repeat sample collection and analyses based on the extent of the deficiencies and their importance in the overall context of the project. This is the responsibility of the RMT project manager.
- Assessing the usability of results against the data quality objectives (DQOs). .

The data validation report will address the following items:

- The usability of the data if QC results indicate that there are potential problems with all or some of the data.
- Potential sample contamination as a result of blank contributions.

9.3 Data Reporting

9.3.1 Field Data Reporting

Field data reporting will consist of field notes documenting sample collection and the sample Chain-of-Custody Records. Field instrument chemical analysis and associated QA/QC data will be tabulated and summarized.

9.3.2 Laboratory Data Reporting

The analytical laboratory will retain full analytical and QC documentation. Such retained documentation need not be hard (paper) copy, but may be in other storage media (*e.g.*, compact disk or network server).

The laboratory report will be appropriate to meet project data quality objectives. For example, if data requires validation, the laboratory will provide the following information with the analytical data package:

1. Cover sheets listing the samples included in the report and narrative comments describing problems encountered in analysis.
2. Tabulated results of inorganic and organic compounds identified and quantified.
3. Analytical results for QC sample spikes (including matrix spike/matrix spike duplicate [MS/MSD] samples), laboratory sample replicates, procedural/method blanks, laboratory control samples, surrogate compound recoveries, and internal standard areas and retention times. The laboratory will include percent recoveries, relative percent difference (RPD) results, and the acceptable range of QC results on the QC data summaries, where applicable.

4. Analytical method numbers, sample preparation (*e.g.*, extraction, digestion) dates, sample analysis dates, and practical quantitation limits (PQLs) for the specified analyses. Appropriate documentation to easily associate QC results with field samples will be provided by the laboratory.

5. Actions regarding specific QC criteria exceedences.

Section 10
Performance and System Audits

Performance audits consist of the observation of project activities in progress, for their compliance with the established quality control (QC) procedures and requirements. System audits consist of a review of the QC system to ensure that a comprehensive set of QC methods, procedures, review, and sign-off approvals are established or in place.

System audits will be performed on a periodic basis throughout the duration of the project. Audits of non-laboratory activities will be performed at project milestones (*e.g.*, completion of draft reports) and at any point deemed necessary by the RMT Quality Assurance (QA) Manager or the RMT Project Manager. The audits will include reviews of the QC procedures, implementation of those procedures, documentation, and implementation of corrective actions.

Performance and systems audits are conducted as part of the USEPA or other regulatory programs. External laboratory and field audits are the responsibility of the regulatory QA Manager.

10.1 Field Performance and System Audits

10.1.1 Internal Field Audits

Designated field staff will conduct periodic field audits. The audits will consist of a review of the following procedures:

- Field documentation procedures;
- Performance of field measurements of pH, specific conductivity, temperature, and water level measurements;
- Calibration and maintenance procedures for field instruments;
- The determination of the accuracy of field measurements through pre-measurement calibrations and post-measurement modifications;
- Purging, sampling, filtering, preservation, and Chain-of-Custody methods; and
- Sampling equipment cleaning procedures.

RMT's Field Coordinator or Technical Coordinator will oversee field audits. These audits will verify that established procedures are followed.

Designated RMT field staff will review field notes on a daily basis for completeness and clarity. Any deficiencies will be corrected or addressed prior to the next day's sampling events.

10.1.2 External Field Audits

The USEPA Project Manager may conduct external field audits at any time during the field operations. These audits may or may not be announced and are at the discretion of USEPA. External field audits will be conducted according to the field activity information presented in the QAPP. The external field audit process can include (but not be limited to): sampling equipment decontamination procedures, sample bottle preparation procedures, sampling procedures, examination of field sampling and safety plans, sample vessel cleanliness and QA procedures, procedures for verification of field duplicates, sample preservation and preparation for shipment, as well as field screening practices.

10.2 Laboratory Performance and System Audits

10.2.1 Internal Laboratory Audits

Audit procedures are specified in the laboratory's QA Manual. The audits consist of random data reviews, continuous trend analysis of laboratory QA data, and periodic analysis of performance evaluation samples. System audits are performed to verify the continuity of personnel, instrumentation, and quality control requirements contained in the QA Manual.

10.2.2 External Laboratory Audits

An external audit will be conducted as required, by appropriate QA staff of the Waste, Pesticides and Toxics Division, USEPA Region 5. An external audit may be conducted prior to the initiation of the sampling and analysis activities. These audits may or may not be announced and are at the discretion of the USEPA. External audits may include any or all of: review of laboratory analytical procedures, laboratory site visits, and/or submission of performance evaluation samples to the laboratory for analysis. Failure of any or all audit procedures chosen can lead to laboratory disqualification and the requirement that another suitable laboratory be chosen. An external on-site review can consist of: sample receipt procedures, custody and sample security and log in procedures, sample through-put tracking procedures, review of instrument calibration records, instrument logs and statistics (number and type), review of QA procedures, log books, sample prep procedures, sample analytical SOP review, instrument reviews,

personnel interviews, review of deadlines, and glassware prep. It is common practice when conducting an external laboratory audit to review one or more data packages from sample lots recently analyzed by the laboratory. This review will most likely include but not be limited to:

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

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

Section 11
Preventive Maintenance Procedures

11.1 Field Instrument Preventative Maintenance

Field preventive maintenance will include a cursory check of instrument operation without disassembly of test equipment. If any one of these preliminary checks is negative, the instrument may not be functioning properly and the backup meter will be procured and used. Critical spare parts, such as pH probes, tapes, and batteries, will be kept on-site to reduce downtime.

11.2 Laboratory Instrument Preventative Maintenance

As part of their quality assurance/quality control (QA/QC) Program, the laboratory conducts a routine preventive maintenance program to minimize the occurrence of instrument failure and other system malfunctions. All laboratory instruments are maintained in accordance with manufacturers' specifications. Major instrumentation is maintained under a maintenance agreement.

For the test instruments, the preventive maintenance procedures that are listed in the laboratory manual will be followed. All maintenance activities must be documented in logbooks to provide a history of maintenance records.

Section 12
Specific Routine Procedures to Assess Data Precision, Accuracy, and Completeness

The procedures used to assess data precision and accuracy will include a review of the laboratory's quality control (QC) data, and the results of the field blanks and the duplicates using the procedures outlined in USEPA guidance documents. The completeness of the sampling plan will be assessed against the data quality objectives of the QAPP after the analytical results have been received. Laboratory results will be assessed for compliance with required precision, accuracy, completeness, and sensitivity criteria as described below. In addition, the data will be reviewed for indications of interferences to results caused by sample matrices, cross contamination during sampling, cross contamination in the laboratory, and sample preservation and storage anomalies (*i.e.*, samples holding time or analytical instrument problems).

12.1 Accuracy Assessment

The accuracy of the laboratory results will be assessed for compliance with the established QC criteria that are described in this QAPP using the analytical results of method blanks, reagent/preparation blanks, matrix spike/matrix spike duplicate samples, field blanks, and bottle blanks. At a minimum one sample spike should be included in every set of 20 samples tested on each instrument, for each batch of soil of water samples to be analyzed. The percent recovery (%R) of matrix spike samples will be calculated using the following equation.

$$\%\,R = \frac{A - B}{C} \times 100\%,$$

where

A = the analyte concentration determined experimentally from the spiked sample,
B = the background level determined by a separate analysis of the unspiked sample,
C = the amount of the spike added.

12.2 Precision Assessment

The precision of the laboratory analysis will be assessed by comparing the analytical results between matrix spike/matrix spike duplicate (MS/MSD) and/or laboratory duplicate analyses.

The relative percent difference (%RPD) will be calculated for each pair of duplicate analysis using the following equation:

$$\% \text{ RPD} = \frac{S - D}{(S + D)/2} \times 100\%,$$

where

S = first sample value (original or MS value),
D = second sample value (duplicate or MSD value).

12.3 Completeness Assessment

The data completeness of laboratory analysis results will be assessed for compliance with the amount of data required for decision-making. The completeness is calculated using the equation below:

$$\text{Completeness} = \frac{\text{Valid Data Obtained}}{\text{Total Data Planned}} \times 100\%$$

12.4 Sensitivity Assessment

The achievement of method detection limits depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to ensure data quality through constant instrument performance. The instrument sensitivity will be monitored through the analysis of method blanks, calibration check samples, and laboratory control samples, *etc.*, in accordance with SW-846 methodology.

12.5 Assessment of Data

The field and laboratory data collected during this investigation will be used to evaluate the nature and extent of contamination at the site. The QC results associated with each analytical parameter for each matrix will be compared to the objectives described in Section 8 of this QAPP. Only data generated in association with QC results meeting these objectives will be considered useable for decision-making purposes. In addition, the data obtained will be both

qualitatively and quantitatively assessed on a project wide, matrix-specific, parameter-specific and unit-specific basis. The RMT QA Manager or designee will perform this assessment, and the results will be presented and discussed in detail in the final investigation report. Factors to be considered in this assessment of field and laboratory data will include, but not necessarily be limited to, the following:

- Were all samples obtained using the methodologies and SOPs proposed in the QAPP?
- Were all proposed analyses performed according to the SOPs provided in the QAPP?
- Were samples obtained from all proposed sampling locations and depths?
- Do any analytical results exhibit elevated detection limits due to matrix interferences or contaminants present at high concentrations?
- Were any analytes not expected to be present at the facility, or a given unit, identified as either target parameters or Tentatively Identified Compounds (TICs)?
- Were all field and laboratory data validated according to the validation protocols, including project-specific QC objectives, proposed in the QAPP?
- Which data sets were found to be unusable (qualified as "R") based on the data validation results?
- Which data sets were found to be usable for limited purposes (qualified as "J") based on the data validation results?
- What affect do qualifiers applied as a result of data validation have on the ability to implement the project decision rules?
- Has sufficient data of appropriate quality been generated to support presumptive corrective measures (CM), risk assessment, or other project activities?
- If applicable, were risk assessments conducted properly?
- Can valid conclusions be drawn for all matrices at each unit and/or area under investigation?

Were all issues requiring corrective action, as presented in the quarterly progress reports to management fully resolved?

Section 13
Corrective Action

Corrective actions may be required for two classes of problems: analytical and equipment problems and noncompliance problems.

Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrument analysis, and data review. If the problem is analytical in nature, and directly impacts project data quality, information on these problems will be promptly communicated, and implementation of corrective action will be confirmed in writing through the same channels.

For noncompliance problems, a formal corrective action program will be developed and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the appropriate field or laboratory personnel or the RMT Project Manager.

Any nonconformance with the established quality control (QC) procedures in the QAPP will be identified and corrected in accordance with the QAPP.

Corrective actions will be implemented and documented in the field notes. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by a stop work order by the RMT Project Manager.

13.1 Field Corrective Action

Technical staff and project personnel will be responsible for reporting suspected technical or QC nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Technical Coordinator or designee. The Technical Coordinator, or designee, will be responsible for assessing the suspected problems in consultation with RMT Project Manager and/or the RMT QA Manager. The Technical Coordinator is also responsible for making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the Project Manager.

The Project Manager, or designee, will be responsible for ensuring that corrective action for nonconformances are initiated by the following:

- Evaluating all reported nonconformances;
- Controlling additional work on nonconforming items;
- Determining disposition or action to be taken;
- Maintaining a log of nonconformances;
- Reviewing nonconformance reports and corrective actions taken; and
- Ensuring that nonconformance reports are included in the final facility documentation in project files.

If appropriate, the Technical Coordinator will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed.

Corrective action for field measurements may include the following:

- Repeating the measurement to check the error;
- Checking for all proper adjustments for ambient conditions such as temperature;
- Checking the batteries;
- Checking the calibration;
- Replacing the instrument or measurement devices; and/or
- Stopping work (if necessary).

13.2 Laboratory Corrective Action

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. Investigative actions taken are somewhat dependent on the analysis and on the event.

Laboratory personnel are alerted that corrective actions may be necessary if any of the following occurs:

- QC data are outside the warning or acceptable windows for precision and accuracy.
- Blanks contain target analytes above acceptable levels.
- Undesirable trends are detected in spike recoveries or in the relative percent difference (RPD) between duplicates.
- Unusual changes are noted in detection limits.
- Deficiencies are detected by the QA Department during internal or external audits or from the results of performance evaluation samples.
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst who reviews the preparation or extraction procedure for possible errors and checks the instrument calibration, the spike and calibration mixes, the instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager, and/or QA Department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA Department.

13.3 Corrective Action During Data Validation and Data Assessment

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory. These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required QA objectives (*e.g.*, the holding time for samples is not exceeded), etc. If the data reviewer or QA Manager identifies a corrective action situation, the RMT Project Manager is responsible for approving the implementation of corrective action, including resampling.

Section 14
Quality Assurance Reporting

Reports, technical memorandums, and other deliverables which present new data will include, in a separate section, a discussion of data quality. This section will include relevant data quality information collected during the collection of any new data presented. This quality assurance (QA) reporting will be the responsibility of the RMT Project Manager and will include the QA manager or designee's report on the accuracy, precision, and completeness of the data, as well as the results of the performance and system audits, and any corrective action needed or taken during the project.

14.1 Contents of Project Quality Assurance Report Sections

The QA report section will contain on a routine basis, results of field and laboratory audits, information generated during data collection reflecting on the achievement of specific DQOs, and a summary of corrective action that was implemented, and its immediate results on the project. Whenever necessary, updates on training provided, changes in key personnel, anticipated problems in the field or laboratory for the coming work that could bear on data quality along with proposed solutions, will be reported. Detailed references to QAPP modifications will also be highlighted. All QA report sections will be prepared in written, final format by the RMT project manager or his designee. To the extent possible, assessment of the project should also be performed on the basis of available QC data and overall results in relation to originally targeted objectives. In the event of an emergency, or in case it is essential to implement corrective action immediately, QA reports can be made by telephone to the appropriate individuals, as identified in the Project Organization and Corrective Action sections of this QAPP. However, these events, and their resolution will be addressed thoroughly in the appropriate QA report section.

14.2 Frequency of Quality Assurance Reporting

A QA Report Section will be prepared for each portion of the work conducted under a separate workplan. Reports, technical memorandums, and other deliverables which present new data will be submitted to USEPA in a timely manner, typically as part of the next quarterly progress report submitted in accordance with the consent order. If work related to a single workplan continues over several months, quarterly progress reports may include a discussion of ongoing QA activities. At a minimum each quarterly progress report will summarize any problems (including QA problems) encountered during the reporting period and any actions taken to rectify the identified problems.

14.3 Individuals Receiving / Reviewing Quality Assurance Report Sections

The following individuals will routinely receive copies of the quarterly progress reports and other deliverables which include QA sections.

- Graham Crockford - RMT Project Manager
- Michelle Mullin – USEPA Region 5 Project Manager
- Jason Smith – TPC Corporate Environmental Director
- Lynn Dennison - TPC Counsel
- Douglas McClure – Conlin, McKenney & Philbrick, PC (TPC outside counsel)
- Stacy Metz - RMT Technical Coordinator
- Thomas Stolzenburg – RMT QA Manager

Tables

Table 1
Summary of Sceening Levels and Laboratory Detection Limits for Project Target Compounds in Soils
Former Tecumseh Products Company Site
Tecumseh, Michigan

Analyte	CAS Number	Units	Analytical Method	Laboratory Reporting Limit[1,2]	Laboratory MDL[1,3]	Soil Screening Levels[4]			
						Drinking Water Protection	Groundwater Surface Water Interface	Residential Direct Contact	Industrial Direct Contact
Acetone	67-64-1	µg/kg	5035/8260B	1000	94	15,000	34,000	2.30E+07	7.30E+07
Acrylonitrile	107-13-1	µg/kg	5035/8260B	100	22	100	100	16,000	74,000
Benzene	71-43-2	µg/kg	5035/8260B	50	8.8	100	4,000	1.80E+05	4.00E+05
Bromobenzene	108-86-1	µg/kg	5035/8260B	100	9.6	550	NA	5.40E+05	7.60E+05
Bromochloromethane	74-97-5	µg/kg	5035/8260B	100	6.6	NC	NC	NC	NC
Bromodichloromethane	75-27-4	µg/kg	5035/8260B	100	6.6	1,600	NC	1.10E+05	4.90E+05
Bromoform[5]	75-25-2	µg/kg	5035/8260B	100	7.2	1,600	NC	8.20E+05	8.70E+05
Bromomethane[5]	74-83-9	µg/kg	5035/8260B	200	8.9	200	700	3.20E+05	1.00E+06
2-Butanone (MEK)	78-93-3	µg/kg	5035/8260B	750	28	2.60E+05	44,000	2.70E+07	2.70E+07
n-Butylbenzene	104-51-8	µg/kg	5035/8260B	50	9.1	1,600	NC	2.50E+06	8.00E+06
sec-Butylbenzene	135-98-8	µg/kg	5035/8260B	50	8.8	1,600	NC	2.50E+06	8.00E+06
tert-Butylbenzene	98-06-6	µg/kg	5035/8260B	50	7.7	1,600	NC	2.50E+06	8.00E+06
Carbon Disulfide	75-15-0	µg/kg	5035/8260B	250	11	16,000	NC	2.80E+05	2.80E+05
Carbon Tetrachloride	56-23-5	µg/kg	5035/8260B	50	6.5	100	900	96,000	3.90E+05
Chlorobenzene	108-90-7	µg/kg	5035/8260B	50	9.4	2,000	940	2.60E+05	2.60E+05
Chloroethane	75-00-3	µg/kg	5035/8260B	250	8.4	8,600	NC	9.50E+05	9.50E+05
Chloroform[5]	67-66-3	µg/kg	5035/8260B	50	8.4	1,600	3,400	1.20E+06	1.50E+06
Chloromethane	74-87-3	µg/kg	5035/8260B	250	22	5,200	NC	1.10E+06	1.10E+06
Dibromochloromethane (Ethylene Dibromide)[5]	124-48-1	µg/kg	5035/8260B	100	6.9	1,600	NC	1.10E+05	5.00E+05
1,2-Dibromo-3-chloropropane[6]	96-12-8	µg/kg	5035/8260B	50	30	10	NC	1,200	1,200
1,2-Dibromoethane[6]	106-93-4	µg/kg	5035/8260B	50	11	20	20	92	430
Dibromomethane	74-95-3	µg/kg	5035/8260B	250	11	1,600	NC	2.00E+06	2.00E+06
1,2-Dichlorobenzene	95-50-1	µg/kg	5035/8260B	100	6.2	14,000	360	2.10E+05	2.10E+05
1,3-Dichlorobenzene	541-73-1	µg/kg	5035/8260B	100	6.3	170	1,100	1.70E+05	1.70E+05
1,4-Dichlorobenzene	106-46-7	µg/kg	5035/8260B	100	6.0	1,700	290	4.00E+05	1.90E+06
trans-1,4-Dichloro-2-butene	110-57-6	µg/kg	5035/8260B	50	9.4	NC	NC	NC	NC
Dichlorodifluoromethane	75-71-8	µg/kg	5035/8260B	250	22	95,000	NC	1.00E+06	1.00E+06
1,1-Dichloroethane	75-34-3	µg/kg	5035/8260B	50	6.6	18,000	15,000	8.90E+05	8.90E+05
1,2-Dichloroethane	107-06-2	µg/kg	5035/8260B	50	6.3	100	7,200	91,000	4.20E+05
1,1-Dichloroethene	75-35-4	µg/kg	5035/8260B	50	7.1	140	1,300	2.00E+05	5.70E+05
cis-1,2-Dichloroethene	156-59-2	µg/kg	5035/8260B	50	12	1,400	12,000	6.40E+05	6.40E+05
trans-1,2-Dichloroethene	156-60-5	µg/kg	5035/8260B	50	10	2,000	30,000	1.40E+06	1.40E+06

Notes:

1. Reporting limits and method detection limits (MDLs) presented in this table are from Trimatrix Laboratories. The reporting limits and MDLs for ECCS, the designated mobile laboratory, are similar.
2. The actual reporting limit is calculated in dry weight as per SW-846 methods, the reporting limits will be slightly higher depending on the percent moisture in the sample.
3. MDLs listed are current as of July 2010. MDLs are updated annually in accordance with the Laboratory Quality Assurance Manual and are subject to change.
4. Drinking Water Protection Criteria, Groundwater Surface Water Interface Protection Criteria, Residential Direct Contact Criteria, and Industrial Direct Contact Criteria are taken from MDEQ RRD Op Memo 1 Part 201 Generic Cleanup Criteria/Part 213 Risk Based Cleanup Levels, January 23, 2006.
5. Concentrations of trihalomethanes (bromodichlormethane, bromoform, chloroform, and dibromochloromethane) shall be added together prior to comparison to the applicable soil screening level (1,600 ug/kg).
6. One or more screening levels are below the laboratory reporting limit. 1,2-dibromo-3-chloropropane and 1,2-dibromoethane are not constituents of concern at the site; therefore the screening level will default to the reporting limit.

µg/kg = micrograms per kilogram
NC = no criteria or screening level

Table 1
Summary of Sceening Levels and Laboratory Detection Limits for Project Target Compounds in Soils
Former Tecumseh Products Company Site
Tecumseh, Michigan

Analyte	CAS Number	Units	Analytical Method	Laboratory Reporting Limit[1,2]	Laboratory MDL[1,3]	Soil Screening Levels[4]			
						Drinking Water Protection	Groundwater Surface Water Interface	Residential Direct Contact	Industrial Direct Contact
1,2-Dichloropropane	78-87-5	µg/kg	5035/8260B	50	7.3	100	5,800	1.40E+05	5.50E+05
cis-1,3-Dichloropropene	10061-01-5	µg/kg	5035/8260B	50	13	170	NC	10,000	2.40E+05
trans-1,3-Dichloropropene	10061-02-6	µg/kg	5035/8260B	50	12	170	NC	10,000	2.40E+05
Ethylbenzene	100-41-4	µg/kg	5035/8260B	50	7.2	1,500	360	1.40E+05	1.40E+05
Ethyl Ether (Diethyl Ether)	60-29-7	µg/kg	5035/8260B	200	9.2	200	NC	7.40E+06	7.40E+06
2-Hexanone	591-78-6	µg/kg	5035/8260B	2500	15	20,000	NC	2.50E+06	2.50E+06
Iodomethane	74-88-4	µg/kg	5035/8260B	100	17	NC	NC	NC	NC
Isopropylbenzene	98-82-8	µg/kg	5035/8260B	250	7.6	91,000	NC	3.90E+05	3.90E+05
4-Isopropyltoluene	99-87-6	µg/kg	5035/8260B	100	14	NC	NC	NC	NC
4-Methyl-2-pentanone (MIBK)	108-10-1	µg/kg	5035/8260B	2500	14	36,000	NC	2.70E+06	2.70E+06
Methyl tert-Butyl Ether	1634-04-4	µg/kg	5035/8260B	250	14	800	15,000	1.50E+06	5.90E+06
Methylene Chloride	75-09-2	µg/kg	5035/8260B	100	13	100	19,000	1.30E+06	2.30E+06
2-Methylnaphthalene	91-57-6	µg/kg	5035/8260B	330	12	57,000	NC	8.10E+06	2.60E+07
Naphthalene	91-20-3	µg/kg	5035/8260B	330	18	35,000	870	1.60E+07	5.20E+07
n-Propylbenzene	103-65-1	µg/kg	5035/8260B	100	7.5	1,600	NC	2.50E+06	8.00E+06
Styrene	100-42-5	µg/kg	5035/8260B	50	5.6	2,700	2,200	4.00E+05	5.20E+05
1,1,1,2-Tetrachloroethane	630-20-6	µg/kg	5035/8260B	100	7.9	1,500	NC	4.40E+05	4.40E+05
1,1,2,2-Tetrachloroethane	79-34-5	µg/kg	5035/8260B	50	18	170	1,600	53,000	2.40E+05
Tetrachloroethene	127-18-4	µg/kg	5035/8260B	50	7.8	100	900	88,000	88,000
Tetrahydrofuran	109-99-9	µg/kg	5035/8260B	1000	32	1,900	2.20E+05	2.90E+06	9.50E+06
Toluene	108-88-3	µg/kg	5035/8260B	100	7.9	16,000	2,800	2.50E+05	2.50E+05
1,2,3-Trichlorobenzene	87-61-6	µg/kg	5035/8260B	330	9.1	NC	NC	NC	NC
1,2,4-Trichlorobenzene	120-82-1	µg/kg	5035/8260B	330	8.1	4,200	1,800	9.90E+05	1.10E+06
1,1,1-Trichloroethane	71-55-6	µg/kg	5035/8260B	50	9.7	4,000	4,000	4.60E+05	4.60E+05
1,1,2-Trichloroethane	79-00-5	µg/kg	5035/8260B	50	9.2	100	6,600	1.80E+05	8.40E+05
Trichloroethene	79-01-6	µg/kg	5035/8260B	50	15	100	4,000	5.00E+05	5.00E+05
Trichlorofluoromethane	75-69-4	µg/kg	5035/8260B	100	9.9	52,000	NC	5.60E+05	5.60E+05
1,2,3-Trichloropropane	96-18-4	µg/kg	5035/8260B	100	14	840	NC	8.30E+05	8.30E+05
1,2,4-Trimethylbenzene	95-63-6	µg/kg	5035/8260B	100	8.3	2,100	570	1.10E+05	1.10E+05
1,3,5-Trimethylbenzene	108-67-8	µg/kg	5035/8260B	100	9.1	1,800	1,100	94,000	94,000
Vinyl Chloride	75-01-4	µg/kg	5035/8260B	40	9.2	40	300	3,800	34,000
Xylenes	136777-61-2, 95-47-6	µg/kg	5035/8260B	150	21	5,600	700	1.50E+05	1.50E+05

Notes:

1. Reporting limits and method detection limits (MDLs) presented in this table are from Trimatrix Laboratories. The reporting limits and MDLs for ECCS, the designated mobile laboratory, are similar.
2. The actual reporting limit is calculated in dry weight as per SW-846 methods, the reporting limits will be slightly higher depending on the percent moisture in the sample.
3. MDLs listed are current as of July 2010. MDLs are updated annually in accordance with the Laboratory Quality Assurance Manual and are subject to change.
4. Drinking Water Protection Criteria, Groundwater Surface Water Interface Protection Criteria, Residential Direct Contact Criteria, and Industrial Direct Contact Criteria are taken from MDEQ RRD Op Memo 1 Part 201 Generic Cleanup Criteria/Part 213 Risk Based Cleanup Levels, January 23, 2006.
5. Concentrations of trihalomethanes (bromodichlormethane, bromoform, chloroform, and dibromochloromethane) shall be added together prior to comparison to the applicable soil screening level (1,600 ug/kg).
6. One or more screening levels are below the laboratory reporting limit. 1,2-dibromo-3-chloropropane and 1,2-dibromoethane are not constituents of concern at the site; therefore the screening level will default to the reporting limit.

µg/kg = micrograms per kilogram
NC = no criteria or screening level

Table 2
Summary of Screening Levels and Laboratory Detection Limits for Project Target Compounds in Groundwater
Former Tecumseh Products Company Site
Tecumseh, Michigan

Analyte	CAS Number	Units	Analytical Method	Laboratory Reporting Limit[1,2]	Laboratory MDL[1,3]	Groundwater Screening Levels			
						Drinking Water[4]	Groundwater Surface Water Interface[4]	Residential Vapor Intrusion[5]	Non-Residential Vapor Intrusion[5]
Acetone	67-64-1	µg/L	8260B	20	2.1	730	1,700	NC	NC
Acrylonitrile	107-13-1	µg/L	8260B	2.0	0.25	2.6	4.9	NC	NC
Benzene	71-43-2	µg/L	8260B	1.0	0.18	5.0	200	NC	NC
Bromobenzene	108-86-1	µg/L	8260B	1.0	0.19	18	NC	NC	NC
Bromochloromethane	74-97-5	µg/L	8260B	1.0	0.18	NC	NC	NC	NC
Bromodichloromethane[6]	75-27-4	µg/L	8260B	1.0	0.13	80	NC	NC	NC
Bromoform[6]	75-25-2	µg/L	8260B	1.0	0.15	80	NC	NC	NC
Bromomethane	74-83-9	µg/L	8260B	5.0	0.18	10	35	NC	NC
2-Butanone (MEK)	78-93-3	µg/L	8260B	5.0	0.55	13,000	2,200	4.60E+06	6.40E+06
n-Butylbenzene	104-51-8	µg/L	8260B	1.0	0.18	80	NC	NC	NC
sec-Butylbenzene	135-98-8	µg/L	8260B	1.0	0.18	80	NC	NC	NC
tert-Butylbenzene	98-06-6	µg/L	8260B	1.0	0.15	80	NC	NC	NC
Carbon Disulfide	75-15-0	µg/L	8260B	1.0	0.18	800	NC	NC	NC
Carbon Tetrachloride	56-23-5	µg/L	8260B	1.0	0.13	5.0	45	NC	NC
Chlorobenzene	108-90-7	µg/L	8260B	1.0	0.19	100	47	NC	NC
Chloroethane	75-00-3	µg/L	8260B	5.0	0.15	430	NC	NC	NC
Chloroform[6]	67-66-3	µg/L	8260B	1.0	0.17	80	170	NC	NC
Chloromethane	74-87-3	µg/L	8260B	5.0	0.16	260	NC	NC	NC
Dibromochloromethane (Ethylene Dibromide)[6]	124-48-1	µg/L	8260B	1.0	0.14	80	NC	NC	NC
1,2-Dibromo-3-chloropropane[7]	96-12-8	µg/L	8260B	5.0	0.40	0.20	NC	NC	NC
1,2-Dibromoethane[7]	106-93-4	µg/L	8260B	1.0	0.22	0.05	0.2	NC	NC
Dibromomethane	74-95-3	µg/L	8260B	1.0	0.23	80	NC	NC	NC
1,2-Dichlorobenzene	95-50-1	µg/L	8260B	1.0	0.12	600	16	NC	NC
1,3-Dichlorobenzene	541-73-1	µg/L	8260B	1.0	0.13	6.6	38	NC	NC
1,4-Dichlorobenzene	106-46-7	µg/L	8260B	1.0	0.12	75	13	NC	NC
trans-1,4-Dichloro-2-butene	110-57-6	µg/L	8260B	1.0	0.21	NC	NC	NC	NC
Dichlorodifluoromethane	75-71-8	µg/L	8260B	5.0	0.21	1,700	NC	NC	NC
1,1-Dichloroethane	75-34-3	µg/L	8260B	1.0	0.13	880	740	130	440
1,2-Dichloroethane	107-06-2	µg/L	8260B	1.0	0.13	5.0	360	NC	NC
1,1-Dichloroethene	75-35-4	µg/L	8260B	1.0	0.13	7.0	65	390	550
cis-1,2-Dichloroethene	156-59-2	µg/L	8260B	1.0	0.23	70	620	440	610
trans-1,2-Dichloroethene	156-60-5	µg/L	8260B	1.0	0.20	100	1,500	330	460

Notes:

1. Reporting limits and method detection limits (MDLs) presented in this table are from Trimatrix Laboratories. The reporting limits and MDLs for ECCS, the designated mobile laboratory, are similar.
2. These are standard reporting limits for water, matrix interference if found in the sample, may result in elevated reporting limits.
3. MDLs listed are current as of July 2010. MDLs are updated annually in accordance with the Laboratory Quality Assurance Manual and are subject to change.
4. Drinking Water and Groundwater Surface Water Interface Screening Levels are taken from MDEQ RRD Op Memo 1 Part 201 Generic Cleanup Criteria/Part 213 Risk Based Cleanup Levels, January 23, 2006.
5. Generic Groudnwater Screening Levels for Vapor Intrusion (GWSLs) were calculated in accordance with the MDNRE Remediation and Redevelopment Division Program Redesign 2009 document titled "Background Document: Draft Proposed Vapor Intrusion Indoor Air Criteria (IAC), Soil Gas Criteria (SGC), and Groundwater Screening Levels (GW_{VI}SLs) for Vapor Intrusion," using both residential and non-residential exposure scenarios and the most recent chemical specific toxicity values accepted and/or published by the United States Environmental Protection Agency (USEPA). These GWSLs were approved by the USEPA in a letter dated August 24, 2010.
6. Concentrations of trihalomethanes (bromodichlormethane, bromoform, chloroform, and dibromochloromethane) shall be added together prior to comparison to the applicable human health preliminary remediation goal (80 ug/L).
7. One or more screening levels are below the laboratory reporting limit. 1,2-dibromo-3-chloropropane and 1,2-dibromoethane are not constituents of concern at the site; therefore the screening level will default to the reporting limit.

µg/L = micrograms per liter
NC = no criteria or screening level

Table 2
Summary of Screening Levels and Laboratory Detection Limits for Project Target Compounds in Groundwater
Former Tecumseh Products Company Site
Tecumseh, Michigan

Analyte	CAS Number	Units	Analytical Method	Laboratory Reporting Limit[1,2]	Laboratory MDL[1,3]	Groundwater Screening Levels			
						Drinking Water[4]	Groundwater Surface Water Interface[4]	Residential Vapor Intrusion[5]	Non-Residential Vapor Intrusion[5]
1,2-Dichloropropane	78-87-5	µg/L	8260B	1.0	0.15	5.0	290	NC	NC
cis-1,3-Dichloropropene	10061-01-5	µg/L	8260B	1.0	0.25	8.5	NC	NC	NC
trans-1,3-Dichloropropene	10061-02-6	µg/L	8260B	1.0	0.23	8.5	NC	NC	NC
Ethylbenzene	100-41-4	µg/L	8260B	1.0	0.14	700	18	NC	NC
Ethyl Ether (Diethyl Ether)	60-29-7	µg/L	8260B	5.0	0.18	3,700	NC	NC	NC
2-Hexanone	591-78-6	µg/L	8260B	5.0	0.29	1,000	NC	NC	NC
Iodomethane	74-88-4	µg/L	8260B	1.0	0.21	NC	NC	NC	NC
Isopropylbenzene	98-82-8	µg/L	8260B	1.0	0.15	800	NC	NC	NC
4-Isopropyltoluene	99-87-6	µg/L	8260B	5.0	0.29	NC	NC	NC	NC
4-Methyl-2-pentanone (MIBK)	108-10-1	µg/L	8260B	5.0	0.28	1,800	NC	NC	NC
Methyl tert-Butyl Ether	1634-04-4	µg/L	8260B	5.0	0.28	240	730	NC	NC
Methylene Chloride	75-09-2	µg/L	8260B	5.0	0.26	5.0	940	NC	NC
2-Methylnaphthalene	91-57-6	µg/L	8260B	5.0	0.23	260	NC	NC	NC
Naphthalene	91-20-3	µg/L	8260B	5.0	0.37	520	13	NC	NC
n-Propylbenzene	103-65-1	µg/L	8260B	1.0	0.15	80	NC	NC	NC
Styrene	100-42-5	µg/L	8260B	1.0	0.11	100	80	NC	NC
1,1,1,2-Tetrachloroethane	630-20-6	µg/L	8260B	1.0	0.16	77	NC	NC	NC
1,1,2,2-Tetrachloroethane	79-34-5	µg/L	8260B	1.0	0.070	8.5	78	NC	NC
Tetrachloroethene	127-18-4	µg/L	8260B	1.0	0.16	5.0	45	11	37
Tetrahydrofuran	109-99-9	µg/L	8260B	5.0	1.0	95	11,000	NC	NC
Toluene	108-88-3	µg/L	8260B	1.0	0.16	1,000	140	NC	NC
1,2,3-Trichlorobenzene	87-61-6	µg/L	8260B	5.0	0.18	NC	NC	NC	NC
1,2,4-Trichlorobenzene	120-82-1	µg/L	8260B	5.0	0.16	70	30	NC	NC
1,1,1-Trichloroethane	71-55-6	µg/L	8260B	1.0	0.19	200	200	15,000	21,000
1,1,2-Trichloroethane	79-00-5	µg/L	8260B	1.0	0.18	5.0	330	NC	NC
Trichloroethene	79-01-6	µg/L	8260B	1.0	0.092	5.0	200	58	190
Trichlorofluoromethane	75-69-4	µg/L	8260B	1.0	0.20	2,600	NC	370	510
1,2,3-Trichloropropane	96-18-4	µg/L	8260B	1.0	0.28	42	NC	NC	NC
1,2,4-Trimethylbenzene	95-63-6	µg/L	8260B	1.0	0.16	1,000	17	NC	NC
1,3,5-Trimethylbenzene	108-67-8	µg/L	8260B	1.0	0.18	1,000	45	NC	NC
Vinyl Chloride	75-01-4	µg/L	8260B	1.0	0.10	2.0	15	5.0	17
Xylenes	136777-61-2, 95-47-6	µg/L	8260B	3.0	0.42	10,000	35	NC	NC

Notes:

1. Reporting limits and method detection limits (MDLs) presented in this table are from Trimatrix Laboratories. The reporting limits and MDLs for ECCS, the designated mobile laboratory, are similar.
2. These are standard reporting limits for water, matrix interference if found in the sample, may result in elevated reporting limits.
3. MDLs listed are current as of July 2010. MDLs are updated annually in accordance with the Laboratory Quality Assurance Manual and are subject to change.
4. Drinking Water and Groundwater Surface Water Interface Screening Levels are taken from MDEQ RRD Op Memo 1 Part 201 Generic Cleanup Criteria/Part 213 Risk Based Cleanup Levels, January 23, 2006.
5. Generic Groudnwater Screening Levels for Vapor Intrusion (GWSLs) were calculated in accordance with the MDNRE Remediation and Redevelopment Division Program Redesign 2009 document titled "Background Document: Draft Proposed Vapor Intrusion Indoor Air Criteria (IAC), Soil Gas Criteria (SGC), and Groundwater Screening Levels (GW_{VI}SLs) for Vapor Intrusion," using both residential and non-residential exposure scenarios and the most recent chemical specific toxicity values accepted and/or published by the United States Environmental Protection Agency (USEPA). These GWSLs were approved by the USEPA in a letter dated August 24, 2010.
6. Concentrations of trihalomethanes (bromodichlormethane, bromoform, chloroform, and dibromochloromethane) shall be added together prior to comparison to the applicable human health preliminary remediation goal (80 ug/L).
7. One or more screening levels are below the laboratory reporting limit. 1,2-dibromo-3-chloropropane and 1,2-dibromoethane are not constituents of concern at the site; therefore the screening level will default to the reporting limit.

µg/L = micrograms per liter
NC = no criteria or screening level

Table 3

Summary of Screening Levels and Laboratory Detection Limits for Project Target Compounds in Soil Gas

Former Tecumseh Products Company Site

Tecumseh, Michigan

Analyte	Units	Analytical Method	Laboratory Reporting Limit[1]	Laboratory MDL[2]	Screening Level: Sub-Slab[3]		Screening Level: DEEP[4]	
					Residential	Industrial	Residential	Industrial
1,1-Dichloroethane	ppbv	TO-15	0.2	0.032	190	640	1,300	4,300
1,2-Dichloroethane	ppbv	TO-15	0.2	0.027	12	39	78	260
1,1-Dichloroethene	ppbv	TO-15	0.2	0.046	2,600	3,600	17,000	24,000
cis-1,2-Dichloroethene	ppbv	TO-15	0.2	0.028	460	640	3,000	4,300
trans-1,2-Dichloroethene	ppbv	TO-15	0.2	0.074	780	1,100	5,200	7,300
Tetrachloroethene	ppbv	TO-15	0.2	0.10	31	100	210	690
1,1,1-Trichloroethane	ppbv	TO-15	0.2	0.025	47,000	66,000	310,000	440,000
Trichloroethene	ppbv	TO-15	0.2	0.026	120	390	770	2,600
Vinyl Chloride	ppbv	TO-15	0.2	0.093	54	180	360	1,200

Notes:

1. These are standard reporting limits for air, matrix interference if found in the sample, may result in elevated reporting limits.
2. Method detection limits (MDLs) listed are current as of July 2010. MDLs are updated annually in accordance with the Laboratory Quality Assurance Manual and are subject to change.
3. Sub-slab soil gas screening levels (SGSLs) were calculated according to the methods described in the MDNRE document titled "Background Document: Draft Proposed Vapor Intrusion Indoor Air Criteria (IAC), Soil Gas Criteria (SGC), and Groundwater Screening Levels (GWVISLs)." The most recent chemical specific toxicity values accepted and/or published by the USEPA were used in the calculations, and in accordance with USEPA comments received April 8, 2010 SGSL calculations were revised such that the exposure frequency is 250 days per year and exposure duration is 25 years.
4. Site Specific DEEP soil gas screening levels (SGSLs) were calculated using an attenuation factor (0.003). This attenuation factor was determined using the USEPA Johnson and Ettinger Model calculation spreadsheet, Version 3.1. The site specific model used the spreadsheet default parameters conservatively assuming a sand substrate, a depth to foundation of 200 cm (basement), and a sample depth of 200 cm.

ppbv = parts per billion by volume

Table 4

Summary of Screening Levels and Laboratory Detection Limits for Project Target Compounds in Indoor Air

Former Tecumseh Products Company Site

Tecumseh, Michigan

Analyte	Units	Analytical Method	Laboratory Reporting Limit[1]	Laboratory MDL[2]	Screening Level[3]	
					Residential	Industrial
1,1-Dichloroethane	ppbv	TO-15	0.2	0.032	3.8	13
1,2-Dichloroethane	ppbv	TO-15	0.2	0.027	0.23	0.79
1,1-Dichloroethene	ppbv	TO-15	0.2	0.046	52	73
cis-1,2-Dichloroethene	ppbv	TO-15	0.2	0.028	9.1	13
trans-1,2-Dichloroethene	ppbv	TO-15	0.2	0.074	16	22
Tetrachloroethene	ppbv	TO-15	0.2	0.10	0.62	2.1
1,1,1-Trichloroethane	ppbv	TO-15	0.2	0.025	940	1,300
Trichloroethene	ppbv	TO-15	0.2	0.026	2.3	7.8
Vinyl Chloride	ppbv	TO-15	0.2	0.093	1.1	3.6

Notes:

1. These are standard reporting limits for air, matrix interference if found in the sample, may result in elevated reporting limits.
2. Method detection limits (MDLs) listed are current as of July 2010. MDLs are updated annually in accordance with the Laboratory Quality Assurance Manual and are subject to change.
3. Indoor air screening levels were calculated according to the methods described in the MDNRE document titled "Background Document: Draft Proposed Vapor Intrusion Indoor Air Criteria (IAC), Soil Gas Criteria (SGC), and Groundwater Screening Levels (GWVISLs)." The most recent chemical specific toxicity values accepted and/or published by the USEPA were used in the calculations, and in accordance with USEPA comments received April 8, 2010 SGSL calculations were revised such that the exposure frequency is 250 days per year and exposure duration is 25 years.

ppbv = parts per billion by volume

Table 5
Summary of Sample Containers and Analytical Methods
Former Tecumseh Products Company Site
Tecumseh, Michigan

Sample Matrix	Analytes	Analytical Method	Sample Container	Sample Preservative	Hold Time	Fixed Laboratory	Mobile Laboratory
Soil / Sediment	Volatile Organic Compounds	5035/8260B	O2SI or Lock-in-Load Sample Kit	Methanol, cool to 4° C	14 days	TriMatrix	ECCS
			En Core® Sampler,	cool to 4° C	14 days	TriMatrix	Not Applicable
	Total Organic Carbon	MSA 29-3.5.2	4 oz glass jar	cool to 4° C	28 days	TriMatrix	Not Applicable
	pH	9045C	glass jar	cool to 4° C	7 days	TriMatrix	ECCS
Groundwater / Surface Water / Storm Water	Volatile Organic Compounds	8260B	(3) 40 mL VOA	HCl, cool to 4° C	14 days	TriMatrix	ECCS
	Ferrous Iron	SM 3500 Fe	250 mL plastic bottle	HCl, cool to 4° C	24 hours	TriMatrix	Not Applicable
	Nitrate / Nitrite	SM 4500 NO3, NO2	150 mL plastic bottle	cool to 4° C	48 hours	TriMatrix	Not Applicable
	Chloride / Sulfate	SM 4500 Cl, SO4	150 mL plastic bottle	cool to 4° C	28 days	TriMatrix	Not Applicable
	Alkalinity	SM 2320 B	150 mL plastic bottle	cool to 4° C	14 days	TriMatrix	Not Applicable
	Total Organic Carbon	SM 5310	(3) 40 mL VOA	H2SO4, cool to 4° C	28 days	TriMatrix	Not Applicable
Drinking Water	Volatile Organic Compounds	524.2	(3) 40 mL VOA	HCl, cool to 4° C	14 days	TriMatrix	Not Applicable
Acitive Soil Gas	Volatile Organic Compounds	TO-15	Summa Canister	ambient air temperature	28 days	Pace Analytical	Not Applicable
Air	Volatile Organic Compounds	TO-15	Summa Canister	ambient air temperature	28 days	Pace Analytical	Not Applicable
Passive Soil Gas	Volatile Organic Compounds	8260C	BeSure Kit	ambient air temperature	28 days	Beacon Environmental	Not Applicable

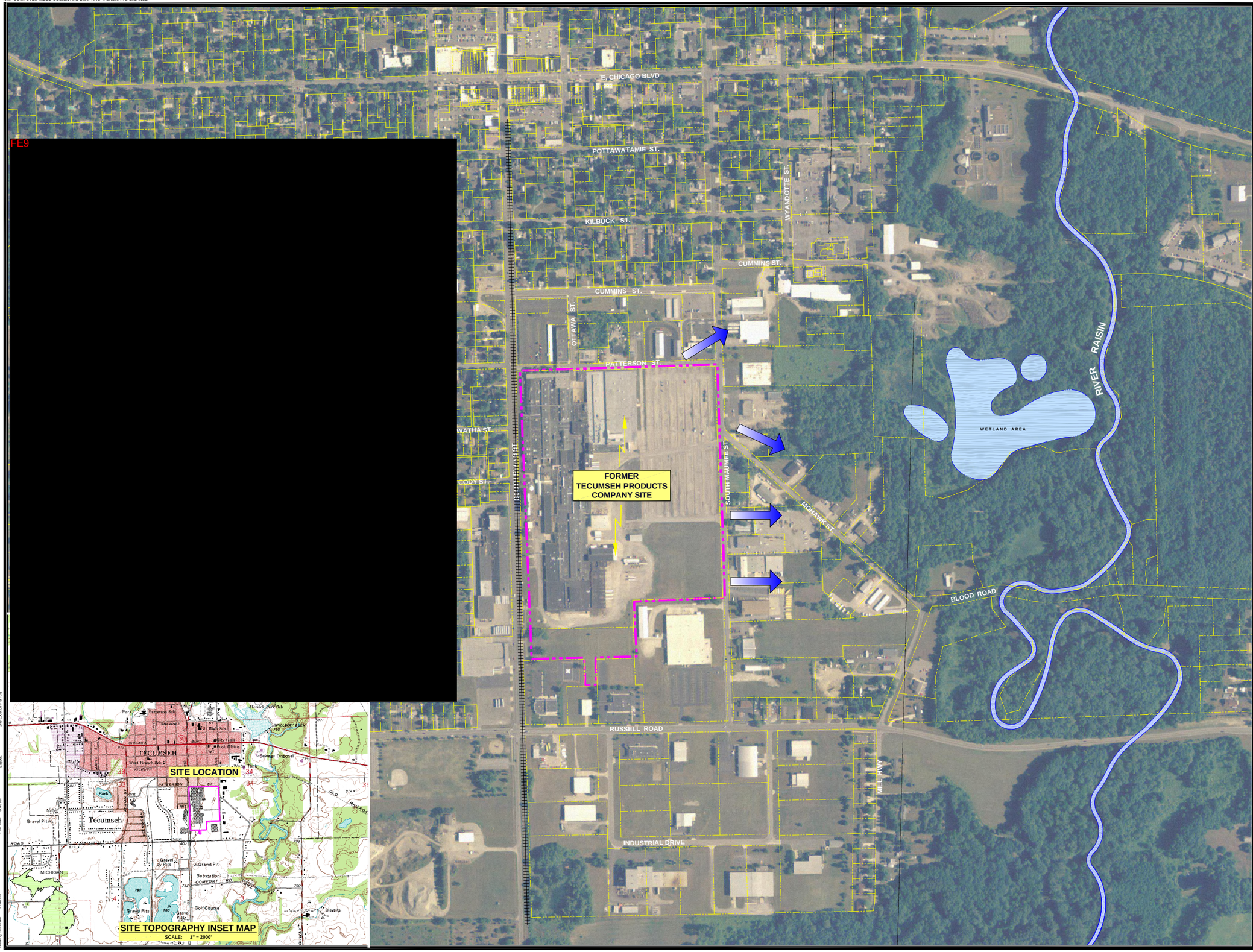
Table 6
Summary of Project Quality Control Sample Requirements
Former Tecumseh Products Company Site
Tecumseh, Michigan

Sample Matrix[1]	Field Duplicate		Field Blank		Trip Blank[2]		Matrix Spike		Matrix Spike Duplicate	
	QC	Primary	QC	Primary	QC	Primary	QC	Primary	QC	Primary
Soil / Sediment	1	20	NA	NA	1	NA	1	20	1	20
Groundwater / Surface Water / Storm Water	1	20	1	20	1	NA	1	20	1	20
Drinking Water	NA	NA	NA	NA	1	NA	NA	NA	NA	NA
Acitive Soil Gas	1	20	NA	NA	NA	NA	NA	NA	NA	NA
Air	1	20	NA	NA	NA	NA	NA	NA	NA	NA
Passive Soil Gas	1	20	NA	NA	1	NA	NA	NA	NA	NA

Notes:
1. All samples will be analyzed for volatile organic compounds (VOCs), the constituents of concern.
2. One trip blank required for every cooler or shipping container which contains VOC samples. If drinking water samples are shipped in the same cooler as other water or soil samples the trip blank will be analyzed by Method 8260B.

NA = Not Applicable

Figures



FE9

LEGEND

- FORMER TECUMSEH PRODUCTS SITE BOUNDARY
- CITY OF TECUMSEH PROPERTY BOUNDARIES
- PARCEL BOUNDARIES
- GROUNDWATER FLOW DIRECTION

NOTES

1. BASE MAP DEVELOPED FROM SITE PLAN PROVIDED BY THE CITY OF TECUMSEH, DRAWING NO. CITY.DWG, MARCH 2009. AERIAL PHOTOGRAPH PROVIDED FROM REMOTE SENSING & GIS RESEARCH AND OUTREACH SERVICES (RS&GIS), *PUBLICATION_DATE:* 06-29-2007, File:TECUMSEHSOUTH_NE.ECW.

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SCALE IN FEET

NO.	BY	DATE	REVISION	APP'D.
5.				
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FORMER TECUMSEH PRODUCTS SITE
TECUMSEH, MICHIGAN

SITE LOCATION PLAN AND VICINITY

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CHECKED BY:	SEM		FILE NO: 8070.10.01.dwg
APPROVED BY:	GC	DATE PRINTED:	**FIGURE 1**
DATE:	August 2010		

RMT
3754 Ranchero Drive
Ann Arbor, MI 48108-2237
Phone: 734-971-7080 • Fax: 971-9022

PLOT DATA
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Operator Name: LUCIDO, SAM
Drawing Plot Scale: 0.39983

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Attached Ref's:
Attached Images:
Layout

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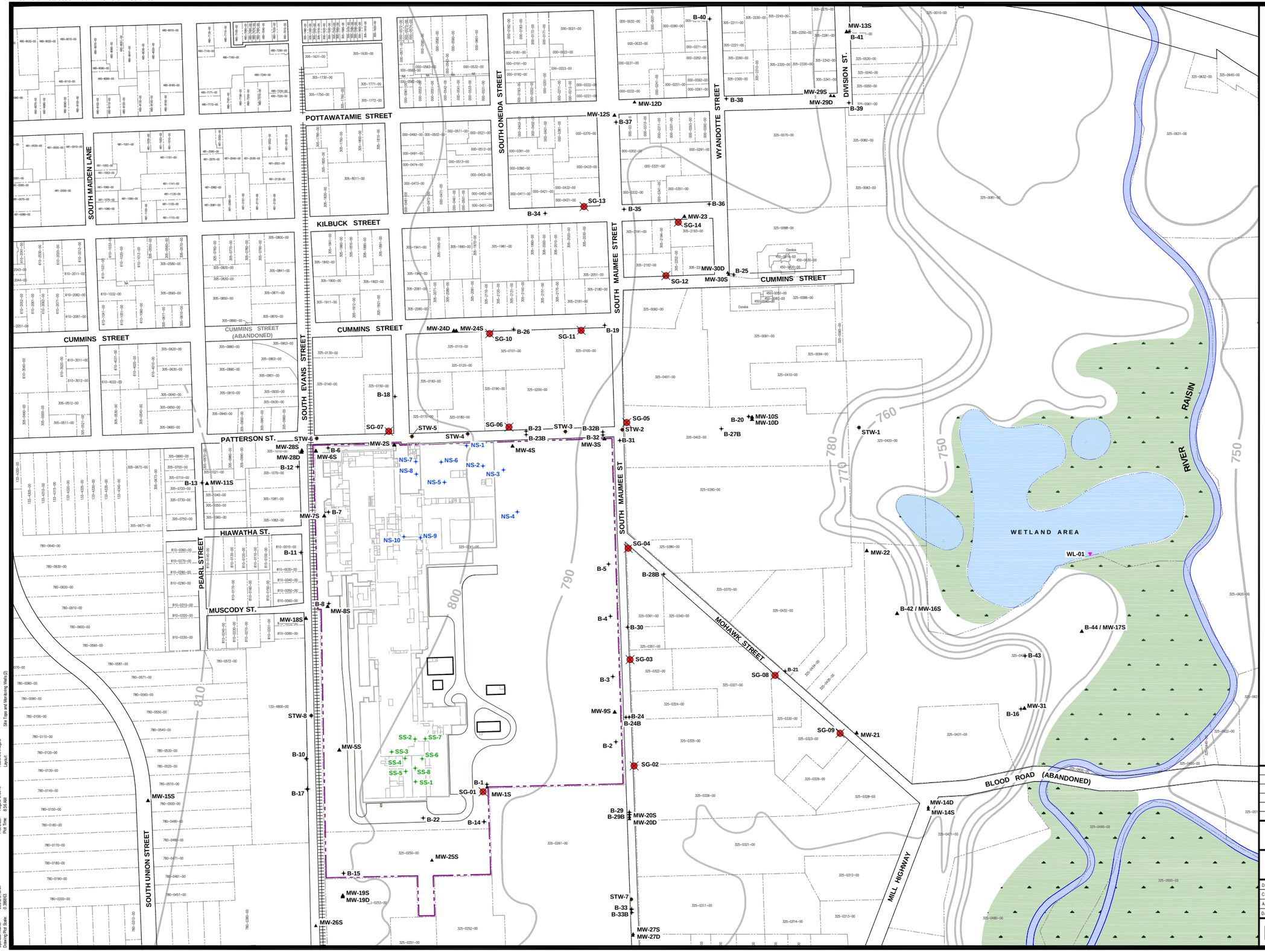

LEGEND

- FORMER TECUMSEH PRODUCTS SITE BOUNDARY
- PARCEL BOUNDARY
- RAILROAD TRACKS (APPROXIMATE LOCATION)
- 750 — APPROXIMATE GROUND TOPOGRAPHY BASED OFF 7.5 MINUTE U.S.G.S. TOPOGRAPHIC QUADRANGLE MAP
- B-2 ✦ PERIMETER / OFF-SITE INVESTIGATION SOIL BORING LOCATION AND NUMBER
- MW-4S ▲ MONITORING WELL LOCATION AND NUMBER
- NS-6 ✦ NORTHERN SOURCE AREA INVESTIGATION BORING LOCATION AND NUMBER
- SS-2 ✦ SOUTHERN SOURCE AREA INVESTIGATION BORING LOCATION AND NUMBER
- STW-2 ● STORM WATER SEWER SAMPLE LOCATION AND NUMBER
- WL-01 ▼ WETLAND SURFACE WATER SAMPLE LOCATION
- SG-02 ⊠ SOIL GAS SAMPLE LOCATION AND NUMBER
- FLOODPLAIN / WOODED WETLAND AREA

NOTES

1. BASE MAP DEVELOPED FROM SITE PLAN PROVIDED BY THE CITY OF TECUMSEH, DRAWING NO. CITY.DWG, MARCH 2009.
2. GROUND TOPOGRAPHY BASED OFF 7.5 MINUTE U.S.G.S TOPOGRAPHIC QUADRANGLE MAP AND GROUND SURVEY DATA.

0 200' 400' 600' 800'
SCALE IN FEET

NO.	BY	DATE	REVISION	APP'D.
5.				
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FORMER TECUMSEH PRODUCTS SITE
TECUMSEH, MICHIGAN

SURFACE TOPOGRAPHY AND SAMPLE LOCATIONS

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CHECKED BY:	SEM	AS INDICATED	FILE NO: 8070.10.02.dwg
APPROVED BY:	GC	DATE PRINTED:	**FIGURE 2**
DATE:	August 2010		

RMT
3754 Ranchero Drive
Ann Arbor, MI 48108-2237
Phone: 734-971-7080 • Fax: 734-971-9022

Site Topo and Monitoring Wells(2)
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Attached Images:
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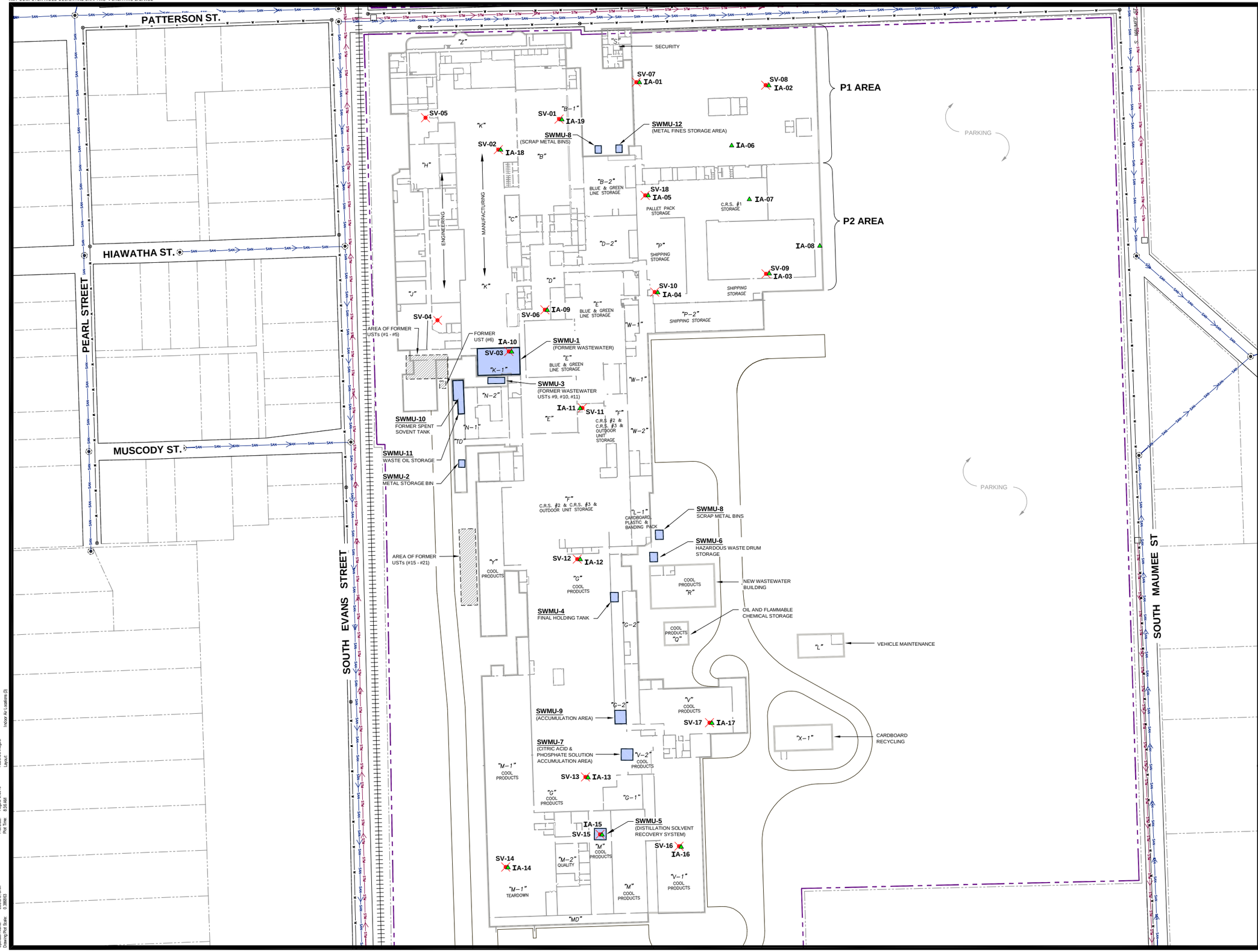

LEGEND

- FORMER TECUMSEH PRODUCTS SITE BOUNDARY
- BUILDING OUTLINE
- PARCEL BOUNDARY
- RAILROAD TRACKS (APPROXIMATE LOCATION)
- SAN SAN — SANITARY SEWER (APPROXIMATE LOCATION)
- STM STM — STORM SEWER (APPROXIMATE LOCATION)
- W — WATER MAIN
- MANHOLE
- WATER MAIN VALVE
- APPROXIMATE LOCATION OF FORMER SWMUs
- SV-08 — SUB-SLAB VAPOR SAMPLING LOCATION
- IA-02 — INDOOR AIR SAMPLING LOCATION

NOTES

1. BASE MAP DEVELOPED FROM SITE PLAN PROVIDED BY THE CITY OF TECUMSEH, DRAWING NO. CITY.DWG, MARCH 2009.
2. ALL UTILITY LOCATIONS APPROXIMATE AND OBTAINED FROM SITE PLANS PROVIDED BY THE CITY OF TECUMSEH.
3. SEE APPENDIX C OF THE CURRENT CONDITION REPORT FOR UST CONTENTS AND OTHER RELEVANT DATA.

N

0 80' 160' 240' 320'

SCALE IN FEET

NO.	BY	DATE	REVISION	APP'D.
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4.				
3.				
2.				
1.				

FORMER TECUMSEH PRODUCTS COMPANY
TECUMSEH, MICHIGAN

SITE FEATURES WITH INDOOR AIR AND SUB-SLAB VAPOR SAMPLING LOCATIONS

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CHECKED BY:	SEM	AS INDICATED	FILE NO: 8070.10.03.dwg
APPROVED BY:	GC	DATE PRINTED:	**FIGURE 3**
DATE:	August 2010		

RMT
3754 Ranchero Drive
Ann Arbor, MI 48108-2237
Phone: 734-971-7080 • Fax: 734-971-9022

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LUCIDO, SAM
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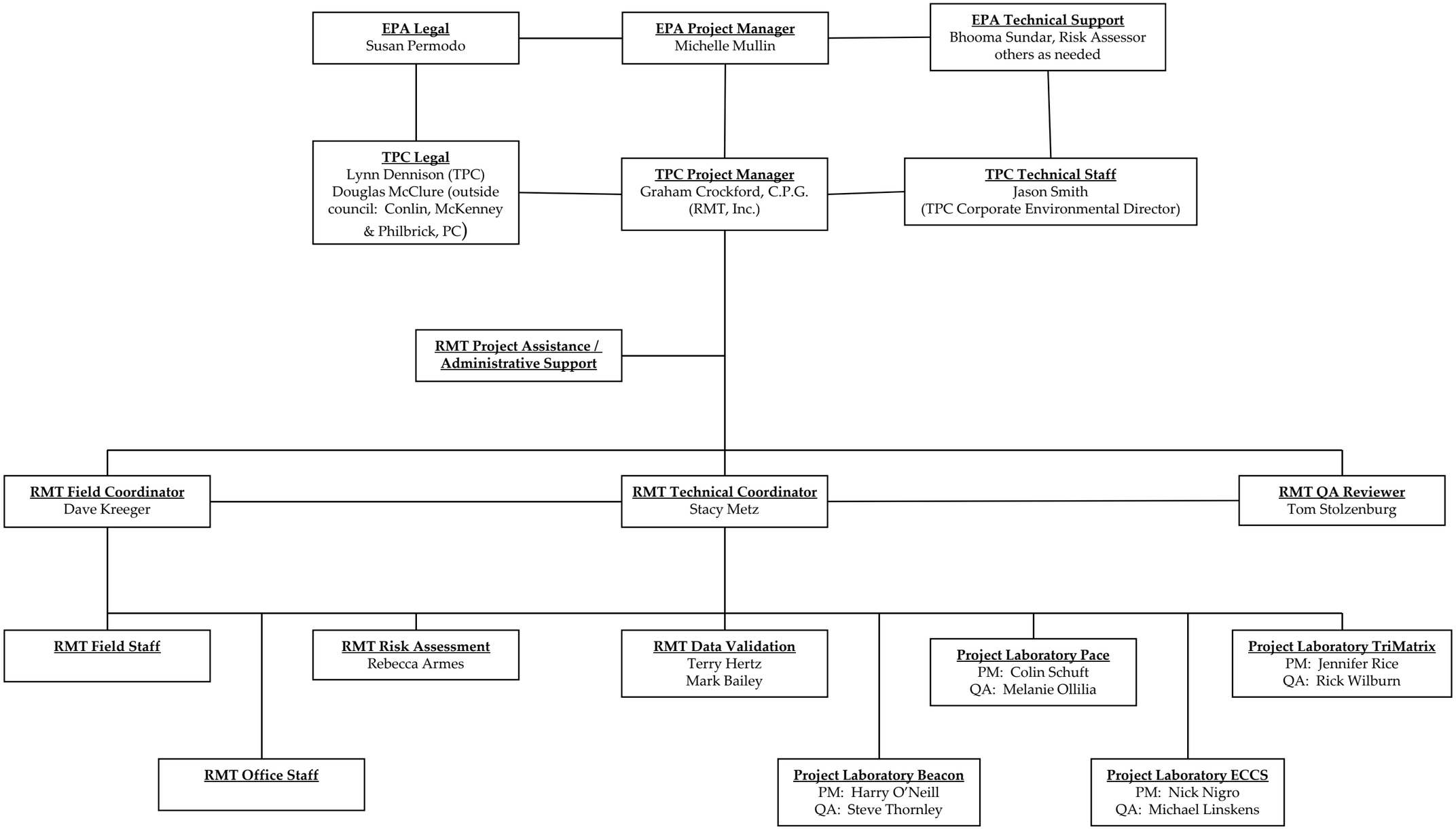
Layout:

Figure 4
Project Organization Chart
Former Tecumseh Products Company
Tecumseh, Michigan



Appendix A
PID Screening Methods

Technical Memorandum

Date: August 17, 2010

To: TPC Project Team

cc: Graham Crockford

From: Stacy Metz

Project No.: 00-08070

Subject: Use of Photoionization Detectors for Chemical Screening of Soil Samples at the Former Tecumseh Products Site in Tecumseh, Michigan

This memorandum was prepared to address the use of photoionization detectors (PIDs) designed for air monitoring as soil-screening tools at the former Tecumseh Products Company (TPC) Site in Tecumseh, Michigan (Site).

Volatile organic compound (VOC)-affected media are either known or suspected throughout a large portion of the Site. During subsurface investigations for TPC, our primary purpose for PID screening at these sites is to ensure that we are using appropriate personnel protective equipment (PPE). Over time, we, along with our industry as a whole, have expanded the use of PID screening to include the assessment of relative VOC concentrations in soil and/or groundwater samples[1]. Data generated by this method must be applied with caution and good professional judgment because of the numerous variables that can affect the measurement, such as the following:

- The PID is not a chemical specific instrument; you may be looking for TCE, but the instrument may be responding to another compound (*e.g.*, toluene).
- Not all chemicals are detected with equal sensitivity (PIDs are more sensitive to benzene than to ethenes and are more sensitive to ethenes than to ethanes). In addition, it is necessary to change the PID lamp to detect some compounds. The operations manual of the PID to be used for a project should be reviewed for details on lamp energies and instrument response factors.
- The field screening method requires that VOCs partition from a wet soil sample into the air space over the sample. The amount of partitioning is a function of the following:
    1. The concentration of the target chemical
    2. The soil sample temperature
    3. The vapor pressure of the target chemical
    4. The Henry's Law Constant of the target chemical
    5. The organic carbon and mineral partition coefficients of the target chemical
    6. The presence of semi-volatile oils into which the target chemical may be dissolved

[1] Fitzgerald, 1993. *Principles and Practices for Petroleum Contaminated Soils* (Boca Raton, Florida: Lewis Publishers, 1993), P46-66.

Technical Memorandum

7. The soil sample porosity
8. The soil sample lithology (*e.g.*, clay vs. sand) and mineralogy (*e.g.*, quartz vs. calcium carbonate)
9. The soil moisture content; some models of PIDs, such as Hnu, are more sensitive to the moisture content of the air being sampled. This should be considered when reviewing data from the Hnu PIDs.

All of these factors need to be considered when evaluating the applicability of PID screening and assessing the reliability of the resulting data. In most cases, we can use the PID readings to qualitatively identify samples containing moderate concentrations of VOCs (PID readings in the 10s of ppm-v) to high concentrations of VOCs (PID readings in excess of 100 ppm-v). In areas with uniform soil samples, "simple" contamination, and adequate laboratory verification, it may be possible to develop correlations between the field-screen and laboratory results and use the field screening data for "semi-quantitative" site characterization. Other areas may be too chemically or geologically complex to even warrant the use of PID screening methods for purposes other than health and safety screening. It is the responsibility of the professional designing the site investigation to decide whether PID screening is warranted based on available site information and professional judgment.

The following guidelines should be following when using a PID for field screening:

- The primary constituents of concern at the site are chlorinated solvents; therefore PIDs should be equipped with an 11.7 eV lamp. A standard 10.6 eV lamp will have little or no response to chlorinated compounds.
- Operate the instrument in accordance with manufacturer instructions.
- Calibrate the instrument daily.
- PIDs equipped with 11.7 eV lamps are particularly sensitive to moisture. Use an appropriate moisture filter at all times. If high moisture conditions are encountered, check instrument calibration frequently (*i.e.*, between each sample location). Do NOT attempt to collect PID readings from saturated soils.
- Use professional judgment in evaluating PID screening data. Typically low level PID readings (less than 10 ppm-v) should not be interpreted as reliable indicators of the presence of constituents of interest in soil unless they are accompanied by odors or other observations that suggest the presence of chlorinated VOCs.
- A "reporting limit" (*e.g.*, 10 ppm-v) may be established for field-screening soils and that all readings below this value will be recorded as less than (*e.g.*, <10 ppm-v). Alternatively, at some locations it may be appropriate to remove PID screening results from boring logs altogether. We will need to address these options when developing workplans. By using one of these options, we can minimize potential agency comments and criticisms where low level readings are measured but samples are not selected for laboratory analysis.

Technical Memorandum

This memorandum should not be used to contravene approved workplans that contain regulatory agency specific requirements. However, we should use the recommendations contained in this memorandum in preparing workplans that will be subject to regulatory agency review and approval.

Appendix B
Laboratory QA Manuals on CD

Quality Assurance Manual

Analytical Services

Release Date December, 2008

Prepared by:
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1.0 TITLE PAGE

QUALITY ASSURANCE MANUAL

Policies and Procedures Required of the Personnel Employed by TriMatrix Laboratories, Inc., Including the Organic, Inorganic, and Metals Laboratory Areas

Revision Number: 7.0

Effective Date: December 2008

Initial Approvals:

Quality Assurance Manager: _____ Date: 12/2/08

Technical Director: _____ Date: 12/5/08

Laboratory President: _____ Date: 12/4/2008

Subsequent Approvals:

Quality Assurance Manager: _____ Date: 2/16/10

Quality Assurance Manager: _____ Date: _____

Quality Assurance Manager: _____ Date: _____

Quality Assurance Manager: _____ Date: _____

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7.0 APPENDICES

3.0 QUALITY SYSTEM

3.1 INTRODUCTION: THE TriMatrix QUALITY SYSTEM

3.1.1 Manual Purpose

The purpose of this manual is to outline the organization, specify the procedures, and define the technical requirements utilized by TriMatrix Laboratories, Inc. The goal is to ensure that all data generated is of the required quality, is reproducible, and is generated in a timely manner. This manual details a Quality Assurance/Quality Control (QA/QC) program encompassing the entire analytical efforts at TriMatrix, from project initiation to report generation. Some areas are covered with only a cursory discussion, while others are covered in detail, or are included in more than one section, depending on their importance. This manual describes the realistic functions of the quality programs in place, with an understanding that not every situation is covered nor every contingency explored.

3.1.2 The Need for Analytical Quality Assurance/Quality Control

In the increasingly competitive business of environmental laboratory services, the primary tenet of continued success is to efficiently provide results of the necessary quality. TriMatrix agrees with this tenet, considers analytical quality assurance and quality control to be of prime importance, and has incorporated it as the central pillar of our efforts to remain on the leading edge of the environmental laboratory field. The requirements we place on ourselves are in concert with the needs and agendas of other organizations, such as the Environmental Protection Agency (EPA), governmental and industrial clients, and various state and local regulatory agencies.

Quality assurance and quality control (QA/QC) functions absorb nearly fifty percent of the available effort involved in routine analysis, and continues to evolve and grow in importance. This level of quality is absolutely essential for two reasons: 1) accurate analytical data is obtained only with the concurrent

use of extensive QA/QC to regulate and monitor the many process variables that can potentially introduce errors into chemical analyses, and 2) clients make crucial business decisions based on the data supplied by the laboratory. Lab data not properly supported by adequate quality assurance/quality control practices and procedures can be questionable at best, and can lead to faulty or erroneous decisions in the field. In the overall analytical effort the additional time spent for QA/QC is time necessarily spent.

3.1.3 Definition of Terms

3.1.3.1 Quality Assurance

Quality Assurance (QA) is defined as those operations and procedures undertaken to provide measurement data of documentable quality that have a stated probability of being accurate. The measurement system part of the quality assurance program must be in statistical control to justify this probability statement.

The operations and procedures established as part of the overall quality assurance program encompass all aspects of the laboratory operations, including but not limited to: organizational structure, human resources, physical resources, methodology, analyst training and certification, data reduction, data validation, and instrument maintenance and troubleshooting. All aspects of QA are organized, implemented, and monitored through written standard operating procedures.

3.1.3.2 Quality Control

Quality control is defined as the basic checks necessary to produce a good measurement program. These checks include but are not limited to: proper calibration and calibration verification, statistical monitoring of accuracy and precision, of quality control samples

(e.g. laboratory control samples, blanks, duplicates, spikes, etc.), interference monitoring, and reagent control.

Adequate records are maintained to support data quality, to locate assignable causes in measurement problems, to improve the accuracy and precision of the measurement system, and to provide a historical record of traceability.

3.1.3.3 Quality Assessment

Quality assessment is defined as those specific steps utilized to evaluate the quality of the measurement process. These steps include use of control charts to plot multiple data points over time, monitoring parameters by statistical control, internal performance audits, external performance audits, certification programs conducted by individual states, and performance evaluation sample programs.

3.2 QUALITY POLICY STATEMENTS FROM MANAGEMENT

As communicated from top management through the entire organization, TriMatrix Laboratories, Inc. is driven by the following quality objectives and commitments.

3.2.1 Corporate Quality Objectives

- To create and maintain a uniform and controlled pattern for performing routine tasks within the organization, based on standard operating procedures.

- To generate legally defensible, scientifically sound laboratory data of documented quality.

- To build quality into the workplace ensuring services contributing to successful relationships with our customers, employees, and vendors.

- To develop, deliver, and maintain, excellence in all operational areas.

- To provide a service that consistently meets or exceeds client expectations.

3.2.2 Corporate Quality Commitments

- To support quality by underwriting the substantial cost of the quality commitment even though such expenses do not result in increased productivity or a tangible product.

- To maintain a work environment in which all employees are free from commercial pressures in the performance of their duties.

- To maintain a work environment in which all employees are free from internal organization or external client related pressures that may influence the quality of their work.

- To educate all employees in fraud prevention and their ethical responsibilities associated with analytical and data reporting activities.

- To ensure that client confidentiality and information are strictly protected.

- To implement on-going improvement in every area of laboratory activity.

- To create and maintain a Quality Environment with an all-encompassing determination to meet the needs and quality objectives of our clients.

- To commit and adhere to the requirements specified in ISO/IEC 17025.

- To commit and adhere to the requirements specified by the NELAC Standards.

Included with these improvements and commitments is an annual review process where the management of TriMatrix Laboratories performs a comprehensive review of the quality system. This review monitors the effectiveness of the quality system and provides feedback for on-going improvement. Policy changes made as a result of the annual review will be reflected in the QA Manual.

3.3 ORGANIZATION AND RESPONSIBILITIES

An efficient organizational operation requires a quality control program facilitating a high level of multi-directional communication and information flow. Each person in the TriMatrix organization inputs and receives information from the quality system. This information flow optimizes management directives with minimum disruption, and provides the means for creating improvements.

3.3.1 Corporate Structure

Flow of both administrative and quality control information is presented in Figure 3-1. This diagram graphically displays the corporate philosophy concerning the interaction of QA/QC and the generation of analytical data. The general flow of data in this format gives QA/QC independence in fulfilling its function while still acting as a liaison with the administrative staff. To further explain this interaction, a detailed description of roles and responsibilities is presented for each key laboratory position.

3.3.2 Laboratory President

Responsibilities of the Laboratory President are directed at the overall operation and management of the laboratory. Primary responsibilities include, but are not limited to: 1) develop and meet budgets established for the laboratory, 2) manage analytical services productivity and quality, 3) oversee and develop new business activities including client relations development, 4) plan analytical services organization, leadership and management programs, 5)

develop and manage human resources including career path planning, and 6) performing duties as Deputy Technical Director when necessary.

3.3.3 Quality Assurance Manager

The Quality Assurance Manager is primarily responsible for the implementation, maintenance, reporting, and development of all QA/QC activities performed within the laboratory. Duties include, but are not limited to: 1) QA/QC systems development and monitoring, 2) coordination of all documentation procedures including the development and control of standard operation procedures, 3) monitoring method and quality control requirements as published by regulatory agencies ISO/IEC 17025, and the NELAC Standards, 4) performing internal lab audits, 5) maintaining in-house QA/QC monitoring procedures and policies, and 6) providing quality assurance guidance and training to all staff members. The Quality Assurance Manager has the authority to stop work as a result of poor data quality.

3.3.4 Technical Director

The Technical Director is responsible for the overall technical capabilities and direction of the laboratory. Specific responsibilities include: 1) organization and management of new analytical technologies developed by the laboratory, 2) adherence to ISO/IEC 17025 requirements and NELAC Standards, 3) equipment procurement management.

3.3.5 Health and Safety Officer

The Health and Safety Officer is responsible for implementation, monitoring, and maintenance of all laboratory safety and chemical hygiene programs. Specific responsibilities include the development and maintenance of health and safety programs and manuals.

3.3.6 Vice President of Laboratory Operations

The Vice President of Laboratory Operations is responsible for the overall supervision of the individual laboratory areas. General responsibilities include management of staff activities such as scheduling, budgeting, training, and general supervision. The Vice President of Laboratory Operations also is responsible for 1) the development and management of all chemists, analysts, technicians, 2) implementation of quality systems and controls within the laboratory, 3) scheduling analysis activities, 4) meeting productivity goals and project deadlines, 5) technical development of the laboratory staff, 6) approval of laboratory's SOPs, 7) coordination of methods development with the staff and Technical Director, 8) approval of laboratory data, or the delegation thereof, 9) Approval of procurement activities, 10) Overall laboratory performance, and 11) adherence to ISO/IEC 17025 requirements.

3.3.7 Client Services Manager

The laboratory Client Services Manager supervises both the Client Services and the Data Management Group. Responsibilities of the Client Services Manager include management of scheduling and method development needs, budgeting, training, and general supervision, with specific emphasis on the following activities: 1) development and management of all project chemists, project chemist technicians, log-in staff, bottle preparation staff, laboratory couriers, the Field Services Group, and Data Management Group, 2) project management, 3) coordination of proposal preparation and marketing activities for existing and new clients, 4) monitoring of final report turnaround times and, 5) monitoring client satisfaction with laboratory services.

3.3.8 Deputy Quality Assurance Manager/Deputy Technical Director

The Deputy Quality Assurance Manager/Technical Director has the responsibility of fulfilling an interim role as outlined in sections 3.3.3, 3.3.4, 3.5.1.2, and 3.5.1.3.

3.3.9 Sales and Marketing Staff

The Sales and Marketing Staff are responsible for all marketing, business development, and client maintenance activities. These activities include but are not necessarily limited to: 1) market research/gathering market intelligence, 2) consulting with company management to develop a corporate business strategy and plan, 3) development and implementation of a corporate image campaign, 4) development and distribution of marketing materials (corporate literature, etc.), 5) client prospecting, 6) presenting/introducing company services to prospective clients, 7) account development, management and maintenance (in conjunction with Project Chemists), 8) development of corporate pricing guidelines, 9) development of proposals, quotations, bids and qualifications summaries, and 10) contract review, negotiation and execution.

3.3.10 Organizational Chart

Presented in Figure 3-2 is an organizational chart illustrating the personnel structure within the laboratory.

3.4 RELATIONSHIPS

Relationships within the analytical laboratory are organized through management into three main categories: Technical Operations, Support Services, and the Laboratory Quality System. The relationships between management and these operations define and maintain the delicate balance in a cost-effective, highly-technical, quality laboratory operation. An overview of each relation is presented below:

3.4.1 Management-Technical Operations

The relationship between management and technical operations is illustrated in Figure 3-3. In this relationship, the main role of management is to provide guidance and financial support to the programs and directives of the Technical Director. Through this structure, technical operational enhancements and developments occur and are applied through the laboratory staff.

3.4.2 Management-Support Services

The relationship between management and support services is illustrated in Figure 3-4. In this relationship, management's role is substantial in the day-to-day operation of each service.

The primary laboratory support groups are Client Services, Sales and Marketing, and LIMS system support. These groups report directly to the Laboratory President for all aspects of their daily activities.

Secondary relationships are maintained with the Laboratory Administrative Assistant, Laboratory Receptionist, Accounting, and the Human Resources Department. Some groups within this secondary category maintain relationships not only with the Laboratory President, but also with other management groups within the TriMatrix organization.

A tertiary relationship has been developed between the Laboratory President and Vice President of Laboratory Operations. This relationship supports productivity monitoring, cost containment, equipment procurement, operations management, personnel/human resources activities, technical support, data validation, and method development.

3.4.3 Management-Quality System

The relationship between management and the laboratory quality system is illustrated in Figure 3-5. In this relationship, management plays a secondary role in the overall scheme. This secondary role provides the quality assurance manager with guidance, company perspective, and structured support in the development, implementation, and maintenance of quality system programs and activities.

This relationship is vital to the success of TriMatrix Laboratories. Without a cost-effective quality system, the overall caliber of laboratory data and the success of all laboratory operations would be jeopardized.

A relationship also exists between management, the quality system, the laboratory support, and the HR staff. This relationship includes but is not limited to: laboratory management directives, and human resources/personnel activities. These activities are implemented and maintained without disruption to the quality system, and are depicted via the dashed lines on Figure 3-5.

3.5 JOB DESCRIPTIONS

The strength of a laboratory lies in the experience and dedication of its employees. TriMatrix hires quality personnel based both on work attitude and past job experience. Job descriptions have been written to define the employee qualifications required for each position.

3.5.1 Management Staff Members

Managerial positions are responsible for the development of their respective employees. These positions have specific minimum requirements for years of experience.

3.5.1.1 Laboratory President

Job Description

The Laboratory President (LP) directs the laboratory. The LP works through the Vice President of Laboratory Operations to improve data quality, overall productivity, staff development, safety/training programs, and overall profitability. This position has profit/loss accountability. Budgets are developed annually with senior management. The LP is also directly involved in business development/sales activities, and the sales staff reports directly to him.

Background/Educational Requirements

The LP possesses minimally a bachelor's degree in science, preferably chemistry. The LP has a minimum of 10 years direct

work experience in the environmental testing industry. This work experience includes having conducted environmental analyses and several years of demonstrated supervisory experience.

Duties and Responsibilities

1. Development and fulfillment of budgets.
2. Management of total laboratory productivity and quality.
3. Management of proposal preparation.
4. Development of new business and maintenance of client relationships.
5. Development of laboratory organization, leadership, and management planning.
6. Working with the Human Resources department to develop staff members and their career paths.

3.5.1.2 Quality Assurance Manager

Job Description

The Quality Assurance (QA) Manager is responsible for the development, implementation, improvement, and maintenance of all quality systems at TriMatrix. The QA Manager monitors all the analytical methods and procedures performed by the laboratory, and assures compliance with regulatory agency requirements.

Background/Educational Requirements

The QA Manager possesses a B.S. in science, preferably chemistry, and suitable work experience. Work experience must include several years of analytical work and a demonstrated ability to work with and train staff members. A strong working knowledge of quality assurance and statistical quality control procedures, specifically as they apply to analytical protocols, is required.

Duties and Responsibilities

1. Development and implementation of systems to measure and monitor laboratory data quality.
2. Maintenance of the documentation system for generation, control, and archiving laboratory forms, SOPs, and protocols.
3. Approving SOPs and monitoring their compliance with regulatory agency requirements.
4. Maintaining and updating the laboratory Quality Assurance Manual.
5. On-going investigation for optimizing procedures to minimize out-of-control data.
6. Maintenance of federal, state, and industrial certifications and accreditations as required.
7. Monitoring internal quality programs within the laboratory and reporting their status to management.
8. Training and training documentation of all staff members in all aspects of the laboratory quality system.
9. Perform other duties as deemed necessary by management.

3.5.1.3 Technical Director

Job Description

The Technical Director (TD) is responsible for the development and improvement of technical operations within the laboratory division. The TD oversees the investigation of all new instruments and equipment, method development, and general technical advancement of the laboratory. The TD is also responsible for informing the Deputy TD of current and pending projects and activities.

Background/Educational Requirements

The TD possesses a B.S. in science, preferably chemistry, and suitable work experience. Such work experience includes several years of analytical work and a demonstrated ability to work with and train staff members. A strong working knowledge of

instruments and methodologies, specifically as they apply to analytical protocols, is required.

Duties and Responsibilities

1. On-going technical development of the TriMatrix Laboratory pertaining to current and future analytical practices.
2. Overseeing the technical development of TriMatrix staff in the areas of method comprehension and implementation.
3. Development of new analytical procedures within the laboratory.
4. Providing technical advice regarding all equipment and apparatus procurement, and acquisitions.
5. Performing technical review of all Quality Assurance Project Plans (QAPPs).
6. Perform other duties as deemed necessary by management.

3.5.1.4 Client Services Manager

Job Description

The Client Services (CS) Manager is responsible for the supervision of the project chemists, project chemist technicians, sample log-in staff, bottle preparation staff, laboratory couriers, field services group, and laboratory administrative staff. These responsibilities include meeting project due dates, preparing and reviewing quotations, project initiation and management, client satisfaction management, and supervision and training of staff. The CS Manager strives for improvement in the on-time delivery of laboratory projects.

Background/Educational Requirements

The CS Manager possesses a B.S. in science, preferably chemistry, and has 5-10 years of work experience. The work experience includes 3-5 years of laboratory experience, involvement in client

management activities, and a demonstrated ability to supervise and train laboratory staff.

Duties and Responsibilities

1. Responsible for the productivity and quality of the client services group.
2. Management of large Level 3 or higher projects.
3. Quality control program implementation and maintenance.
4. Supervision and technical development of employees.
5. Development and maintenance of standard operating procedures.
6. Assisting and coordinating marketing activities through proposal preparation and client visitation.
7. Perform other duties as deemed necessary by management.

3.5.1.5 Vice President of Laboratory Operations

Job Description

The Vice President of Laboratory Operations (VPLO) is responsible for the individual laboratory areas and the supervision of laboratory staff. These responsibilities include meeting project schedules, and the supervision and training of staff members. The VPLO continually works to improve the quality of data generated.

Background/Educational Requirements

The VPLO possesses a B.S. degree in science, preferably chemistry, and 5-10 years work experience. The work experience includes a minimum of 5 years in the laboratory utilizing a variety of techniques. The VPLO must also demonstrate an ability to supervise and train staff members.

Duties and Responsibilities

1. Responsible for the productivity and quality of the laboratory areas.

2. Operation and maintenance of instrumentation and apparatus.
3. Quality control program implementation and maintenance.
4. Reviewing and final approval of all organic data.
5. Scheduling in-house to allow on-time report generation.
6. Supervision of supply acquisition activities.
7. Supervision and technical development of employees.
8. Approval of standard operating procedures.
9. Methods development.
10. Perform other duties as deemed necessary by management.

3.5.1.6 Laboratory Computer Systems Administrator

Job Description

Provide technical review, guidance, and training in current and future laboratory computer applications.

Background/Educational Requirements

Requires a degree in computer sciences with an emphasis in a chemistry or general science curriculum.

Duties and Responsibilities

1. Developing a complete understanding of the Laboratory Information Management System (LIMS).
2. Reviewing laboratory computer applications and processes, including instrument computer interfaces, data transmission/archiving processes and document control.
3. Providing database maintenance support activities for the LIMS system.
4. Providing technical direction and orchestrating implementation of electronic storage systems for the laboratory.
5. Providing technical training of the laboratory staff in software applications and basic computer operational activities.
6. Perform other duties as deemed necessary by management.

3.5.2 Technical Staff Members

Technical staff members are classified into chemist or technician levels dependant on job type, education, and years of experience. Level Classifications are Chemist I-V and Senior Chemist, Project Chemist I-V and Senior Project Chemist, Technician I-V and Senior Technician. In addition, qualified candidates are also eligible for group leader status. Classification descriptions are provided in Appendix A. To aid the employee in identifying the different classification requirements, the differences are printed in bold italicized text. The various classifications are also used by the employee and by management for career path development at TriMatrix.

3.6 MANAGEMENT RESUMES

Laboratory President
Quality Assurance Manager
Vice President of Laboratory Operations
Human Resources Manager

DOUGLAS E. KRISCUNAS — Laboratory President

EDUCATION

B.S., Environmental Sciences, Grand Valley State University, 1976

PROFESSIONAL SUMMARY

Mr. Kriscunas is responsible for the accuracy and integrity of all analytical data finalized at this location. He is continuously available for client support to resolve analytical issues as they pertain to environmental problems.

PROFESSIONAL EXPERIENCE

- **Detroit, Michigan**. Laboratory Supervisor for a field laboratory established at the Detroit Wastewater Treatment Plant. The project involved a one-year pilot study of the overall operation and plant performance to upgrade and modify existing treatment processes to meet current and future discharge limits. Approximately 20,000 samples were analyzed by seven full-time analysts.

- **Edmore, Michigan. Hitachi Magnetics Corporation.** Participated in the development and implementation of an on-site, flow-through bioassay of the plant discharge. The study was performed in conjunction with the Michigan Department of Natural Resources, Water Quality Division.

- **Grand Rapids, Michigan. EDI Laboratory Certification.** Direct responsibility for the inorganic parameters analysis and quality control measures necessary for laboratory certification under the Safe Drinking Water Act (SDWA) of 1974. Certification involved both analysis of unknown control samples and corresponding on-site evaluation by the U.S. EPA Region V laboratory certification team.

- **Muskegon, Michigan. Uniroyal Chemical Company.** Participated in the soil survey and on-site evaluation of potential soil contamination from deposited chemical waste materials produced by a major chemical company. On-site sample analyses for select parameters were made to locate and detail the extent of contamination.

- **Edmore, Michigan. Hitachi Magnetics Corporation.** Participated in the implementation of a treatability study to effectively remove cobalt and samarium from industrial waste. The study results led to the design and installation of treatment facilities.

- **Columbia, Missouri. A.B. Chance Corporation**. Responsible for implementing a treatment study for effective removal of heavy metals from process wastewater in order to achieve acceptable discharge limits.

- **Kent County, Michigan. Mill Creek Watershed Management Project**. Participated in the collection, mapping, and interpretation of environmental characteristics to be used as prototype guidelines for the management of area wide streams in the Great Lakes Basin. The project was funded by the Environmental Protection Agency.

- **Three Rivers, Michigan. Hydramatic Division, General Motors Corporation.** Responsible for the analytical services conducted on a survey of process wastewater for an automotive transmission manufacturer. The project involved data collection and analytical services including grab samples, setting automatic samplers on an hourly basis for a seven-day period, and installing recording meters for continuous pH monitoring.

- **Grand Rapids, Michigan. Michigan Department of Public Health Laboratory Certification.** Supervised analytical, bacteriological, and quality control activities involved in achieving certification status for the analysis of potable water supplies in Michigan.

- **Higgins Lake, Michigan. Ralph MacMullan Conference Center**. Served on a three-member panel before a meeting of the Northern Michigan Environmental Health Association. The topic of discussion was an overview of organic chemicals now found in much of Michigan's ground waters. A representative from industry and the MDPH laboratory completed the panel.

- **Grand Rapids, Michigan. Haviland Chemical Company.** Coordinated a static bioassay performed on a water-based detergent utilizing fathead minnows in the 96-hour static test.

- **Sparta, Michigan**. Conducted a dendrological survey of a proposed oil drilling site. The survey was incorporated in an overall environmental assessment of the proposed drilling site.

- **Caledonia, Michigan**. Conducted a dendrological survey of riparian vegetation types located along the banks of the Thornapple River in the area of the Labarge Dam.

- **Grand Haven, Michigan**. Conducted a limnological investigation of the estuary waters of the Grand River watershed near Grand Haven. The collected limnological data were evaluated for potential eutrophication problems resulting from nutrient discharges upstream.

- **Kalamazoo, Michigan. American Cyanamid Company**. Supervised laboratory work required in assisting a major chemical manufacturer with a permit application for existing facility hazardous waste management operation to administratively complete four supplemental technical attachments, multidisciplinary services were required in the areas of hydrogeologic investigation, environmental assessment, failure mode assessment, and engineering review. Field work was completed in 19 days with a report to the client in 25 days to meet scheduled deadlines.

- **Kent County, Michigan**. Coordination of field and laboratory services in conjunction with Act 641 monitoring requirements at two county-owned and operated refuse sites.

Specialized studies were also conducted to identify possible use of landfill gases for electric power generation and the source identification of volatile organic contaminants typical of most municipal landfills.

- **Cascade Township, Michigan. Cascade Resource Recovery/Waste Management, Inc.** Implementation of two separate tracer studies aimed at pinpointing possible cracks or defects in the clay liners of four hazardous waste disposal trenches. The study utilized a low absorptivity fluoroscene water soluble dye introduced to each trench. Samples collected from each liner failure detection system were then analyzed for the fluorescent characteristics of the dye.

- **Cascade Township, Michigan. Cascade Resource Recovery/Waste Management, Inc.** Coordination of field and laboratory services in connection with Michigan Department of Natural Resources Act 64 and U.S. EPA RCRA monitoring requirements. Each sampling event involves collection of ground waters, surface waters, and leak detection monitoring sites.

- **Cascade Township, Michigan. Cascade Resource Recovery/Chemical Waste Management, Inc.** Acted as project chemist and field services coordinator for activities involved in the excavation and site decontamination of an Act 64/RCRA hazardous waste disposal facility. The decontamination program involved the analysis of soils collected in and around each disposal trench after the removal of approximately 20,000 cubic yards of waste materials.

- **Cincinnati, Ohio. Rumpke Waste Systems, Inc.** Acting project manager for a large waste disposal firm headquartered in Ohio, with 20+ landfills located in a 5 state geographical area. Mr. Kriscunas is responsible for coordination of laboratory activities in conjunction with all ground water, surface water, and NPDES monitoring requirements.

RICK D. WILBURN — Quality Assurance Manager

EDUCATION

B.S., Environmental Studies, Earlham College, 1985

PROFESSIONAL SUMMARY

Mr. Wilburn is responsible for all aspects of the laboratory Quality Control/Quality Assurance Program. Primary responsibilities include conducting internal and external auditing of the laboratory, procurement and maintenance of state and federal certifications, and ensuring that all facets of the quality control program remain at the highest level possible. Mr. Wilburn also manages the external and internal Quality Control check sample programs.

PROFESSIONAL EXPERIENCE

- **TRACE Analytical Laboratories, Inc. – Quality Assurance Manager, 12/95 – 10/96.** Responsible for designing, implementing, and monitoring a formal quality control program. The program included: conducting internal and hosting external audits, implementing corrective actions resulting from any deficiencies, scheduling and reporting performance evaluation sample results, and the review of all Level 5 data packages.

- **EARTH TECH – Organic Laboratory Manager, 10/95 – 12/95.** As Organic Laboratory Manager, Mr. Wilburn was responsible for the day-to-day operations of the organic laboratory, including volatile and semi-volatile analyses by gas chromatography and gas chromatography/mass spectrometry. His responsibilities included scheduling, instrument maintenance, the writing and implementation of standard operating procedures, quality assurance, analytical data review, the technical development of all the organic laboratory personnel, and project management. Mr. Wilburn was also responsible for research and development in the organic laboratory, focusing on ways to automate and improve sample analysis, data quality, and turnaround time.

- **EARTH TECH (Formerly WW Engineering & Science) – Semi-Volatile Laboratory Supervisor, 1/94 – 10/95.** Responsible for the daily operation of the semi-volatile laboratory. The semi-volatile laboratory utilizes gas chromatography, gas chromatography/mass spectrometry, and high performance liquid chromatography in the analysis of semi-volatile organic compounds.

- **WW Engineering & Science – Supervisor, Organic Extraction Laboratory, 4/93 – 1/94.** Supervisor of the staff of chemists responsible for all organic extractions. Accountable for the processing, quality, and turn around of a wide variety of samples involving many extraction techniques and methodologies. Continually experimenting with automation and new technologies to improve extraction quality and turn around time, including solid phase and supercritical fluid extractions.

- **WW Engineering & Science – Supervisor, Mass Spectrometry Laboratory, 9/89 – 1/94.** Supervisor of the staff of chemists analyzing samples for semi-volatile organics in the mass spectrometry laboratory. Oversee all analysis and daily activities involved with the mass spectrometry laboratory. Evaluate, recommend, and implement new technologies. Implementations of these include sub-ambient injections using a Varian SPI injector, sub-ambient temperature programs for optimized chromatography, and the use of ion trap mass spectrometers for lower operating detection limits

- **IT Corporation, (formerly PEI Associates, Inc.) – Chemist, Level 3, GC/MS Semi-Volatile Team Leader, 7/88 – 9/89.** Along with daily analysis of samples, responsible for coordinating the efforts of the three analysts and three instruments used for semi-volatile analysis. This included scheduling each instrument/analyst to make sure analyses were completed correctly and on time, training new personnel, instrument maintenance, data checking, and reporting project results to management for client distribution. Leader of GC/MS Quality Circle group.

- **PEI Associates, Inc. – Chemist, Level 2, GC/MS Analyst, 12/86 – 7/88.** Primary responsibilities included analyzing soil, water, and other media with an Extrel ELQ-400 mass spectrometer system. Analyses performed included semi-volatile and volatile organics listed on the EPA's Toxic Compounds List according to the Contract Laboratory Program protocol. Also analyzed various other non - Toxic Compounds List compounds using appropriate methods.

- **PEI Associates, Inc. – Chemist, Level 1, GC Analyst, 7/85 – 12/86.** Carried out a variety of organic analyses in a wide range of matrixes. Was a primary analyst conducting CLP testing for pesticides and PCBs, and was the primary analyst for routine and non-routine testing for herbicides, and volatile organics.

JEFFREY P. GLASER — Vice President of Laboratory Operations

EDUCATION

B.S., Biochemistry, Michigan State University, 1987

PROFESSIONAL SUMMARY

Mr. Glaser is responsible for the operation and management of the laboratory areas. Main functions include supervision and training of personnel, formulation of standard operating procedures, final approval of laboratory data, and laboratory purchase approval.

PROFESSIONAL EXPERIENCE

- **TriMatrix Laboratories, Inc., Muskegon – Laboratory Manager, 1994 – 1996.** Responsible for all aspects of laboratory performance. He was responsible for all aspects of laboratory performance including, analytical testing and reporting; business development; customer service; capital expenditures, quality control; quality assurance; laboratory safety; and laboratory profitability. He was responsible for the hiring, training, guidance, and evaluation of all laboratory personnel, and for direction of overall laboratory policies and practices.

- **Great Lakes Environmental Laboratories – Senior Chemist, 1992 – 1994.** Mr. Glaser's responsibilities included supervision and training of other laboratory personnel, coordination of sample workloads, data review and evaluation, and quality control. He was also responsible for analysis of pesticides, PCBs, and herbicides using an HP 5890 GC w/ECD detectors.

- **Anatech Analytical Laboratories – GC/MS Operator, 1990 – 1992**. Mr. Glaser was responsible for the mass spectrometry analysis of environmental samples in a variety of matrixes for both volatile and semi-volatile organics. For volatiles, Mr. Glaser operated and maintained a Finnigan Ion Trap GC/MS system consisting of a Varian GC and a Tekmar purge and trap autosampler. Primary methodology used was 624/8240. For semi-volatiles, he operated and maintained a Hewlett Packard GC/MSD UNIX-based Chem Station. Primary methodology used was 625/8270. He was also responsible for method development. He served as the Organic Supervisor for the first quarter of 1991.

- **Anatech Analytical Laboratories – Volatile Organic Chemist, 1989 – 1990**. Mr. Glaser was responsible for operation and maintenance of two volatile GC systems utilizing ELCD, FID, and PID detectors, and Tekmar and O.I. Analytical purge and trap autosamplers. Primary analyses were 601 and 602.

STACY K. VANDEN AKKER — Human Resources/Business Manager

EDUCATION

B.S. Business Management, Davenport Business College, 1996.

PROFESSIONAL SUMMARY

As Business Manager, Ms. Vanden Akker is responsible for the record keeping and review of all financial data for the company. She manages accounts payable, accounts receivable, cash flow, and the generation of financial statements and other management reports. She maintains accurate records for potential audit or other review.

Ms. Vanden Akker also manages all Human Resource functions for TriMatrix Laboratories. She processes payroll on a biweekly basis, coordinates employee benefits, handles internal employee questions and concerns, assures compliance with all federal, state, and local employment laws and regulations, and maintains complete and accurate personnel data files.

PROFESSIONAL EXPERIENCE

- **EARTH TECH – Environmental Laboratory Business Office, Administrative Assistant, 9/95 – 1/97.** Responsible for assisting the Business Office Manager with accounts receivable, accounts payable, and the daily input of purchases and invoices.

- **EARTH TECH – Lowell Wastewater Treatment Plant Operator/Laboratory Technician, 8/93 – Present.** Responsible for sample collection, equipment maintenance, and the daily laboratory analysis of suspended solids, CBOD, ammonia, zinc, fecal coliform, pH, residual chlorine, and phosphates. She is also responsible for the correct input of all results into the reports required by the State of Michigan Department of Environmental Quality.

- **EARTH TECH – Lowell Wastewater Treatment Plant Assistant Laboratory Technician, 8/90 – 8/93.** Assisted the Laboratory Technician in the laboratory analysis of suspended solids, CBOD, ammonia, zinc, fecal coliform, pH, residual chlorine, and phosphates.

3.7 APPROVED SIGNATORIES

Designated laboratory staff members have the responsibility of validating laboratory documents on behalf of the laboratory organization. General categories and documents requiring a valid signature are presented below.

3.7.1 Client/Invoice Reports

All laboratory reports compiled and mailed contain at least one representative signature validating the contents of the laboratory report. By default, a report is signed by the appropriate project chemist. Alternate and/or additional signatures include the Laboratory President, Client Services Manager, Technical Director, Quality Assurance Manager, and Vice President of Laboratory Operations. No other individuals are approved to perform signatory approval of client/invoice reports.

3.7.2 Proposals, Price Quotations, and Laboratory Contracts

Proposals or price quotations for laboratory services contain at least one representative signature, validating the pricing, terms, and conditions of the quotation. At least one representative signature is required. Approved signatures for proposals and price quotations include the Laboratory President, Client Services Manager, project chemists, and a sales or marketing representative.

Required signatures for laboratory contracts are the Laboratory President and a Sales or Marketing representative.

3.7.3 Quality Assurance Project Plans (QAPP)

Quality Assurance Project Plans contain representative signatures of several responsible parties outside the laboratory. The only laboratory signature generally found on a QAPP is that of the QA Manager. The QA Manager has designated QA/QC responsibilities that are fully documented in QAPP

documents. All QAPPs are signed prior to submission to a governing body or client.

Signatures on the QAPP ensure all procedures, materials, quality control practices and project reports meet the predefined goals of the plan.

3.7.4 Purchase Orders and Agreements

Because the laboratory spends a significant portion of its annual budget on supplies and equipment, guidelines have been established to document and control purchasing.

Purchasing of general supplies is handled through a contracted vendor within the budgetary guidelines established for each laboratory area.

For major purchases such as equipment, service assessments, or building renovations in excess of $500.00, purchase orders or agreements must be approved by the Laboratory President or CEO.

3.7.5 Binding Statements - Laboratory Certification Documents or Accreditation

Many certification or accreditation programs require the laboratory to provide items and statements regarding details on the laboratory's operations and staff. In some cases these statements must be presented to the certifying body accompanied by a binding signature of the laboratory president or CEO.

3.8 CAPABILITIES, CERTIFICATIONS, ACCREDITATIONS, AND PROFICIENCY TESTING PROGRAMS

3.8.1 Capabilities

TriMatrix conducts analytical laboratory services in support of all major environmental regulations, including CERCLA, RCRA, CWA, CAA, and TSCA.

The laboratory is capable of routinely analyzing a variety of sample matrices, including drinking water, surface water, wastewater, soil, groundwater, solid waste(s), and sludge(s). In addition, analyses have been performed on fish tissue, biota, and air samples by project request.

TriMatrix routinely performs a wide array of environmental and non-environmental, chemical and physical analyses. A list of methods currently utilized by TriMatrix is provided in Appendix B. To maintain a quality system of analytical protocols, TriMatrix uses written Standard Operating Procedures (SOPs) derived from methodology specified by the United States Environmental Protection Agency, other federal and state agencies, and professional compendia.

When requested by the client, samples for analyses outside the analytical scope of TriMatrix can be subcontracted to another laboratory. Unless otherwise specified or required by the client, samples will be subcontracted to a NELAP accredited or ISO-17025 certified laboratory.

3.8.2 Laboratory Certification - Federal, State, and Independent

TriMatrix has been formally recognized for its commitment to quality. The laboratory maintains certification through various federal agencies, as well as several state regulatory agencies and private entities. As required by most of the programs, including NELAP and A2LA, certification and accreditation claims must be made in such a manner as to not imply certification or accreditation beyond that given on the laboratory's actual scope of accreditation. Generic certification or accreditation claims must not be made. The use of symbols (such as the A2LA symbol) and other forms of accreditation must always be analyte and/or method specific. Certification

programs in which TriMatrix currently participates are listed in the subsections below:

3.8.2.1 Federal Certification/Approval Programs

U.S. Army Corps of Engineers – DoD QSM

U.S. Air Force – AFCEE

U.S. Army Center for Health Promotion and Preventative Medicine – NELAC/A2LA

U.S. Navy – Navy (IR/QA – DoD QSM)

NELAP – National Environmental Laboratory Accreditation Program

3.8.2.2 State Certification Programs

Arkansas	Department of Environmental Quality
Florida	Department of Environmental Protection
Georgia	Environmental Protection Division
Illinois	Environmental Protection Agency
Kansas	Department of Health and Environment
Kentucky	Petroleum Storage Tank Environmental Assurance Fund
Louisiana	Department of Environmental Quality
Michigan	Department of Environmental Quality
Minnesota	Department of Health
New York	Department of Health
Ohio	Ohio VAP Program
Wisconsin	Department of Natural Resources

3.8.2.3 Independent Certification Programs

The American Association for Laboratory Accreditation (A2LA)

3.8.3 Proficiency Testing Studies

An integral part of most certification programs are Proficiency Testing (PT) Studies. PT studies are analyzed periodically as external "blind" or "double blind" spiked samples containing specific (known only to the administrators of the study) concentrations of target analytes. The laboratory reports the results to the agency or firm administering the PT study. The administrator then evaluates the laboratory's performance based on a comparison of the reported values with the known analyte concentrations. Laboratory results are scored and reports are prepared by the study administrator. The reports are submitted to the laboratory, certifying programs, and agencies or private entities that subscribe to the program.

TriMatrix routinely participates in the following proficiency testing programs:

- Water Supply (WS) Study
- Water Pollution (WP) Study
- Soil PT Study
- USEPA DMRQA

3.9 LABORATORY FACILITIES, EQUIPMENT, AND SUPPLIES

3.9.1 Physical Plant

3.9.1.1 Laboratory Demographics

The current TriMatrix Laboratories facility, located at 5560 Corporate Exchange Court SE, Grand Rapids, Michigan, was constructed in 1999. The 20,000 square foot structure was designed predominantly by the laboratory staff, with careful consideration given to the strict analytical testing requirements of today's environmental marketplace. Special attention was given to the sample preparation areas and the segregation of non-

compatible areas such as semi-volatile and volatile organics. Samples are stored according to type, with a large centrally located walk-in cooler used for the storage of all non-volatile, non-hazardous waste samples, to which both the sample receiving personnel and the laboratory staff have ready access. Quiet office areas were also built in, to provide space for data review, report compilation, and technical review discussions. A breakdown of each general area of analysis and the space allocated is as follows:

Laboratory Area	**Space Allotted, ft^2**
Wet Chemistry/Microbiology	Approx. 2000
Atomic Absorption/Emission	Approx. 2000
Volatile Organics	Approx. 1600
Semi-Volatile Organics	Approx. 2300
Sample Processing & Storage	Approx. 2400
Administrative Offices	Approx. 4200
Organic Pretreatment	Approx. 1300
Miscellaneous Space	Approx. 4200

The attached facility layout (Figure 3-6) shows the general lab areas and other space allocations.

Access to all laboratory areas including sample storage, sample container preparation, sample preparation, sample disposal, documents storage and clients files are secured. Non-authorized personnel may enter these areas only if escorted by a laboratory staff member.

Project initiation, sample control, and analysis, are all controlled using a Laboratory Information Management System (LIMS).

Under the direction of the Laboratory President, TriMatrix is organized into the following operating areas and support services.

Laboratory Administration

Client Services
Data Management
Sales/Marketing
Project Management
Health and Safety
Quality Assurance
Computer Services

Analytical Operations

Inorganic Laboratory
- Metals Laboratory
- Non-Metals Laboratory

Organic Laboratory
- Volatile Organic Laboratory
- Semi-Volatile Organic Laboratory
- Organic Extraction Laboratory

(Refer to Figure 3-2 for a graphical representation of the Laboratory Organization Chart)

3.9.1.2 Reagent Water Systems

Laboratory water originates from the Grand Rapids potable water distribution system. At the laboratory, the water is softened and passed through an activated carbon filter to remove residual chlorine. The water then enters a reverse osmosis system where approximately 90% of the dissolved constituents are removed. The water is temporarily stored in a 120 gallon holding tank until demand activates a mechanical pump that transfers the water through two mixed bed deionizing canisters. This water meets the requirements of ASTM Type II, and is utilized for glassware cleaning and as a feed-water to a variety of polishing systems.

The polishing systems are comprised of a distillation unit and a Milli-Q 4 Bowl System. Distilled RO-Deionized water is used primarily for BOD and metals analyses. Mill-Q water, which is equivalent to an ASTM Type I designation, is primarily used for the preparation of standard solutions and reagents.

Each water system is periodically monitored for specific quality requirements. Monthly, heterotrophic plate count and total residual chlorine analyses are performed. Weekly, the water system itself is checked for operational readiness and a hardness test is performed. Daily, additional readiness checks including a conductivity test are performed.

Responsibility for monitoring the TriMatrix reagent water systems is carried out by the Quality Assurance Department and personnel in the inorganic wet chemistry laboratory.

3.9.1.3 Ventilation Systems

The laboratory ventilation system was specifically designed to minimize or eliminate airborne contamination. Externally, the air conditioning unit intakes were located taking into consideration prevailing wind patterns, positioning them upwind of the fume hood exhaust stacks. Taking into account wind-shifts, the exhaust stacks were equipped with high velocity fans to disperse potential contaminants well above the building. Internally, the air-handling systems controlling heating, cooling, and humidity, also maintain maximum cfm air turnover. Additionally, the air-handling systems are monitored and controlled via a NOVAR computer controller.

3.9.1.4 Compressed Air

Compressed air must be free of dirt, water, and oil. Compressed air purchased from vendors is high purity grade (breathing air).

Compressed air produced in the laboratory uses filters at the compressor to remove water from the delivery lines. For the gas chromatographs and atomic absorption spectrophotometers, additional filters are located on the instrument to remove any residual oil at the point of use.

3.9.1.5 Electrical Services

The electrical system in use at TriMatrix was designed specifically for a laboratory environment. Special attention was paid to instrument requirements, including the isolation of separate lines for critical applications like GC, GC/MS, atomic absorption, and automated analyzers.

All laboratory benches, hoods, and work areas were designed with sufficient outlets to accommodate a variety of laboratory applications, such as distillations, digestions, and extractions.

Surge protection devices are in place for all laboratory computing equipment. The laboratory LIMS system is also protected by an Uninterrupted Power Supply (UPS). This UPS allows for a sequenced shutdown of the LIMS system during a power failure. This sequenced shutdown provides excellent protection of the LIMS database during a power interruption.

3.9.2 Equipment, Supplies, and Chemical Procurement; Reception, Storage, and Inventory

For an environmental testing laboratory where trace analyses are routinely performed, certain specifications for laboratory equipment, supplies, and chemicals are critical to quality. A minimum specification for accuracy and precision of equipment such as analytical instrumentation, balances, glassware, and water baths is required for each analytical procedure. The Technical Director in conjunction with the Laboratory President and laboratory area

managers are responsible for determining minimum specifications before equipment is procured. The analytical specifications are based on a detailed review of the test methods. Purchasing is coordinated through the purchasing department. Records are maintained on all vendors exhibiting poor performance on either their service or product. Relationships will be terminated with any vendor whose records indicate sub-standard performance.

3.9.2.1 Equipment Management/Maintenance/Inventory

A sufficient inventory of equipment is maintained to prevent testing delays resulting from equipment failure. Service is performed on equipment on a scheduled basis. A stock supply of spare parts that are known to wear out regularly is maintained.

Adequacy of equipment for its intended purpose must be verified before use. Maintenance logbooks are kept to document maintenance procedures on major equipment, allowing preventive maintenance frequency and requirements to be determined. Maintenance procedures are discussed in the various analytical SOPs.

A complete listing of Laboratory Equipment is presented in Appendix C of this manual.

3.9.2.2 Glassware

Only glassware providing the required precision is used for a particular analytical procedure. TriMatrix purchases Class A pipets, burettes, and volumetric flasks, to meet this specification. A standard operating procedure is utilized for cleaning each type of glassware. Cleaning of glassware is performed according to the analysis being conducted and the sample matrix involved, but certain general rules apply to all glassware washing procedures:

- Use hot water to wash away water-soluble substances.
- Use detergent, dichromate solution, organic solvent, nitric acid, or aqua regia to remove other materials according to the specific glassware cleaning procedures.
- Avoid using detergents on glassware to be used for phosphate determinations.
- Use ammonia-free water for ammonia and kjeldahl nitrogen analyses.

For all analyses, it is advisable to rinse glassware with tap water followed by deionized water immediately after use, as residue allowed to dry on glassware is more difficult to remove.

3.9.2.3 Reagents, Solvents, and Gases

Purchasing of reagents, solvents, and gases are carefully controlled through an ordering system that maintains a minimum level of quality in the testing process. The Quality Assurance Department defines the suitable grades of ordered materials. Designates from each laboratory area verify upon receipt that incoming materials meet these requirements. Certificates of Analysis are forwarded to the Quality Assurance department where they are scanned and stored. Each laboratory area will monitor the proper storage and the eventual removal of reagents, solvents, and gases, when their shelf life has expired. All consumable reagents and chemicals must be labeled with the date received to ensure a First-In-First-Out (FIFO) system of use.

Reagents, solvents, and gases are available from vendors in a broad range of purity, from technical to ultra pure grades. The analysis, as well as the sensitivity and specificity of the method, must be considered when choosing a grade. Analytical reagent (AR) grade is suitable for most inorganic analyses. Trace organic analyses frequently require ultra pure grades. AR grade is the minimum

approved for reagents used in organic analysis. The absence of certain impurities is required for some GC detectors - notably sulfur and phosphorus in an FID detector. Trace metals analyses including atomic emission and atomic absorption spectroscopy usually requires spectro-quality reagents, although AR grade may be suitable in some cases. Florisil, silica gel, and alumina used as absorbents in organic extract cleanups, must be checked for interfering components and activated according to the analytical method. Compressed gases are available in various purities, usually expressed as a percent (e.g. 99.999). Gases are filtered in the laboratory delivery lines to remove moisture, oil, and other contaminants. Refer to the analytical method and instrument manufacturers operating manual for gas purity requirements.

Provided they are available, expiration dates of unopened chemicals are based on the date determined by the manufacturer. They may also be derived from the analytical method. A new expiration date may be required once the chemical is opened. The following guidelines are utilized in assigning expiration dates:

Unopened Reagents, Solvents, and Neat Chemicals
Manufacturers assigned expiration date or 5 years from date received, whichever occurs first.

Opened Reagents, Solvents, and Neat Chemicals
2 years from date opened or remainder of manufacturers assigned expiration date, whichever occurs first.

Prepared Solutions - Stock
Manufacturers assigned expiration date or 1 year from date opened, whichever occurs first.

Prepared Solutions - Working

Assigned expiration date of stock, or 6 months from date prepared, whichever occurs first.

Unpreserved ethers have an expiration date of 34 days due to the potential for peroxide formation.

In order to maintain expiration date accuracy it is critical that the date opened is recorded on all containers, and the expiration date originally entered into the LIMS system be updated based on the date opened.

3.9.2.4 Certified Standards

The purity and traceability of standards used in the analytical process is crucial to the quality of the data generated. Only high quality standards certified by established vendors are to be utilized. Calibration standards must be of the purity required by the method for a particular analysis.

Upon receipt all purchased standards are entered into the LIMS system and labeled with a unique identifier and an expiration date. The date received is also recorded on the container. Stock and working standards are likewise labeled.

All calibration standards are validated against a second source standard. A second source standard is analyzed with every initial calibration. The quantitated value is compared to laboratory established limits. Recovery must fall within these limits for the calibration and calibration standard to be considered acceptable. Stock and working standards are also monitored for visible signs of deterioration (precipitates, color change, volume change).

Vendor expiration dates for purchased stock standards must not be exceeded. Expiration dates for laboratory prepared standards are

based on guidelines in the analytical method, generally 6 months for working, and 1 year for stock standards.

3.9.2.5 Chemical / Reagent Storage

Bulk chemicals and reagents are stored in a several locations and under a wide variety of conditions within the laboratory. Specific storage conditions for many reagents are presented in each laboratory testing SOP. Additional storage information is referenced in both the TriMatrix Laboratory Safety Manual and the TriMatrix Chemical Hygiene Plan. For general purposes, the following storage conditions are used:

Chemical /Reagent Type	General Storage Requirements	Location/Lab Area
1) Bulk Dry Chemicals	Dry Chemical Storage Cabinets	Inorganic Laboratory
2) Inorganic Acids	Vented Acid Storage Cabinets	Metals Laboratory
3) Organic Solvents-Flammable	Vented Flammable Cabinets	Inorganic & Prep Laboratory
4) Organic Solvents-Nonflammable	Vented Storage Cabinets	Inorganic & Prep Laboratory
5) Compressed Gases	Secured Gas Storage Area	Garage & Outside Storage
6) Bacteriological Materials	Reagent Refrigerator	Inorganic Laboratory
7) Aqueous Standards	Reagent Refrigerators	All Laboratory Areas
8) Organic Standards-Flammable	Explosion Proof Refrigerators and Freezers	Organic Laboratory Areas
9) Organic Standards-Nonflammable	Standards Refrigerator & Freezers	Organic Laboratory Areas
10) Sample Extracts	Extract Freezers	Organic Laboratory Areas
11) Digestates-Metals	Vented Acid Storage Cabinets	Metals Laboratory

3.10 TRAINING

Proper training of laboratory personnel is an essential part of staff development. Training procedures include documentation of training activities completed and serve as a guideline for continual staff development. All testing personnel must familiarize

themselves with the laboratory's training procedure (TriMatrix SOP GR-10-109) and implement all associated policies and procedures.

Personnel files contain the training documentation related to the development of each laboratory employee. Included are in-house training, external training certificates, safety training, ethics training, and other materials specific to the analyst. The quality assurance department maintains the training file system.

3.10.1 Training Orientation

The human resources department initiates training orientation for each new employee on the first day of employment. Orientation includes completion of various training checklists (Appendix D). These checklists provide documentation of the orientation after being signed by the new analyst and the trainer and become a part of the employee's permanent training record.

3.10.2 Code of Ethics/Data Integrity Training

It is the intent of TriMatrix Laboratories, Inc. to consistently report data of the highest quality. For this to be possible, analysts are instructed in accordance with the level of data quality desired and are provided with an environment conducive to its achievement. Besides providing the analyst with all necessary supplies and equipment, the work environment is maintained as free from undue pressures as possible. Such pressures may be through internal peer pressure or deadlines, or through external customer complaints or priority requests. It is the responsibility of management to insulate the analyst from such pressures as much as possible. Data quality cannot be compromised without reason and the analyst will not be reprimanded for adhering to established quality protocols in the face of such pressures.

During the orientation with human resources, these policies will be explained and the employee asked to review and sign a Code of Ethics/Data Integrity Policy Agreement (Appendix E). This agreement documents the understanding between management and the new employee concerning management's

position on data quality, sample analysis and data reporting, and the consequences of improper actions. The signed agreement is retained as part of the employee's permanent record.

3.10.3 Document Storage

All essential laboratory documents are stored on the laboratory's intranet drive. During orientation, the new employee is shown how to access these documents and instructed on which ones are required reading. These include the Quality Assurance Manual, Chemical Hygiene Plan, Safety Manual, Employee Handbook, a memo containing instructions on TriMatrix error correction policies and standard operating procedures. Forms are signed documenting that the employee has read and understood these documents.

3.10.4 Demonstrations of Capability (DoC, IDC, CDC)

All analysts and instruments used for sample analysis must complete at least one type of Demonstration of Capability (DoC). Three types of demonstrations exist: a method/instrument DoC, an analyst Initial Demonstration of Capability (IDC) and an analyst Continuing Demonstration of Capability (CDC). All demonstrations of capability are documented, reviewed, and signed in accordance with the TriMatrix SOP for analyst training (GR-10-109). All supporting data necessary to reproduce the DoC, IDC, or CDC must be available. Sample analysis may not begin without the successful completion of an appropriate DoC and submission of all associated paperwork to the Quality Assurance Department.

3.10.4.1 Demonstrations of Method Capability

Prior to the acceptance and institution of any Standard Operating Procedure or the use of any new instrument, a satisfactory demonstration of method/instrument capability study is required. This DoC must be performed on all instruments used for the analysis. This is a one-time study, unless there is a significant

change in the instrument or methodology. This procedure must be successfully completed for all applicable matrices prior to sample analysis. The instrument DoC consists of a Demonstration of Accuracy and a Method Detection Limit Study (MDL); two separate studies demonstrating the instrument's capability of producing sufficiently accurate and sensitive results.

1) Demonstrations of Instrument Capability

For instruments that utilize an initial calibration, an acceptable initial calibration will serve as the demonstration of capability. The low point of the calibration must be at or below the lowest desired reporting limit. The high point defines the calibration range. Any sample with an analyte concentration above the high point in the calibration requires a dilution before quantitation. For procedures not using a calibration curve, seven standards at various concentrations covering the range of the analysis must be analyzed to demonstrate accuracy throughout the range. These standards must be prepared from the same source as that used for calibration. The relative standard deviation of the average recovery must be less than 20% and average percent recovery must be between 95 - 105%. The spreadsheet in Appendix F and the form in Appendix G must be completed to document the accuracy test. Return these completed forms to the Quality Assurance Department.

2) Method Detection Limit study (MDL)

A Method Detection Limit (MDL) study is performed in accordance with TriMatrix SOP GR-10-125. MDL studies must be completed for each matrix-specific preparative and/or analytical technique and must be updated annually or whenever a major change is made to the preparative and/or

analytical technique. The MDL procedure is described in section 3.11.2.

3.10.4.2 Initial Demonstrations of Analyst Capability

After orientation and training, each analyst must complete a successful IDC study. The IDC, unlike the DoC, is not instrument dependent. An IDC must be completed any time a significant change to a procedure occurs. Conduct the IDC study by preparing four replicate blank spikes (for any procedure with a pre-treatment) or four replicate second-source calibration verifications (for any procedure without a pre-treatment) at a concentration in the lower half of the calibration or analytical range. In either case, the spiking standard must be prepared from a source other than that used for calibration. For analyses where a spiking standard is not an option, the acceptable analysis of a single blind PT sample will suffice. Alternatively, the analyst may analyze four replicates of a client sample against four replicates of the same sample analyzed by an experienced analyst for statistical comparison.

Process the four spikes, PT sample, or replicates, following every step in the preparative and/or analytical procedure concurrently or over a period of no more than 72 hours. Enter all four results into the IDC spreadsheet (Appendix H) or all eight replicates into the IDC spreadsheet (Appendix I) as appropriate. The spreadsheet will calculate average percent recovery and relative standard deviation then evaluate against default acceptance criteria (which may need changed to fit the procedure). If all acceptance criteria pass, the analysis of actual samples may begin.

When one or more analytes fail any criterion, the study is unacceptable for the failed analyte. Locate and correct the source of the problem then repeat the study for the failing analyte successfully. If none of the options presented above are possible

(such as with the TCLP pre-treatment), the analyst must perform and submit an acceptable method blank with acceptance being that all analytes are at or below the method detection limit.

When complete, forward the IDC spreadsheet, the NELAC Demonstration of Capability Certification Statement (Appendix J), the Laboratory Training Checklist (Appendix K), the MDL study when necessary (Appendix L), and/or PT results to the Quality Assurance department for review and training documentation.

3.10.4.3 Continuing Demonstrations of Analyst Capability

A Continuing Demonstration of Capability (CDC) is required annually. In addition to the IDC study described in section 3.10.4.2, the CDC may be accomplished by inputting the last four results of an MDL study to the IDC spreadsheet if completed exclusively by the analyst, by inputting four *consecutive* SCV results obtained during the course of routine sample analysis if completed exclusively by the analyst or by exclusively running a PT study analysis successfully.

When complete, forward a copy of all applicable data necessary to reconstruct and validate the study to the Quality Assurance department for training documentation.

3.10.4.4 SOP Revision Checklist

SOPs are periodically reviewed and updated. When an update is released, the appropriate form from Appendix M must be completed to record that the applicable analysts have read, understood and agree to follow the revised SOP.

3.10.5 Continuing Training and Education

TriMatrix Laboratories, Inc. is committed to education and training on a continual basis for employees. There are various ways in which continuing education may occur, including:

- seminars
- cross-training for additional job responsibilities
- retraining
- method and technology updates

3.11 DETECTION LIMITS

The process of quantifying an analyte in an environmental matrix using specific analytical procedures must use detection limits as a point of reference. The three levels of analytical detection are described below.

3.11.1 Instrument Detection Limit - IDL

Most analytical instruments produce a signal even when a blank (matrix without analyte) is analyzed. This signal is referred to as the noise level. The IDL is the analyte concentration required to produce a signal greater than three times the standard deviation of the instrument noise level. The IDL can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. If the instrument does not give a signal for the blank, perform the study using standards at the expected IDL concentration. Each measurement should be performed as though it were a separate analytical sample followed by a rinse and/or any other analytical step normally performed between the analysis of separate samples. Where required by the method (for example, SW-846 method 6010B), IDLs need determined at least every three months or at a project-specific designated frequency and kept with the instrument.

The IDL only defines an instrument's limitations and does not take into consideration sample processing in preparation for the analysis. As such, it may not be used to estimate the method detection limit (Figure 3-7).

An IDL study is required only when specified by the analytical method reference.

The requirement for performing an IDL study does not negate the requirement of a method detection limit (MDL) study.

3.11.2 Method Detection Limit - MDL

Many times there is more to an analysis than a direct analysis such as a digestion, dilution, concentration, chemical treatment, extraction, and cleanup. Additional analysis steps propagate error from the uncertainty associated with each step. Since the method detection limit is defined in terms of error, all steps leading to analysis must be included to calculate the MDL (Figure 3-7).

The MDL is defined as the minimum concentration of a substance that can be detected and reported with 99 percent confidence (statistically) that the value is above zero. The MDL is calculated from spiked blanks which go through the entire sample preparation and analysis scheme. MDL studies are run for aqueous and solid methodologies for every analyte targeted. Although MDL studies are completed for all laboratory determinations, if any result were to be obtained without an associated MDL study, it must be reported as estimated. All calculated MDL values must be verified.

The MDL procedure used at TriMatrix Laboratories references 40 Code of Federal Regulations, Part 136, Appendix B where seven replicate aliquots of laboratory reagent water (for an aqueous methodology) are spiked with every analyte of interest at the estimated minimum practical quantitation limit (PQL). For a solid methodology, an inert substance or empty vessel is spiked. The PQL may be estimated using instrument noise, a series of method blanks, the

instrument calibration, or the preliminary MDL estimation as described in TriMatrix SOP GR-10-125.

It is essential that all sample preparative, cleanup, and analytical steps be included in the MDL study. Calculate MDL study results based on all computations required to achieve the final result in sample-designated units.

To calculate the MDL, input all seven results to the MDL spreadsheet located on the laboratory intranet library. The spreadsheet calculates the MDL by multiplying the standard deviation by 3.143 which is the one-sided t-distribution for seven samples (with six degrees of freedom) for a 99% confidence interval. There must be no zero percent recoveries in the dataset and the concentration spiked must be between 1 and 5 times the MDL value.

Repeat the study at a lower concentration if results in the MDL spreadsheet are flagged "Fails Too Good". Repeat at a higher concentration if the MDL value is flagged "FAIL". However, if the spiking concentration is chosen based on a good estimate of the actual MDL, the MDL study should not fail. Re-estimate the actual MDL based on the failed MDL value before repeating the study.

Note: Even if the MDL value appears acceptable, the MDL procedure is not complete until an MDL verification also has been successfully performed.

The MDL verification is accomplished by analysis of a method blank and blank spike. Prepare the blank spike at a concentration between 1 – 4 times the calculated MDL value. If the blank spike response is greater than or equal to three times that found in the method blank, the MDL verification passes and the calculated MDL value is acceptable.

If the blank spike response is less than three times that found in the blank, the MDL value is too low. Repeat the MDL study by estimating the concentration necessary to produce a response equal to or greater than three times the method blank and repeat the verification study. Repeat the MDL verification. Repeat

until the MDL verification is at least three times the method blank. Only the MDL value or MDL verification that passes the MDL verification criterion may be used as the calculated MDL.

Appendix L shows an example of the MDL spreadsheet used to calculate and verify MDL values and practical quantitation limits.

The MDL for all analytes in aqueous and solid methodologies must be determined annually or whenever a significant modification is made to the procedure.

3.11.3 Minimum Practical Quantitation Limit - PQL

The PQL is defined as the minimum concentration of an analyte that can be quantitatively reported (versus qualitatively detected) within specified precision and accuracy limits under normal laboratory operating conditions. (Figure 3-7).

The minimum PQL is the analyte concentration spiked in the MDL study or 3 times the concentration spiked in the MDL verification when the initial MDL value fails. The minimum PQL must be 3-10 times the MDL value.

Note: Practical quantitation limits actually achieved for any given sample analysis will be highly dependent on the matrix and/or required dilutions.

3.12 PROCEDURES FOR ACCEPTING NEW WORK/TESTS

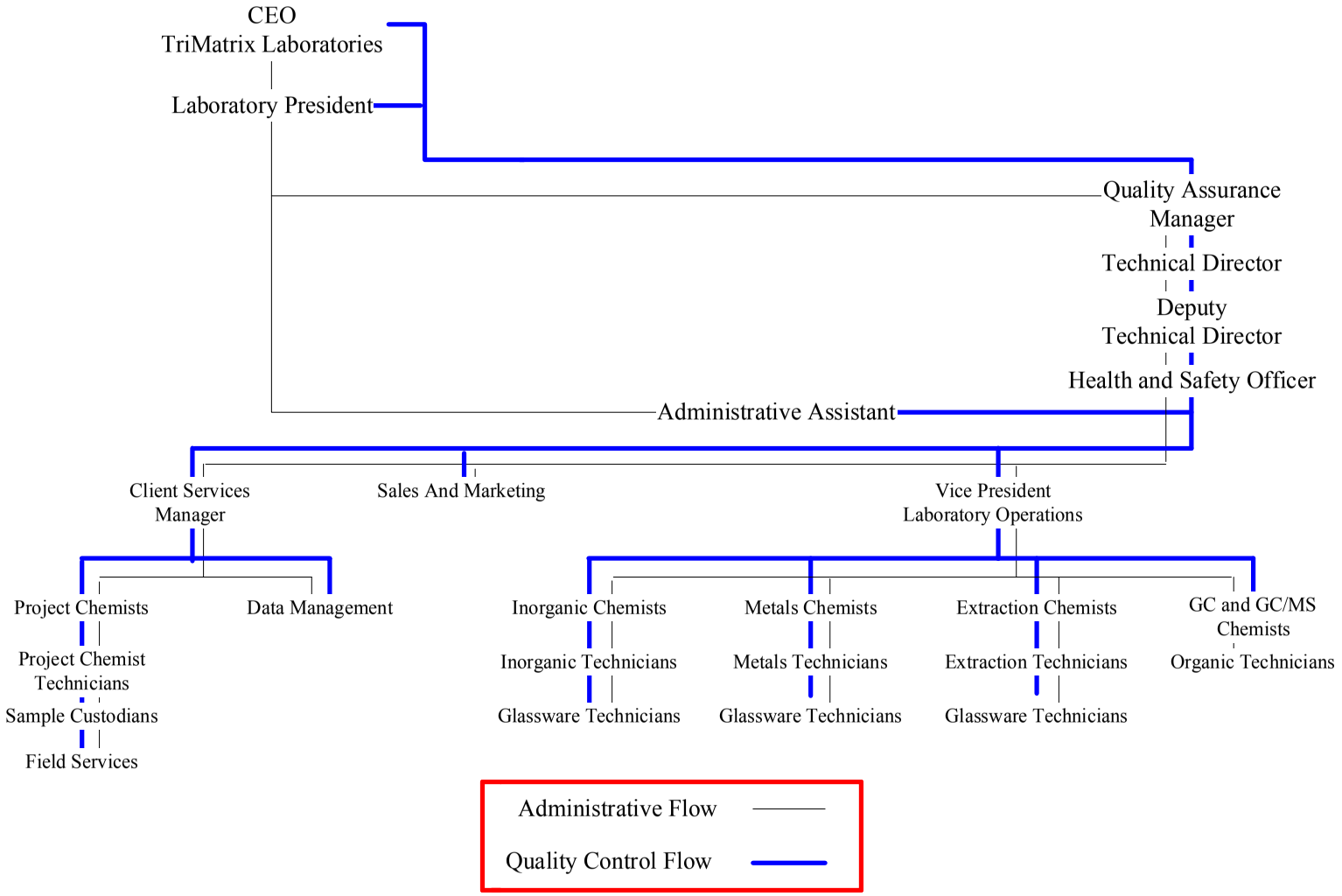
3.12.1 New Test Requests, Development, and Approval

Client Services must submit a request for new analyses to each impacted laboratory area where the request will be formally processed. Evaluation of the request will include the suitability of the analyte for quantitation, availability of existing test methods, instrumentation, capacity, standard materials, etc. The

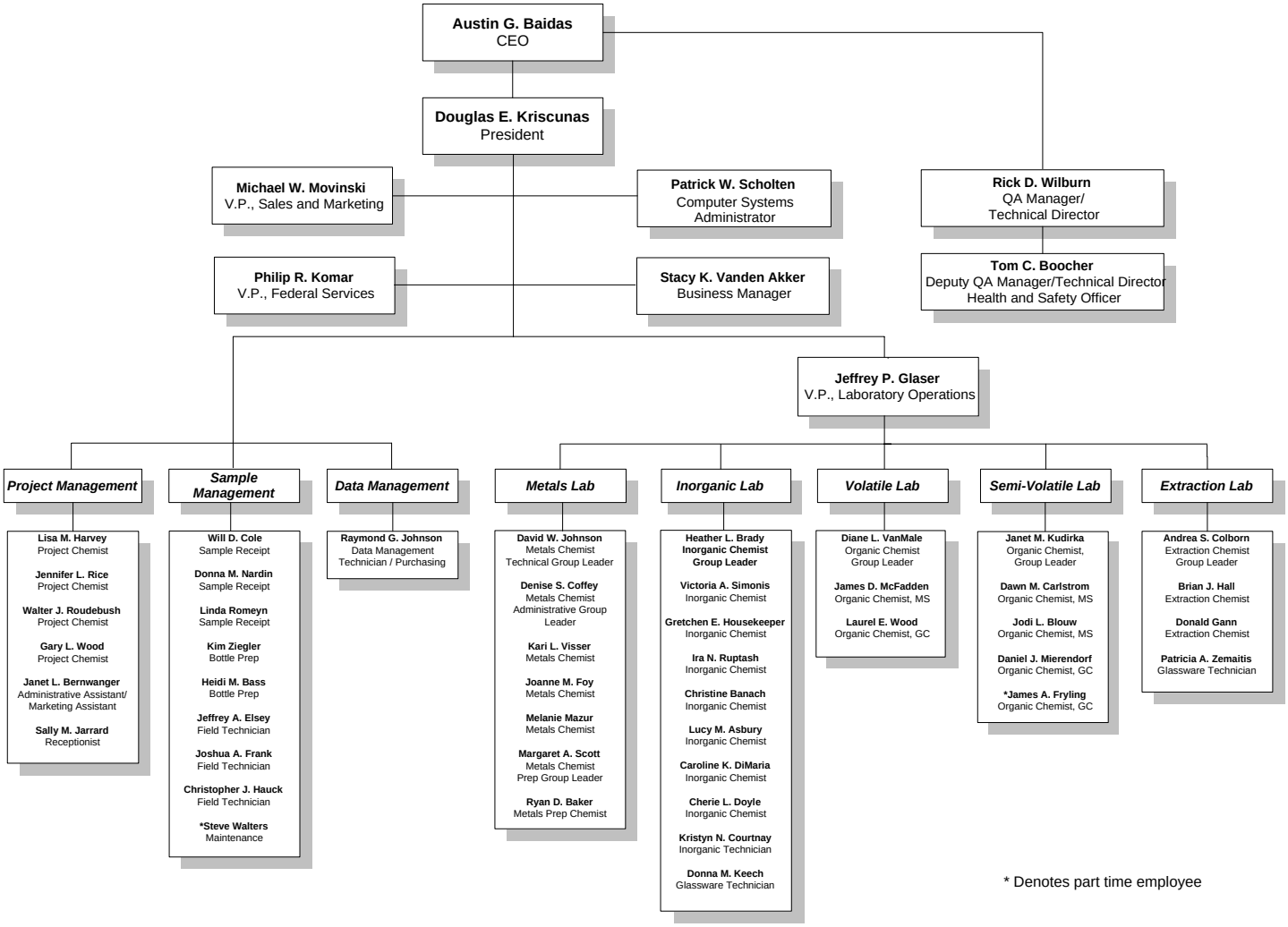
Vice President of Laboratory Operations, Technical Director, and/or Group Leader will provide a prompt response to client services to ensure client needs can be addressed.

All newly developed procedures are reviewed by the laboratory Technical Director and must comply with all requirements outlined in section 3.10.4.

Figure 3-1
Quality Control Chain of Command Flow Chart



TriMatrix Laboratories, Inc.



* Denotes part time employee

Figure 3-3
RELATIONSHIPS
Management to Technical Services

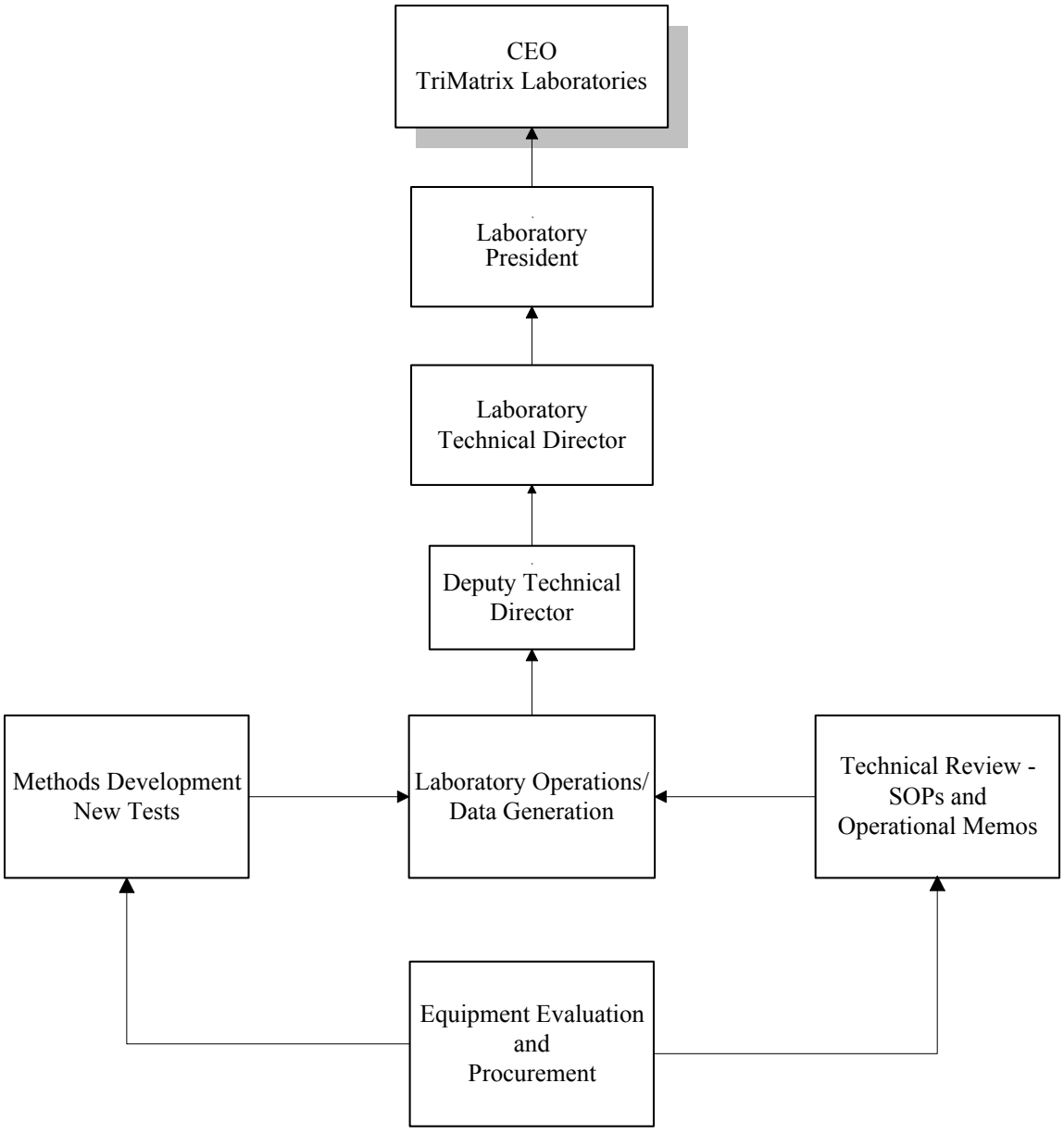


Figure 3-4
RELATIONSHIPS
Management to Support Services

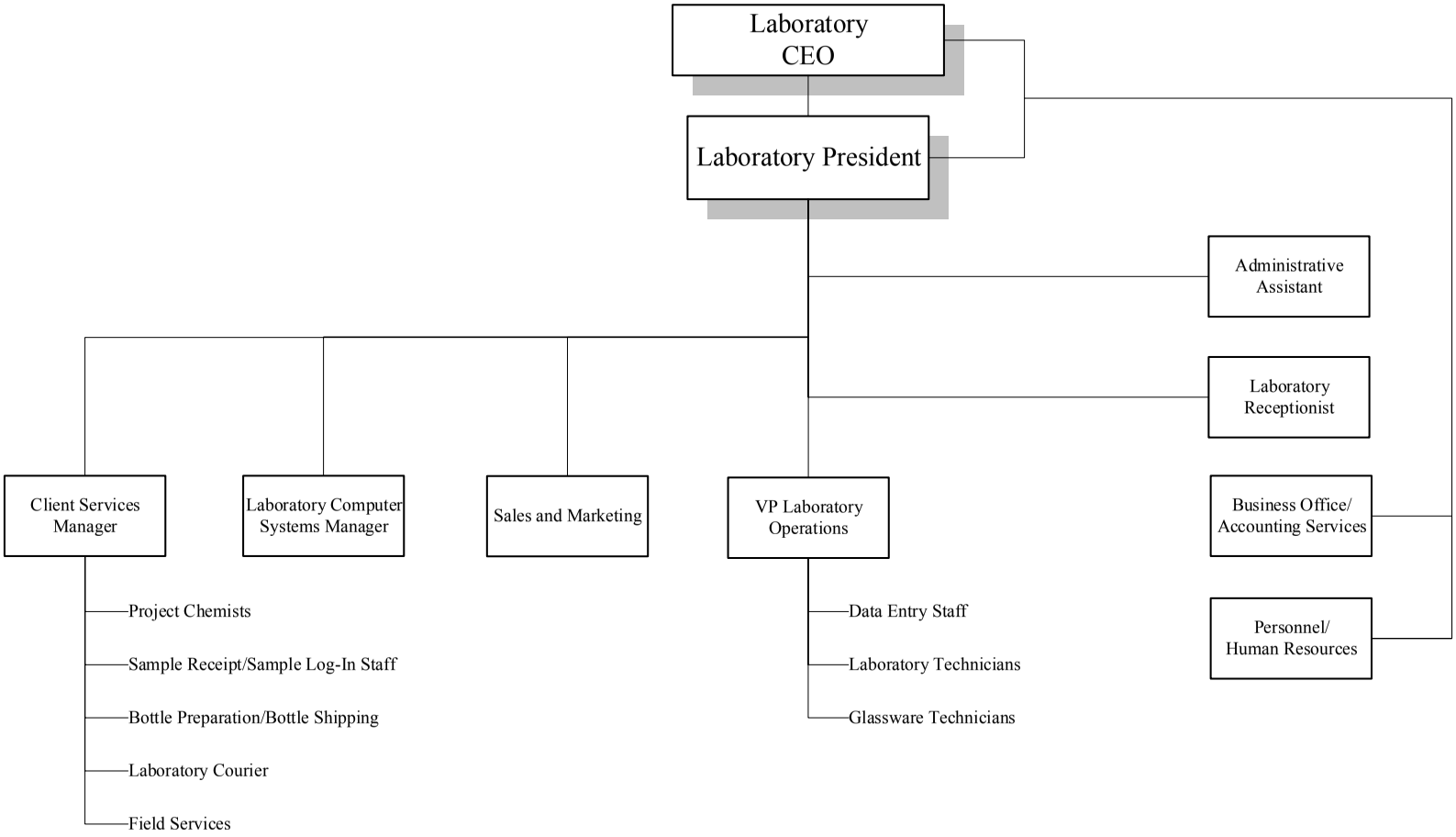


Figure 3-5
RELATIONSHIPS
Management to Quality System

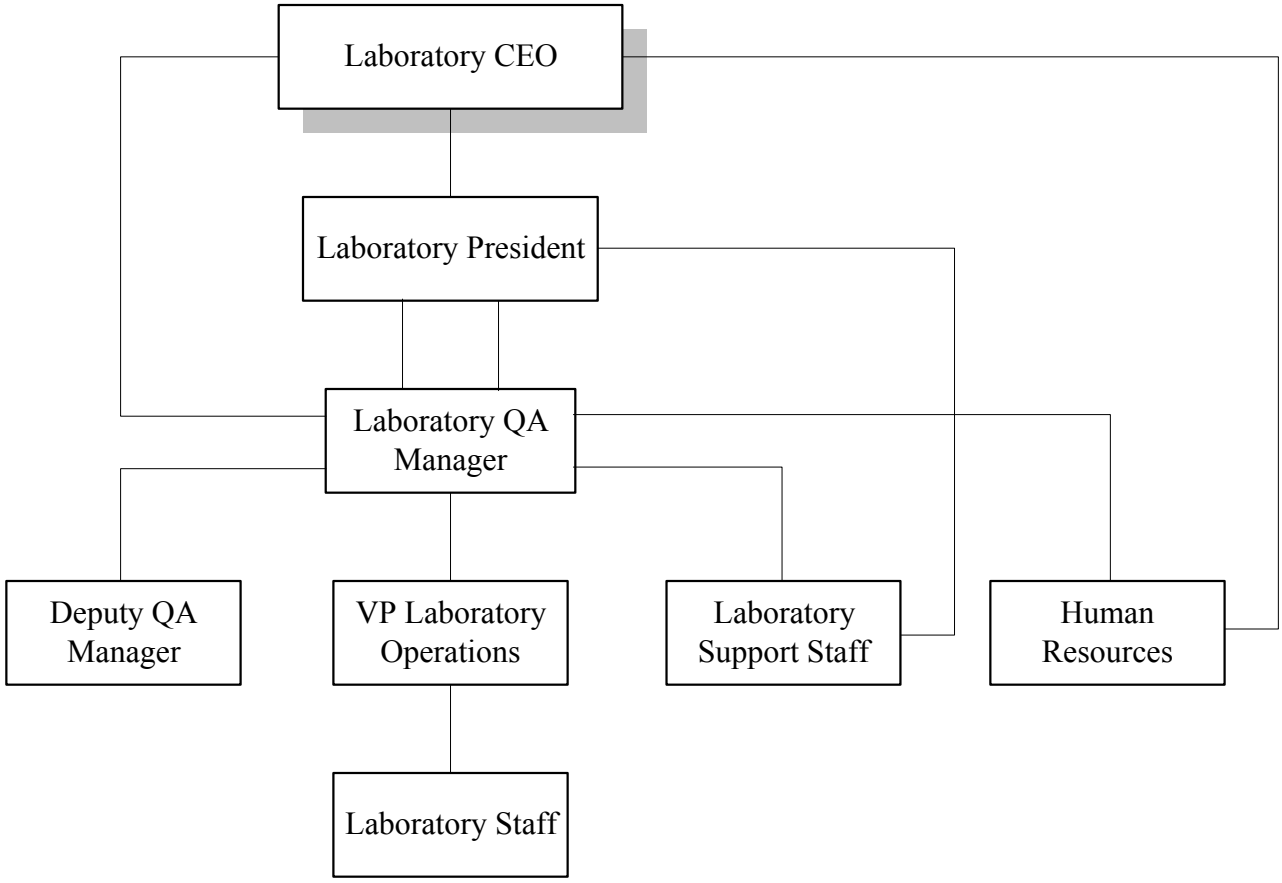


Figure 3-6
Laboratory Layout/Diagram

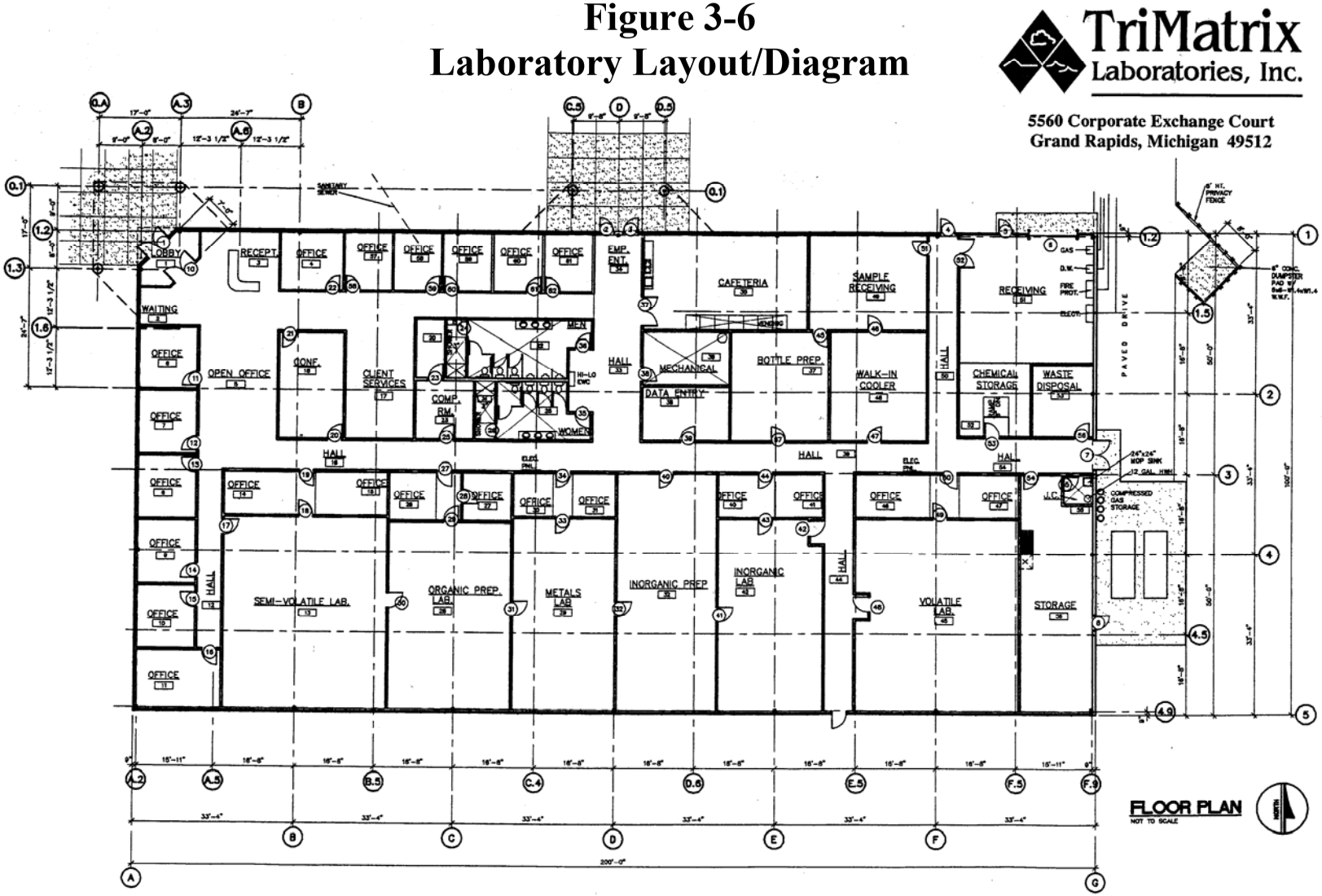
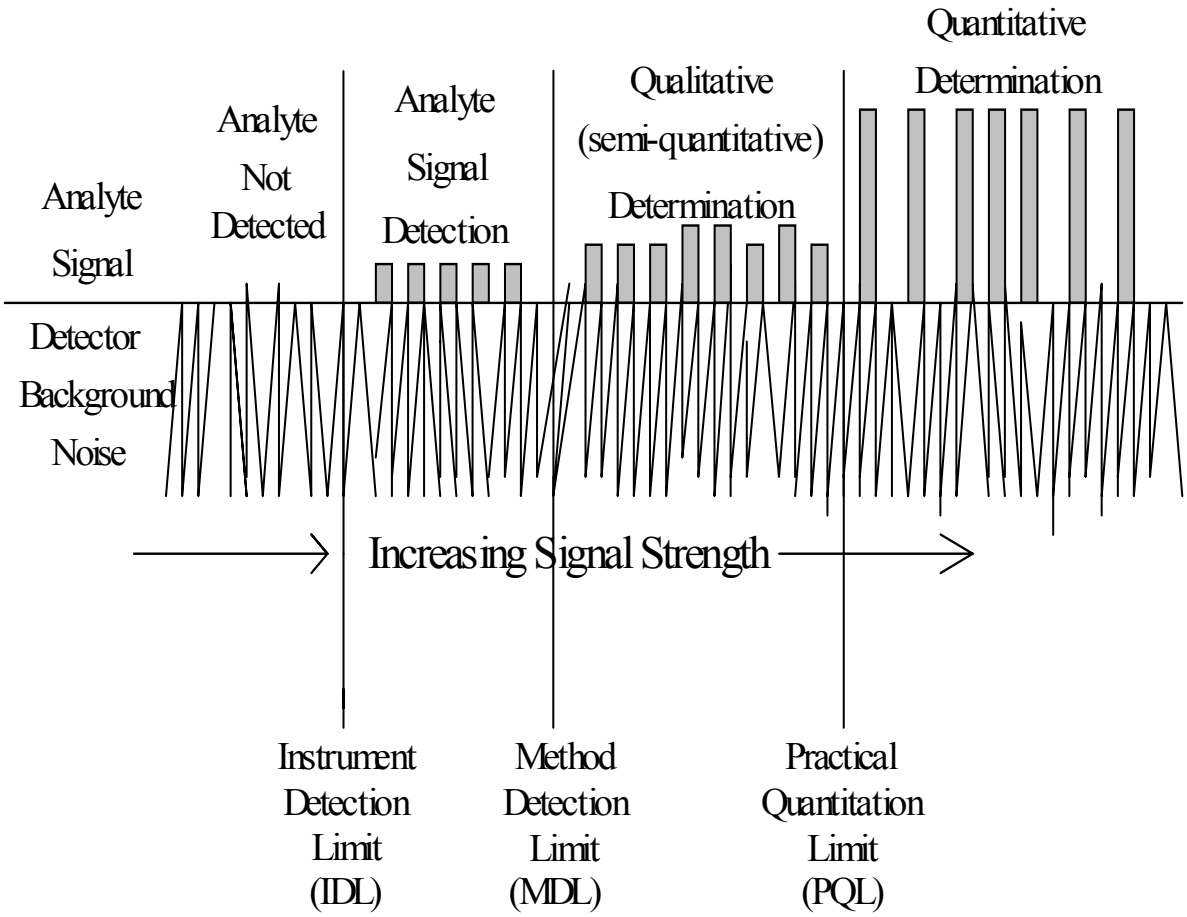


Figure 3-7
Regions of Analyte Signal



4.0 QUALITY CONTROL

4.1 DOCUMENT CONTROL AND MAINTENANCE

4.1.1 Procedures for the Control and Maintenance of Documentation

Documents utilized in the quality system are subject to strict control regarding their creation, revision, approval, use, and distribution. This applies to documents generated both internally, and those received from outside sources. Obsolete documents that are retained in circulation for either legal or knowledge preservation purposes are marked as "obsolete". The structure of the documentation used in the TriMatrix quality system is presented in Figure 4-1.

4.1.1.1 Internal Documentation

Examples of internal documentation include Standard Operating Procedures, the Quality Assurance Manual, miscellaneous forms, and logbooks. All documents must be reviewed and approved by one or more senior staff prior to their use. All documents will print with both the file name and revision number. Where possible, the document will contain the TriMatrix logo. All logbooks must be bound and paginated.

All approved documents are stored on the laboratory intranet read only drive designated as "Library." Document control is maintained through the use of the laboratory computer network. By maintaining only the current version of an approved document on the Library drive, document control and security is maintained. This procedure provides immediate access to the latest revision of all documents.

Document revisions may be made by any applicable, qualified, laboratory employee. Minor document revisions, such as those required to a Standard Operating Procedure, may be made by hand.

All hand amendments must be legible, dated and initialed, and recorded in ink. All hand amendments must be approved by, and distributed through, the Quality Assurance Department. Hand amendments will be incorporated into the next revision of the document. Hand amendments cannot be used for major document revisions. Extensive revisions require a formal document update.

Some documents, such as the QAM, require periodic reviews. The QAM is reviewed annually and updated as necessary.

Completed logbooks are numbered, scanned, and the resulting .pdf file stored on the Library drive.

4.1.1.2 External Documentation

Examples of external documentation include regulations, analytical methods, QAPPs, and client required standards. These documents are maintained by the quality assurance department. When possible, the documents are stored electronically on the Library drive. Instrument manuals are controlled by the individual laboratory areas.

4.1.2 Traceability of Measurements/Documentation Requirements

A properly designed and implemented documentation protocol will assure that all information presented in an analytical report can be traced back to its point of origin. The documentation protocol must also provide for traceability of non-reported information used to provide supporting value to the analytical report. These items include but are not limited to: stock standard records, test calibration records, data reduction and validation activities, sample custody, facilities monitoring, and final data reporting.

A more detailed review of the documentation procedure and traceability of information is presented in the following sections.

4.1.3 Paperwork/Information Flow

As displayed in Figure 4-2, document flow remains constant regardless of the quality control requirements of the sample. The general axiom is, a COC procedure will fail without a pre-existing scheme of rigid documentation control available. The records trace can provide for the following:

- Answers to questions of analytical integrity
- Assistance in finding and solving random and systematic problems
- Assistance in preventing long term degradation of the analytical process
- Assistance in ensuring continuity of analytical effort despite personnel and mechanical changes

The following subsections identify and describe the procedures followed, and the corresponding documents generated, from project initiation through completion.

4.1.3.1 Project Initiation

All samples or sample groups entering the analytical process must be accompanied by the appropriate documentation. This documentation is necessary to define the analytical goals and project objectives. Information concerning analytes, reporting limits, and reporting formats must be provided. An inventory of required sample containers must be prepared for each sampling event. This inventory is documented using the Container Packing List (Appendix N).

All projects are initiated through the LIMS system. All documents created during the project initiation phase are maintained and archived to the client filing system.

4.1.3.2 Sample Receipt/Examination

The receipt of all sample shipping coolers (empty or full) will be documented in the Sample Receipt Record Logbook (Appendix O). This logbook documents the delivery method, date and time, number of coolers received, client, and the name of the TriMatrix employee who received the cooler. This information is entered into the logbook immediately after drop-off.

Observations on the receipt of each sample delivery group, including sample temperatures, are documented on the "Sample Receiving/Log-in Checklist" (Appendix P). This form was designed in a step-by-step format to walk the log-in technician through all the steps required when receiving and logging-in samples. A supplemental "Sample Receiving/Log-in Checklist Additional Cooler Information" form is available when receiving projects consisting of more than four coolers (Appendix Q).

Additional forms to document sample preservation, "Sample Preservation Verification Form" (Appendix R), and non-conformances, "Sample Receiving Non-Conformance Report" (Appendix S), are also completed.

4.1.3.3 Sample Log-In

During log-in, a series of computer entry functions are performed in an effort to document and validate the log-in process. The remainder of the Checklist is also used to record the completion of the various steps that must be followed when logging samples into the LIMS system. Once complete, bottle tags are produced and a Work Order generated (Figure 4.3 and Appendix T). The log-in technician will initiate a project or submittal file for each sample delivery group received. This file is labeled with the LIMS system generated project-submittal sequence, and will contain all documents associated with the sample receiving/sample log-in process. These documents will include: all external chain-of-custody forms, sample preservation records, shipping records, any

client correspondence, and a copy of the actual log for each submittal. Upon completion of the analytical process, the project file becomes part of the permanent record of each project.

4.1.3.4 Worklists/Benchsheets

The worklists and benchsheets produced by the LIMS system are designed to provide the analyst with essential project information. This information not only includes client/project specifications, but also provides an avenue for communication of test specifications and parameter expiration dates and times. This up-front information enables the analyst to make vital decisions in their analytical scheme, and helps to minimize problems after samples are analyzed.

Examples of a laboratory worklists and benchsheets are presented in Appendix U.

4.1.3.5 Management Reports

Several reports are provided within the TriMatrix Laboratory system to help monitor operational conditions of the laboratory. These reports include: workload reports, on-time reports, and aging logs.

The flow of information from these various reports is geared to a variety of personnel within the management structure of the laboratory, and to specific persons outside the laboratory. Information is generally provided to employees' external of the laboratory for corporate management decisions or in providing information to a particular client about their project.

Examples of management reports are presented in Appendix V.

4.1.3.6 Quality Assurance Reports

Quality assurance reports play a vital role in the management of the quality system. Quality systems must be closely scrutinized in order to monitor, maintain, adjust, and add, procedures or systems to meet existing and new QA objectives of the laboratory.

Several quality assurance reports are created in this effort. These reports serve different functions and are designed to inform the ultimate user. In the case of a client/invoice report, the quality assurance data is presented to facilitate the objectives of the project requirements from data assessment through full 3rd party data validation.

Quality control reports are also used within TriMatrix to monitor the analytical process and to provide a means by which the analytical process can be viewed over time. Examples of efforts available for this monitoring process are presented in Appendix W.

Quality control reports are used extensively by the laboratory to access the analytical process. Many of these reports are utilized daily to monitor, for example – method accuracy, precision, completeness, and to provide the means for overall data assessment at the batch level. All QC reports are created through the LIMS system.

4.1.3.7 Project Files

The project file is the comprehensive record of every analytical project completed at TriMatrix. Project files are stored in secure filing cabinets. Items typically retained in a project file include:

- Initial project report/analysis plan/proposal
- All correspondence or documents mailed or received with the samples
- Written record of client phone conversations

- All sample receiving/log-in forms
- Chain-of-custody forms
- Laboratory worksheets
- Copy of the invoice

To save paper and file space, electronic, rather than paper, copies of final reports are typically retained, and can be regenerated on demand.

By default, project files are stored on-site for 1 year, followed by off-site storage at a secured limited access facility for an additional 6 years. Length of storage requirements are determined on a client/project specific basis. If the ownership of the laboratory changes, record storage will become the responsibility of the new owner. In the event the laboratory was to go out of business, each client will be contacted for instructions on record disposition. Client records will be transferred or destroyed as instructed.

4.1.3.8 Quality Control Documents

A) Instrument Logbooks

Two different instrument logbooks are maintained, an instrument run-log and an instrument maintenance log. Each log plays an important role in the documentation of daily instrument activities.

The Instrument Run Logbook is used to document all analytical determinations of a designated instrument. These determinations include not only sample analyses, but also recordings of all calibration and calibration runs, quality control analyses, and where applicable, any instrument tuning activities.

The Instrument Run Logbook also provides a chronology of each day's analyses. This chronology plays an important role in the data validation process. All run logs are identified by instrument

manufacturer name, model number, serial number, and the starting and ending dates encompassed. All completed run logs are issued document control numbers, inventoried, and properly archived.

The Instrument Maintenance Log is used to document instrument maintenance procedures, repairs, or modifications. All activities are documented by recording what was done, by whom, and why.

All completed maintenance logs are identified by instrument manufacturer name and model number, instrument serial number, and the dates encompassed. All maintenance logs are issued document control numbers, inventoried, and properly archived.

B) Controlled Temperature Units (CTU)

Each oven and incubator used for sample processing, and all cold sample and standard storage devices have their temperatures monitored and recorded on a daily basis. Within each CTU is a certified thermometer. Additionally, each CTU used for sample storage, and incubators used for BOD and bacteriological incubation, have their weekend temperature monitored via electronic data loggers. The calibration of liquid and digital thermometers is verified annually.

All temperature readings and thermometer calibrations are recorded in a CTU Logbook. This logbook contains a page for each unit with detailed information on unit identification, serial number, laboratory location, and designated operating temperature. All CTU logbooks are issued document control numbers, inventoried, and properly archived. An example of a Controlled Temperature Log is presented in Appendix X.

C) Balance Monitoring

Each analytical and top loading balance used at TriMatrix is monitored for accuracy. All daily checks are recorded in a TriMatrix Balance Log (Appendix Y). All balance logbooks are issued document control numbers, inventoried, and properly archived.

D) Standard and Reagent Preparation Logbooks

All standards and calibration solutions used at TriMatrix are prepared, when possible, from reagents or solutions traceable to national standards. Whether a stock, an intermediate, or a working concentration, each reagent and standard solution is traceable to its origin. This is accomplished using the laboratory's LIMS system and/or a Standard Preparation Logbook (Appendix Z).

Information available on each standard includes:

- The analyte or analytes contained in the standard
- The concentration
- The solvent used to prepare the standard
- The preservative (i.e., nitric acid)
- The date of preparation
- Initials of the preparer
- The expiration date
- The unique identification number

Unique identification numbers are generated by the LIMS system and/or a book, page, and line number system. All standard and reagent preparation logbooks are issued document control numbers, inventoried, and properly archived.

E) Pipet Logs

All autopipetors utilized for the delivery of standard solutions, diluents, and reagents, are periodically checked for delivery

accuracy. Because these pipetors contain mechanical parts they are subject to inaccuracies if not properly maintained and calibrated.

Daily calibrations (for pipets used to prepare standards), and weekly calibrations (for pipets used to prepare quality control samples) are recorded in a Pipet Calibration Logbook (Appendix AA). Each log is identified by manufacturer name and model number, the pipetor serial number (if available), and the starting and ending dates encompassed. All complete pipet logbooks are assigned document control numbers, inventoried, and properly archived.

4.1.3.9 Confidentiality and Proprietary Rights

Since significant amounts of information regarding the details of a client's operations are received in the laboratory, it is essential that strict confidentiality be maintained in the handling of all client information. Client data is protected in locked filing cabinets and in limited access computer files. Under no circumstances is the name of a client, or any information regarding that client, revealed to another client or to a regulatory agency without the client's written permission, under penalty of employment termination.

Any details of a client's operations that have necessarily been revealed to the laboratory for testing purposes are considered as proprietary and protected by patents, copyrights, infringement laws, or other legal constraints against disclosure.

4.1.3.10 Document Storage and Traceability

Archiving of information at TriMatrix has been designed to meet both short-term and long-term storage needs. Archives are maintained for a wide variety of data and documentation. These archives can be categorized into two main groups, a) document

archives (physical documents) and b) electronic archives (data files). Table 1 illustrates the current TriMatrix archival systems, their location, and duration.

Documentation records or logs are maintained for all archival systems to aid in the quick retrieval of information. Extended archival periods or special procedures are also in place for some projects and clients.

4.1.4 Standard Operating Procedures (SOPs)

Many of the methods published today by various agencies provide only general guidance in performing an analytical determination. A significant part of the variability observed in analytical data is in large part due to minor variations in the analytical process. A Standard Operating Procedure is a guide that clearly defines the exact steps to be followed while performing a procedure. The delineation of these exact steps in an SOP will improve the analytical conditions, which in turn will help the overall reproduction of analytical data.

4.1.4.1 SOP Categories

SOPs are written for nearly all laboratory activities. The categories utilized in the organization of SOPs are presented in Table 2.

4.1.4.2 SOP Development, Formatting, and Review

All standard operating procedures are developed and written to the specifications outlined in the TriMatrix guidelines for the preparation of a SOP. These guidelines are presented in SOP format and have been designed to accommodate analytical tests, non-tests such as extractions or digestions, and documentation or non-analytical activities. The guidelines were developed from both USEPA and ASTM protocols for the creation of standard operating procedures.

All SOPs developed by TriMatrix are subject to a review process where signatures or approvals are required from the appropriate area manager, the quality assurance department, and the Vice President of Laboratory Operations. In addition to this overall approval process, each page of an SOP is individually approved by both the laboratory area and quality assurance department (Appendix AB).

SOPs are reviewed and updated as necessary. Minor modifications can be hand edited on the SOP. These modifications must be made through the Quality Assurance Department. Depending on the modification, distribution of the edited SOP (as described below) may or may not be required. All minor modifications will be incorporated into the next revision of the SOP. Major modifications may require the SOP to undergo an immediate formal update.

4.1.4.3 SOP Documentation and Control

All SOPs are assigned a unique procedure identifier. Other information included in every SOP is the effective date, revision number, information on the author, total number of pages, and identification of any individual page revisions.

All original, approved paper copies of TriMatrix SOPs are controlled by the Quality Assurance department. Approved SOPs are scanned and stored on the network Library drive. This drive is accessible to all laboratory personnel. Copies of all outdated SOPs are destroyed (or marked as obsolete), and the scanned copy of the SOP is removed from the Library drive.

4.1.5 LIMS

TriMatrix utilizes the Element LIMS system developed by Promium Corporation. This system controls all aspects of laboratory operations. The main functions of the LIMS system are:

- Project Management
- Sample Management
- Work Scheduling and Management
- Data Entry, Verification, and Approval
- Report Generation
- Invoicing

4.2 SAMPLE CONTROL, FLOW, AND STORAGE

Presented in the following section is a description of the policies and procedures that were developed to identify, monitor, and document the flow of samples through the Laboratory. A flow chart depicting this process is presented in Figure 4-3.

4.2.1 Project Initiation

When samples are received at TriMatrix, the necessary information that will direct the analytical scheme has already been developed and implemented within the project initiation/project management process. This process starts with the award of a contract or proposal, a client request, or a pre-scheduled sampling event. The basic steps and supporting documentation involved in the project initiation process begins with the gathering of project information, communications with all affected laboratory areas, and the input of required project related data into the LIMS system. All requests for analytical work are reviewed by the project chemist, and when necessary, applicable management staff to verify the laboratory has the capability to perform the requested tests and meet the requested turnaround times. Requests for changes to in-progress projects must be made with the appropriate project chemist. Changes in methodology will typically require client approval. The project chemist will be responsible for coordinating all requests for changes with the impacted laboratory areas. All approved changes will be formally made via the laboratory's LIMS system, thus continuing the normal paperwork flow.

TriMatrix uses test methods that meet the needs of the client and are appropriate for the tests undertaken. Methods published in international, regional, or national standards are used. TriMatrix uses the latest valid edition of a method unless it is not appropriate or possible to do so. All analytical procedures are documented in SOPs supplemented with additional details to ensure consistent application.

When not specified by the client, TriMatrix will select appropriate methods published either in international, regional, or national standards, by reputable technical organizations, in relevant scientific journals, or as specified by the equipment manufacturer. Laboratory developed methods (or methods adopted by the laboratory) are also used when appropriate for the intended use, and have been validated following the various initial demonstration of capability procedures. When specified by the client, TriMatrix will inform the client if the specified method is considered inappropriate, or out of date.

Routine projects include samples matrixes and analyses that are continuously processed by TriMatrix. Non-routine projects are those that require special analyses, include parameters not routinely run by the laboratory, posses unique holding times, or require expedited turnaround. Non-routine projects will require approval from all affected laboratory areas. This approval process is communicated in several different ways, including everything from the signing of a quality assurance project plan (QAPP) to the transmission and receipt of an electronic mail message.

Occasionally, a portion of a project may involve an analytical methodology not currently possible at TriMatrix. When requested by the client, samples for analyses outside the analytical scope of TriMatrix can be subcontracted to another laboratory. It is preferred that the client specify the subcontract laboratory. When the subcontract lab is not specified by the client TriMatrix will only subcontract to laboratories that are NELAP accredited, or ISO-17025 certified, for the specific method of interest. Client specific program requirements will take precedence over this rule. A registry of subcontract

laboratories used by TriMatrix will be maintained, documenting their NELAP accreditation or ISO-17025 certification.

The development of a project within the laboratory also involves the preparation and shipment of sample collection materials and containers. The processes involved in the procurement, preparation, and shipment of sample collection materials and containers are presented in the sections below.

4.2.1.1 Sample Containers and Materials Procurement

TriMatrix utilizes only virgin bottle ware for all sample collection kits. All containers are purchased pre-cleaned and come with Certificate's of Analyses.

Specific projects or programs may require the laboratory to verify the cleanliness of the containers. When this is required specific lots will be sequestered from the container vendor. Each lot will be tested to verify the containers meet the project or program requirements. Only containers whose cleanliness has been verified will be used for the project.

4.2.1.2 Preparation of Containers

All sample containers utilized for the collection and preservation of environmental samples are prepared by the bottle prep group. The staff members of this group focus their activities exclusively in the area of sample container procurement, preparation, and shipping. Project sample container kits are requested using the Container Packing List, presented in Appendix N.

4.2.1.3 Sample Container Shipment

When all containers have been assembled as requested on the Master Bottle Packing List, the bottles are packaged and placed into one or more shipping coolers. 40 mL glass vials are packed in

small bubble pack bags. An attempt is made to organize each sample cooler to help minimize time spent in the field. When possible this is accomplished by packing bottles together by sample point. When complete, each shipping container will be inspected by a project chemist to verify its accuracy. Documentation of this inspection is made on the bottle packing list. A copy of the bottle packing list is placed in each cooler.

Also provided in each cooler is a set of instructions or comments about the containers, material safety data sheets for all chemical preservatives present, a return address label, an external COC form, and if required, TriMatrix sample bottle custody seals. All materials are packaged in a waterproof zip-lock bag. Examples of these additional materials are presented in Appendix AC.

Packing is now added to the cooler and the shipping container is sealed. When requested, signed TriMatrix custody seals can also be applied to the outgoing cooler.

4.2.1.4 Sample Receipt

The receipt of all sample shipping coolers (empty or full) will be documented in the Sample Receipt Record logbook (Appendix O). This logbook documents the delivery method, date, and time, the number of coolers received, the client, and the name of the TriMatrix employee who received the cooler. This information is entered into the logbook immediately after drop-off.

As soon as possible after the shipping cooler is received and all available information entered into the Sample Receipt Record, cooler inspection and sample temperature determination occurs. The observations associated with this step by step process are recorded on the "Sample Receiving/Log-in Checklist" (Appendix P). This Checklist must be completed for all samples for a given project received on a given day. A supplemental "Sample

Receiving/Log-in Checklist Additional Cooler Information" form is available when receiving projects consisting of more than four coolers (Appendix Q).

IMPORTANT: When initiating each Checklist, make sure the Receipt Log Page/Line number from the Sample Receipt Record logbook is recorded at the top of each Checklist. This ties the receipt of the sample coolers in with the samples themselves.

Record the cooler number of the first cooler and the current time. Observe and record the type of coolant used. When possible, the sample temperature of three random samples (locations representative of the coolant present in the cooler) will be taken. If a temperature blank was received, measure and record this temperature as well.

Sample temperatures are recorded using a calibrated infrared thermometer. Because this type of thermometer is actually measuring the temperature of the container, it is critical that the temperature is taken as the sample is removed from the cooler. The container warms up quickly and any other method will result in an incorrect reading. Do not dry the container prior to measuring the temperature. Containers wet from melt water are preferred to dry containers. Record the temperature values on the Checklist. Report all temperatures to the nearest 0.1° C. If a correction factor is necessary, record the correction factor and the corrected temperature on the Checklist. If any temperature exceeds 4° C, average the three sample results and also report the average. If the average temperature of the three samples, or the temperature of the temperature blank exceeds the 6° C required by most regulatory bodies, it must be noted on the Checklist.

If sample receipt and temperature determination occurs outside of normal business hours, place received coolers in the walk-in for

storage. Assemble all the paperwork, and place it in the after-hours basket. The remainder of the receiving process will be performed by a log-in technician during the next business day.

4.2.1.5 Sample Examination

Samples received at TriMatrix are required to be accompanied by a TriMatrix Laboratory Chain-of-Custody (COC) form (Appendix AD). For samples received without this form, the log-in technician will initiate the COC process. Should a submittal or delivery group be identified as an internal COC project, the log-in technician will initiate the procedures outlined in section 4.2.2 B.

The remainder of page 1 of the Checklist is now filled in. Observations are made on the accuracy of the COC and the condition of the sample containers. Many of the aqueous samples received have been subjected to some form of chemical preservation. Verification of the preservation is required; however, depending on the analysis this verification may not occur during the log-in process. The "Sample Preservation Verification Form" (Appendix R) specifies what container types will have their preservation verified during log-in. The form also specifies what container types can have an incorrect preservation adjusted. Preservation verification is performed via a pH check using calibrated pH strips. Determine the correct reading against the color chart on the pH strip container. Document the pH found on the Sample Preservation Verification Form. Use only the pH strips located in the log-in area whose calibration has been verified and recorded in the pH Strip Calibration Logbook (Appendix AE).

Should a) the result of any preservation check indicate that the sample has not been properly preserved in the field (or the buffering capacity of the sample has resulted in an unacceptable sample pH at receipt) or b) there is insufficient evidence indicating that other needed preservation reagents (e.g., Zinc Acetate for

Sulfides) have been added, then a Sample Receiving Non-Conformance Report (Appendix S) is to be initiated and the project chemist contacted as soon as possible. In some instances, the holding time of such samples may be shortened. No preservation adjustment may be made without approval from a project chemist.

IMPORTANT: Shaded boxes on the Checklist indicate an out-of-control situation. The selection of any shaded box during the completion of this form also requires the initiation of the Sample Receiving Non-Conformance Report.

Collect all paperwork and deliver to the appropriate project chemist for review. Any issues that require contact with the client for resolution will be made in a timely manner. The project chemist will create a submittal and return the paperwork. Once the project chemist returns the paperwork, page 2 of the Checklist can be completed, and the samples logged into the LIMS system.

4.2.1.6 Sample Log-In

All samples received by TriMatrix are logged into the LIMS system. The log-in procedure assigns a unique TriMatrix sample number to each sample, allowing samples to be tracked, data stored, and quality control associated for any sequence of events during a particular analytical period. The primary steps involved in the sample log-in process are presented below.

4.2.1.7 Sample Splitting

In the event that TriMatrix is unable to provide sample bottles, or circumstances prevent the splitting of samples in the field, the log-in technician can provide sample splitting services; however, sample splitting will typically be performed by a laboratory area chemist. These services include taking the sample as received and

sub-sampling it into the appropriate bottle with the preservative requirements as set forward in Appendix AF – Sample Collection Guidelines Bottle and Preservative Requirements. Sample splitting will only be performed when instructed by a laboratory project chemist with client approval.

A. Sample Splitting-Water Samples

Laboratory area managers will be consulted in order to insure that sufficient volume will be available to all areas of the lab after splitting. In the event that sufficient volume does not exist, the Project Chemist will be immediately notified for resolution.

When a bulk sample arrives for both organic and inorganic analysis, and sufficient sample exists, the organic aliquots will be removed first. The remainder of the sample will be transferred to properly preserved containers for each inorganic analyses.

B. Sample Splitting-Solid Samples

When solid samples, such as sediment or soil, are to be received at TriMatrix, every attempt will be made by the Project Chemist and field sampling personnel to insure that two samples are provided as replicates for the appropriate tests. One of these samples will be assigned to the organic area and the other to the inorganic area. If only one sample is received and if organic analyses are required, the organic aliquots will be removed first. Prior to sub-sampling, solid samples will be made homogeneous by either one or all of the following manners:

- Stirring
- Grinding
- Particle separation (sieving)

The laboratory area manager is responsible for deciding how a solid sample will be split. Problems or concerns that may arise on splitting a solid sample will be addressed by the Project Chemist and Laboratory Area Manager. After the organic portions have been removed or split, the remaining sample will be provided to the inorganic facilities for any further splitting.

4.2.1.8 Sample Distribution

All samples received at TriMatrix are labeled by the log-in technician. These labels include both the necessary information for proper identification, and information on any potential for flammability, reactivity, contact, or health based risks.

After completing the log-in process of all the various samples connected with a particular project, the log-in technician will store the samples in the correct Controlled Temperature Unit (CTU).

- Routine Water and Solid Samples: Samples that require refrigeration will be stored in the CTU designated for all routine water and soil samples.

- Routine Volatile Water and Solid Samples: All volatile samples are stored in designated VOA CTUs. Volatile water and soil samples are segregated and stored separately. No other sample types are stored in the VOA CTUs.

  All CTUs used for VOA sample storage will also contain a storage blank. The storage blank is a preserved 40 mL VOA vial filled with deionized/distilled water. The storage blank is replaced and analyzed on a weekly basis. If positive results are observed for any target analyte above the laboratory's minimum reporting limit, all samples stored concurrently in the CTU must be evaluated for possible contamination. All sample results

within 5 times the level quantitated in the storage blank must be qualified as estimated.

- Odoriferous and Hazardous Samples: Stored separately in a special vented facility. If volatile analyses are to be performed, samples are stored under refrigeration. Samples are identified to the laboratory by means of a narrative within the LIMS System.

All samples that are involved as physical evidence in a legal procedure or simply identified as Chain-of-Custody will be handled under COC procedural safeguards.

4.2.2 Chain-of-Custody (COC)

All samples received by the laboratory require some form of chain-of-custody (COC). TriMatrix practices two levels of COC, external and internal. The degree of custody tracking and documentation is driven by the final deposition of the laboratory data. Generally, if samples and their analytical results are subject to involvement as physical evidence or in a legal procedure, both external and internal custody procedures will be followed. If samples or results are not subject to legal procedures, only external COC procedures will be followed. A description of these two custody scenarios is presented as follows:

A. External COC

Samples only requiring external COC will have their custody tracked from sample collection to delivery at the laboratory. This process involves the completion of a TriMatrix external COC form, as presented in Appendix AD. This form accompanies the sample containers prepared by TriMatrix to the sample collection site. Any sample or submittal received at the laboratory without a TriMatrix external COC form will initiate a process where the log-in technician will complete the necessary external COC forms for carrier sign-off.

For document control purposes, all external COC forms have a unique identification number.

B. Internal COC

Samples requiring strict COC will initiate the process by which all events or periods of sample handling will require a traceable document protocol.

The internal COC process involves the completion of a TriMatrix internal COC form for all phases of the analytical process. This includes sample extractions, distillations, digestions, analyses, and disposal. An example of the TriMatrix internal COC form is presented in Appendix AG. All internal COC forms are maintained in a series of submittal or delivery group folders.

C. Sample Security

All samples, whether under external or internal COC protocols, are maintained in a limited access secured area. This level of security is applied to all phases of the analytical process from sample log-in to final sample disposal.

D. Sample Disposal

All samples received are subject to disposal as waste once tested and discarded. Three general categories discarded samples fall into are the following:

1. A sample may be returned to the client (specifically, if highly contaminated).

2. Too contaminated for municipal disposal and must be disposed of as waste through a hazardous waste facility.

3. Inert, uncontaminated, and nontoxic samples in accordance with municipal waste regulations may be disposed of in the municipal dumpster and/or the laboratory waste room sink leading to the city sewer.

4.2.3 General Laboratory Security

Access to the laboratory is handled in a secure fashion, with access restricted to authorized personnel only. All laboratory areas including sample storage, sample container preparation, analytical laboratories, sample preparation, sample disposal, analytical documents, and data files are restricted. Non-authorized personnel may enter these areas only when escorted by a laboratory staff member.

It is the responsibility of all laboratory staff members to insure that the rules of restricted access are followed and maintained at all times.

4.3 CALIBRATION AND CALIBRATION VERIFICATION

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment used in conducting laboratory analyses. Calibration of the instruments and equipment is performed prior to each use or on a scheduled periodic basis.

Calibration of laboratory instruments and equipment is performed to verify that the analysis portion of the testing process is functioning properly and at the required sensitivity. A calibration section included in each analytical SOP covers the frequency, stability, and specific calibration steps, based on analytical method requirements and instrument or equipment manufacturer's recommendations.

Initial calibration is performed using standards of certified value to establish the linear range of the analysis for the analytes of interest. Each calibration curve is verified using a Second Source Calibration Verification Standard (SCV) prepared from a source dissimilar to that used in the preparation of the calibration standards. The calibration is also verified at the beginning and during the analytical sequence, using a standard prepared from the same source as that used in the initial calibration.

Calibration activities are divided into three categories:

Field Equipment (section 4.3.1)

Laboratory Instrumentation (section 4.3.2)
Laboratory Equipment (section 4.3.3)

4.3.1 Field Equipment

Perform daily calibration checks on field equipment prior to the commencement of any field analyses. Follow the written calibration procedure for each individual piece of field equipment. The equipment is held out of service until repairs and successful recalibration occurs. A summary table of all calibration procedures and frequencies is included (Table 3).

4.3.2 Laboratory Instrumentation

Calibration of laboratory instruments is based on approved SOPs. Records of calibration, repairs, or replacement are filed and maintained by the designated laboratory analyst. These records are filed at the location where the work is performed and are subject to QA audit. For all instruments, the laboratory maintains in-house spare parts or service contracts with vendors. A summary table of all calibration procedures and frequencies is included (Table 4). Flag any instrument that does not pass daily requirements. Hold the instrument out of service until repair or successful recalibration occurs.

4.3.2.1 Inorganic/Classical Chemistries

Inorganics analysis utilizes a wide variety of wet-chemical procedures and instruments. Calibration steps may vary depending on the specific analytical method being utilized. However, certain general principles of calibration apply to all inorganics testing. Every analytical method requires calibration or calibration verification prior to sample analysis. Using a group of certified standards, the linear range is defined. The calibration is checked on a continuing basis to be certain that the method is within the required test parameters. All inorganic calibrations must meet the specific requirements described below unless required otherwise by the method or manufacturer.

The instrumentation used to conduct these analyses is calibrated using calibration standards prepared by dilution of stock solutions. One standard is prepared at the reporting limit of the analyte of interest while the other standards bracket the concentration range of the samples. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve.

An SCV originating from a dissimilar stock solution than that used for preparation of the calibration standards is prepared and analyzed. Continuing Calibration Verification blanks and standards (same source as that used in the initial calibration curve) are run at the beginning, and periodically, throughout the analytical sequence, typically after every 10 analyses. The value of the continuing calibration standard concentration must agree within the method specified criteria; generally ±15 percent of the initial value or the appropriate corrective action is taken. Corrective action may include recalibrating the instrument and must include reanalyzing the previous 10 samples.

4.3.2.2 AAS/ICP/MS Emission Systems

The atomic absorption spectrophotometer (AAS), inductively coupled plasma emission spectrophotometer (ICP), and inductively coupled plasma mass spectrometer (ICP/MS) instruments are calibrated by the use of a minimum of three calibration standards (6 for ICP/MS) prepared by dilution of certified stock solutions. One standard is prepared at the reporting limit of the analyte of interest while the other standards bracket the concentration range of the samples. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration

standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. Calibration standards contain acids at the same concentration as the digestates. A continuing calibration standard is analyzed after every 10 samples. The value of the continuing calibration standard concentration must agree within method specified criteria, generally ±10 percent of the initial value or the appropriate corrective action is taken. Corrective action may include recalibrating the instrument and must include reanalyzing the previous ten samples.

4.3.2.3 Gas/Liquid Chromatography

Analysis performed by gas chromatography follows USEPA protocols. The instrument is calibrated using three or five point calibration curves (depending on method requirements) for both volatile and semi-volatile compounds. The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. Continuing calibrations are performed after every ten samples. The value of the continuing calibration standard must agree within ±15 or 20 percent (depending on method requirements) of the initial value or the appropriate corrective action is taken, which may include recalibrating the instrument and must include reanalyzing the previous ten samples.

4.3.2.4 Gas Chromatography/Mass Spectrometry (GC/MS)

Prior to calibration, the instruments used for GC/MS analyses are tuned by analysis of p-bromofluorobenzene (BFB) for volatile analyses and decafluorotriphenylphosphine (DFTPP) for semi-

volatile analyses. Once the tuning criteria for these reference compounds are met, the instrument is initially calibrated using a three or five point calibration curve (depending on method requirements). The high or the low standard may be omitted from the calibration curve; however, the minimum number of calibration standards required by the method must still be maintained. Additionally, the minimum reporting limit must be elevated, or the linear range reduced, if the corresponding standard is eliminated from the calibration curve. The instrument tune will be verified each 12 or 24 hours of operation (depending on method requirements). Continuing calibration is verified as specified in the method. The calibration standards are commercially available certified standards containing the target analytes, surrogate spikes, and internal standards.

4.3.3 Laboratory Equipment

Personnel performing calibration should also be alert for any condition that renders a piece of equipment inoperable or unfit for use; for example, inspect thermometers to ensure that mercury or alcohol columns are not separated. If an equipment malfunction is noted during calibration, the equipment must be tagged and removed from service. The equipment is held out of service until repairs and successful recalibration occurs. Record all malfunctions, repairs, and re-calibrations in the appropriate logbook.

Maintain records for each piece of equipment requiring calibration, showing equipment description and identification number, calibration frequency and acceptable tolerances, personnel performance calibration, date, reference material used, calibration results including acceptance or failure, removal from service, repairs, and date and authorization for return to service.

4.3.3.1 Balances

An annual third party maintenance and calibration is performed on all balances. Daily calibration is performed by TriMatrix on all

balances using class S or higher NIST traceable weights. Provided daily calibration is successful the weights themselves are indirectly calibrated on a daily basis via the third party's calibration; therefore, re-certification or replacement of the weights is not required every five years.

4.3.3.2 Thermometers

Thermometer calibration is performed annually, using a NIST certified thermometer. The NIST thermometer must be re-certified or purchased new every five years. Written records are maintained of all annual calibrations.

4.4 DATA REDUCTION, VALIDATION, AND REPORTING

Data reduction is the process by which raw analytical data is tabulated and calculated. Data validation is the review of the data generation and reduction process. Data reporting is the compilation of all sample results for distribution to the client. All analytical data generated by TriMatrix Laboratories is subjected to the reduction, validation, and reporting process as described below.

4.4.1 Laboratory Data

4.4.1.1 Data Reduction

Initial results for most analyses are calculated using a computer directly interfaced to the instument. Data reduction is accomplished using software that has been validated for its intended purpose. The initial result is exported to the LIMS system. Data such as initial volume, final volume, and percent solids, are used by the LIMS system to calculate a final result. When manual data reduction is required, it is performed according to the written standard operating procedure for that analysis.

4.4.1.2 Manual Integrations

Manual integration is defined as any post acquisition adjustment to the automated software peak integration. Manual integrations are often times legitimately required to correct for baseline drift, noisy baselines, poorly resolved peaks, closely eluting or missed peaks, peak tailing, or peak splitting. Manual integration may never be used for the sole purpose of correcting failing quality control parameters (i.e. shaving or enhancing peak areas or heights to make failed calibrations, surrogates, or internal standards pass), or as a substitute for poor or ineffective sample cleanup. Manual integration must be used cautiously due to the increased scrutiny inherent with adjusted data. Particular attention will be paid to manual integrations performed on standards and blanks since these samples are typically free of interferences.

Before and after documentation must be provided with all manual integrations. This documentation must clearly show the original integration "before", and the manual integration "after" baseline. Clear identification of manual integrations must be included in the case narrative for all samples analyzed under Federal Facilities work requirements. All quantitation reports must clearly identify manual integrations by flagging the peak with a designator that cannot be removed by the analyst. Additional documentation requirements include:

- Date of the manual integration
- Reason for the manual integration
- The integration area or height before manual integration
- The integration area or height after manual integration
- A signature/date by both the analyst and the reviewer.

Any questions concerning manual integration must be resolved with the area manager or the quality assurance officer before final results are approved and released to the Project Chemist. The

complete laboratory manual integration requirements are detailed in the TriMatrix manual integration SOP GR-10-115.

4.4.1.3 Four Levels of Data Validation

First Level Review

Data validation begins with the analyst. It is the basic responsibility of the analyst to produce data that is complete, correct, and conforms to all applicable methods and standard operating procedures. If results are not acceptable, it is the duty of the analyst to perform the appropriate corrective action and to thoroughly document that action. The analyst will verify the following before updating the analysis status to "Analyzed":

- Applicable standard operating procedures were followed
- Proper analytical sequence was followed
- Sample preparation information was correct
- Calibration has been performed properly
- Analytical results are complete
- Holding times have been met
- Method criteria were met
- Any special sample preparation or analytical requirements have been achieved
- All analytical abnormalities have been noted
- Corrective actions are thoroughly described
- Good record keeping practices have been followed
- Any problems are communicated to area manager
- Data was correctly transferred to Element
- Calculations were performed properly
- Quality control samples are within established limits
- Documentation is complete
- Raw data, including chromatograms and instrument printouts are complete
- Case narrative or qualifier pages are complete

Second Level Review

A laboratory area peer or designated validator, in essence, performs the same validation steps performed by the analyst. Particular attention should be paid to:

- Dilution factors were entered correctly and detection limits elevated accordingly
- Analysis dates are correct
- Quality control and analytical batch information is correct
- Quality control results and spike amounts are correct and in control
- Project specific limits are correct
- Run a draft copy of the report, specific to the laboratory area, to verify all results have been adjusted correctly
- Any required qualifiers or narratives have been entered

Any problems must be resolved with the analyst, and when appropriate the quality assurance manager, prior to updating the status to "Reviewed."

Third Level Review

Once all analyses associated with a work order have been entered into the LIMS system and approved, the project chemist will perform the Third Level Review. This review will verify that:

- The requirements of the client have been met
- All required narratives and qualifiers have been included
- All quality control parameters required are in the report
- Results of complimentary tests make sense
- The data is accurately presented
- Holding times have been met
- Calibration checks are sufficient

- Documentation is complete

Once this review is complete the project chemist will approve the data and generate a final report. It is during this time that any data package deliverables are collected and reviewed. When printed the work order status updates to "Reported."

Fourth Level Review

The project chemist will perform a final review of the data package hard copy to ensure that:

- All required data package components are complete and accounted for
- Quantitative results are correct
- The overall presentation of data to the client is in an understandable format

In addition to the formal data validation guidelines listed above for the analyst, area manager, and project chemist, there are many practical questions that all of these persons need to keep in mind when reviewing data and finished client reports. Among these "common-sense" evaluations of laboratory data are the following important considerations:

- Data makes good, sound, practical sense
- Multiple runs of the same samples relate, match, or are within acceptable range
- Data from complimentary analyses compares, i.e. COD>BOD>CBOD
- Total cyanide ≥ amenable and free cyanide
- Total solids ≥ suspended and dissolved solids
- TKN ≥ organic N + ammonia N
- Inorganic N = ammonia N + nitrate N + nitrite N
- TOC < BOD or COD

- Total phosphorus ≥ ortho phosphorus
- Calculated total dissolved solids/conductivity = 0.55 – 0.7
- Analytical run looks good; proper decisions were made
- Peaks from chromatogram or instrument printout look normal
- Computer identifications are correct
- Are qualitative/quantitative results real, especially low level
- Know and be sensitive to common laboratory contaminants
- Know area/analytical method pitfalls-be extra cautious
- All practices are sound and are supported by documentation-no appearance of random decisions

When complete the report will be signed. Data packages with deliverables will be scanned and archived. Work order status will be updated to "Completed".

4.4.2 Field Data

All data reduction, validation, and reporting for field activities must meet the same requirements as those required in the laboratory. Many of the field instruments, such as those measuring pH, dissolved oxygen, turbidity, temperature, and specific conductance, require a manual data printout from a computer interface. The analyst is responsible for immediate tabulation and calculation of raw data in the field. The field section manager must perform a prompt, on-site validation of field data before the opportunity is lost to perform any necessary field re-tests.

4.4.3 Subcontracted Data

Analytical results from subcontracted samples will be reported as an attachment to the TriMatrix data package. The attachment will contain the entire subcontracted data package as received by TriMatrix. To eliminate the impression that the subcontracted analyses were performed by TriMatrix, subcontracted results will never be incorporated into the TriMatrix generated report.

4.5 VERIFICATION PRACTICES - EXTERNAL/INTERNAL QUALITY CONTROL

4.5.1 Standard Reference Materials

A crucial step in the generation of quality data is the purity and traceability of reference materials used in the analyses. Reference materials may be physical standards (such as certified thermometers and weights used to calibrate laboratory thermometers and balances) or chemical standards (used to establish and check operational calibration of analytical methods). Physical standards should be traceable to the National Institute of Standards and Technology (NIST). Physical standards must be recalibrated (by an external vendor certified to perform the calibration), or purchased new every five years. Chemical reference materials of high quality can usually be obtained from reliable commercial vendors. For a given analysis, standard reference materials must be kept on hand from more than one vendor source. During the testing operation, standard reference materials from different vendor sources are crosschecked with each other.

4.5.2 Internal Quality Control Programs

TriMatrix routinely adds samples to the sample stream to demonstrate the total testing process is operating within prescribed limits for accuracy and precision. With the exception of Blanks, the concentration of these quality control samples is known prior to the analysis. Types of Quality Control Samples are presented in Table 5. Duplicates and spiked duplicates are selected at random, and when not specified are rotated among clients.

4.5.3 External Quality Control Samples-Proficiency Testing

TriMatrix Laboratories receive Performance Testing (PT) samples on a scheduled basis from state and federal regulatory agencies as well as certain client organizations. A summary of these PE samples is given below:

PT Program	Sample Type	Source	Frequency

WS	Drinking Water	ERA	Semi-Annual
WP	Waste/Ground Water	APG	Semi-Annual
Soil	Soil	ERA	Semi-Annual
Varies	Environmental	State/Federal Programs	Varies
Varies	Environmental	Client	Varies

TriMatrix receives written reports from sponsoring agencies grading not only their performance, but also a comparison to other laboratories participating in the study. This provides feedback to laboratory personnel regarding the satisfactory use of analytical methods and equipment. Additionally, results from all single and double blind PT samples are used as part of the laboratories fraud prevention and detection program.

4.6 DATA ASSESSMENT PROCEDURES

4.6.1 Precision

Precision of laboratory analyses will be assessed by comparing the analytical results between matrix spike/matrix spike duplicate (MS/MSD) for organic analyses, and laboratory duplicate or MSDs for inorganic analyses. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses using the following equation:

$$\%\text{RPD} = \left(\frac{S - D}{\frac{S + D}{2}} \right) \times 100$$

where:

S = first sample value (original of MS value)

D = second sample value (duplicate or MSD value)

4.6.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria using the analytical results of method blanks, reagent/preparation blank, matrix spike/matrix spike duplicate samples, equipment blank, and trip blanks. The percent recovery (%R) of matrix spikes will be calculated using the equation below:

$$\%R = \left(\frac{A - B}{C} \right) \times 100$$

where:

A = the analyte concentration determined experimentally from the spiked sample;

B = the background level determined by a separate analysis of the un-spiked sample

C = the amount of the spike added

4.6.3 Control Limits

Unless fixed in the analytical method, all quality control acceptance limits in use at TriMatrix are derived from historical data, for each method, matrix, and QC type combination. Precision and accuracy control limits are calculated at a 99% confidence level (+/- three standard deviations); warning limits are calculated at a 95% confidence level, (+/- two standard deviations). Accuracy windows are calculated using the mean of the percent recoveries. Precision windows are calculated as specified in SW-846, using the relative percent difference of the amounts found, not the percent recoveries.

4.6.4 Uncertainty

In addition to the precision and accuracy of a result, a value relating to confidence is available in the form of a measurement uncertainty estimate. The measurement uncertainty value is estimated using the QC-based nested approach and is calculated at the 95% confidence level. Uncertainty estimates are reported as "percent relative uncertainty."

4.6.5 Completeness

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. The completeness is calculated as follows:

$$\text{Completeness} = \left(\frac{\text{valid data obtained}}{\text{total data planned}} \right) \times 100$$

4.7 PROCEDURES FOR CORRECTIVE ACTION

When a non-conforming event or process deviation has occurred, corrective action is required. A written standard operating procedure (plan for corrective action) provides the steps for dealing with an out-of-control testing situation. The assessment of whether the process is out-of-control is based on predetermined limits for laboratory operations. Non-conformances based on statistical analysis or quality control samples are readily apparent and easy to identify. A process deviation, which does not have a directly observable impact on data quality, is more difficult to discern. Examples of the latter, subtler types of non-conformances include volatile samples not properly stored; oily layers in certain types of samples that may interfere with analysis; or a water-soaked sample label whose information is barely legible. Discovery of a non-conforming event or process deviation can result from the observations of a staff member, a review of laboratory data at any level, the result of an audit, or a client complaint. A corrective action investigation will be initiated within one week of the discovery of any non-conformance. The time frame required to resolve a specific deficiency and implement the corrective action is dependant on the magnitude of the problem and the defensibility and use of the data. Most non-conformances should be resolved within 60 days from the initiation date. Non-conformances that specifically impact sample results should be resolved within 14 days.

NOTE: The client must be contacted within 48 hours (2 business days) upon the discovery of any event that may cast doubt on the validity of a sample result.

The overall scheme of a corrective action plan can be outlined as follows:

1. Define the problem and evaluate the significance of the non-conformance;

2. Assign responsibility for evaluating the problem and determine if the client should be notified and/or work recalled;
3. Determine thorough investigation of all the pertinent facts what the probable cause of the problem is;
4. Select and implement the action(s) most likely to eliminate the problem and prevent recurrence;
5. Assign responsibility for carrying out the corrective steps and implement the action;
6. Follow-up to ensure that the problem has been eliminated and when necessary authorize the resumption of work.

Specific responsibility for implementing corrective action is as follows:

It is the responsibility of the analyst or other employee who observes a non-conforming event to:

- Identify and define the problem.
- Fill out a Non-Conformance Investigation Report (refer to Appendix AH).
  When applicable, investigate and attempt to determine the cause of the problem. Report the problem promptly to the area manager. When applicable, accept responsibility for implementing the corrective action approved by the area manager.
- When applicable, evaluate the effectiveness of the corrective action.
- When applicable, verify that the corrective action has eliminated the problem.

It is the responsibility of the laboratory area manager to:

- Review the problem and the proposed corrective action.
- If the reporting person does not have a remedy, work together with the person to determine a satisfactory solution.
- Assign the final corrective action steps to be performed.

It is the responsibility of the QA Department to:

- Follow-up to ensure that the problem has been eliminated and when necessary authorize the resumption of work.
- Review, sign, and categorize every Non-Conformance Investigation Report.
- Randomly review corrective action documentation in laboratory through internal audits to ensure that adequate records are being kept.

The ultimate goal of every non-conformance investigation is to resolve the error through identification of the error's root cause. Ideally, once the source of error is found, change can be implemented to prevent reoccurrence of the same error thereby providing a system of continuous quality improvement.

Non-conformances can originate from anyone in the laboratory. Provide the QA department with a copy of the initial report at the time of its distribution, followed by a copy of the completed report. The final report will be distributed to all necessary personnel. Initiation of non-conformance reports associated with out-of-control PT samples will commence with the QA department. The initial non-conformance will be typed up and may include attachments such as a graph charting the history of PT results for that analyte. The history of results for that analyte in PT studies will also be reviewed through the database, looking at additional items such as method, matrix, analyst, vendor, and study type (WP, WS, etc.).

NOTE: Non-conformances associated with PT samples must be completed and distributed to state, federal, and other applicable regulatory agencies within the time frame established by that agency.

Returned non-conformance reports will be typed and the final report may include copies of raw data, information concerning traceability, graphs charting historical data, graphs charting trends in analysis, calibration graphs, or any other information relevant to the investigation.

When investigating a failing PT sample, a questionable analytical result, or a client complaint, the following systematic approach for error analysis should be followed until the primary source of error is located and resolved. Progress through them in the order they are presented below (easy to determine transcription error through difficult to determine analytical/procedural failure).

1. Consolidate all necessary raw information, run data and associated calibration and quality control data for both the reported and any non-reported analyses of that sample.
2. Confirm that the intended result was the reported result (transcription error).
3. Verify that the sample was prepped correctly.

4. Verify the correct analytical and pre-treatment method was used.
5. Double check all manual calculations, looking for incorrectly calculated results, missing dilution factor, wrong initial and final volumes, etc. Where possible manually calculate the result and compare with the reported result.
6. Compare the age of the calibration to the PT analysis date.
7. Review data associated with all quality control samples for biases. Also evaluate all QC solutions with respect to age, source, storage, and handling.
8. Determine the reasonableness of the data. Verify that all QC parameters were in control. Compare results to established limits to the data quality objectives of the study (i.e. tighter QC required for WS studies).
9. Review standard laboratory techniques used on the sample and all associated QC analyses. Were measurements used in quantitation made volumetrically? Were pipets and volumetric flasks used, or were less stringent techniques employed? Were serial dilutions made during the preparation of the curve?
10. Review analytical conditions, integration, background corrections, analyte resolution, and any confirmation runs.
11. Review calibration ranges. Are they too large for the analysis? An over extended calibration range will appear S-shaped. Check the population of curve points in the area of the analyte concentration.
12. Review calibration type (linear, average, response factor, polynomial non-linear, etc.). Reprocess multi-level curve data through a best fit program and if linear, perform a residuals analysis to identify outlier calibration points. If the result was quantitated using an average response factor, compare with the best-fit information and confirm justification for use of the average response factor quantitation.

In general, there are three major areas where corrective action is required. These categories are described below. Non-Conformance Reports are required on indications flagged with a *. Other indications may require a Non-Conformance Report based on the circumstances.

4.7.1 Quality Control Failures

These are usually handled within the laboratory by the analyst.

Indications of Non-Conformance

- Blanks, laboratory control, or spiked samples contain contamination greater than acceptable levels.
- Suspicious trends in spike recoveries or relative percent differences (RPD) between duplicates.
- Initial instrument blank, initial calibration standards, QC check standards, continuing calibration standard spikes, or method blanks are outside acceptance criteria.
- The method blank or instrument blank analysis exceeds the detection limit for the analyte.

Recommended Corrective Action

- Prepare another instrument blank. If the response is still greater than the reporting limit, look for sources of contamination in reagents, the laboratory working environment, and the instrument.
- Reanalyze standard. If results are still unacceptable, prepare new standards. If necessary obtain new primary standards.
- Reanalyze continuing calibration standard. If necessary, recalibrate and reanalyze samples since last successful continuing calibration.
- Evaluate preparation of spikes, spiking techniques, spiking equipment and materials.

4.7.2 Procedural Failures

These are usually handled by the laboratory area manager and the quality assurance department.

Indications of Non-Conformance

- There are unusual changes in detection limits.
- Statistical quality control data is demonstrating unacceptable trends or is outside the warning or acceptance limits.
- Deficiencies are evidenced on performance evaluation samples or internal or external audits.
- Clients express concern about the quality of their data.

Recommended Corrective Action

- Review the method with the analyst.
- Reanalyze the samples and evaluate the results.
- Recalibrate the instrument or analysis method with freshly prepared standards and reanalyze the samples.
- Re-extract and reanalyze the samples per the method.
- Evaluate the data and sample behavior and investigate any possible chemical interferences.
- Re-run the samples using the method of standard additions.
- Check the instrument for possible maintenance deficiencies.
- Seek additional help from other analysts or provide additional training for personnel involved.
- Perform a system audit to evaluate corrective action measures.

4.7.3 Test Specification Failures

These are usually handled by the analyst, laboratory area manager, and the quality assurance department.

Indications of Non-Conformance

- Quality control check standard data is outside the acceptance limits defined for that analyte.

Recommended Corrective Action

- Review the method with the analyst.
- Reanalyze the check standard and evaluate the results.
- Prepare fresh check standard or new primary standard.
- Recalibrate the instrument or analysis method.
- Switch to a different standard vendor.
- Investigate possible chemical interferences.
- Check the instrument for possible maintenance deficiencies.
- Retrain the analyst.

4.7.4 Customer Complaints

The Quality Assurance Department coordinates with the client services staff to receive quality feedback from clients. It is the responsibility of the QA department to communicate any customer complaints to the laboratory operating areas and to follow-up on corrective action taken to prevent a recurrence.

4.8 PROCEDURES FOR PREVENTIVE ACTION

Changes and enhancements to existing policies and procedures are not always made based on the result of failing analytical performance or other non-conformances. Borderline performance, equipment changes/modernization, or outdated internal procedures are all areas that may require modification or enhancement. Employees are encouraged to analyze internal procedures of all kinds, and offer suggestions for improvement. A Preventive Action Investigation form exists for this purpose (Appendix AI). The form is used to record a description of the existing procedure and a proposed solution, an action plan and systematic implementation schedule, and a follow-up section to monitor the effectiveness of any resulting changes.

All Preventive Action Investigations are loaded into a database similar to that used to track non-conformances

4.9 DEPARTURE FROM DOCUMENTED PROCEDURES

4.9.1 Management Policies

Any departure from a laboratory written standard operating procedure not directly involving sample analysis or processing must be approved by the area manager. The area manager must file a Non-Conformance Investigation Report. The Non-Conformance Investigation Report must be included as part of the data package.

Any departure from a SOP involving sample processing or sample analysis must be justified in writing by the analyst and laboratory area manager. The prior written approval of the laboratory president must be received before performing the analysis. The laboratory president must also file a Non-

Conformance Investigation Report. This Non-Conformance Investigation Report must be included as part of the data package (the exception to this requirement is those items in the analytical methods where a written justification for technical and scientific reasons has been determined by the analyst and approved by the Laboratory President as a deviation from the analytical method).

4.9.2 Method Modification and Variances

Modification of, and variances in, analytical methods, except for the deviations justified in writing and approved per section 4.9.1, are strictly prohibited.

4.10 PERFORMANCE AND SYSTEM AUDITS

4.10.1 Internal Audits

Annually the laboratory will be audited by the quality assurance department to verify compliance to ISO-17025 and various State and Federal requirements. Additionally, quarterly internal audits will be conducted by the quality assurance department. Together these audits will encompass all elements of the quality system. A formal written follow-up will be conducted after every internal audit to verify that any deficiencies cited have been corrected, and that the corrective actions have been successful. The following areas will be included in the required internal audits.

4.10.1.1 System Audits

System audits are used to determine that each component within a laboratory system is functioning properly and adheres to the appropriate standard operating procedures, analytical methods, and requirements of the Quality Assurance Manual. Systems to be audited include:

A). Sample Handling and Control
B). Sample Analysis

C). Records Processing and Control
D). Support Systems (such as air handling, DI water, analytical balances, raw materials, etc.)

If during the course of an internal audit, problems were uncovered that may have impacted the laboratories ability to generate quality data, written notification must be provided to all impacted clients. Impacted clients include all those clients who received results from samples analyzed during the time frame the problem occurred. This is accomplished by a letter explaining the problem, and includes revised copies of the report that, if necessary, include any required data qualifiers.

4.10.1.2 Documentation Audits

The Quality Assurance department also performs audits of the laboratory documentation (laboratory notebooks, benchsheets, instrument run logs, client file folders, etc.) to assess the thoroughness and completeness of the documents.

4.10.1.3 Surveillance Audits

The Quality Assurance department, Area Manager, or their designate observes an analyst in detail as a test is being performed. Attention is given to general laboratory demeanor (orderliness, cleanliness, good laboratory practices in measuring, documentation, etc.) as well as to adherence to analytical methods and standard operating procedures.

4.10.1.4 Quality Assurance Reports to Management

The Quality Assurance Manager provides the Laboratory President with a copy of all external audit reports. The report details any deficiencies identified as well as recommended corrective actions.

4.10.2 External Audits

4.10.2.1 On-Site Audits

Audits of the laboratory conducted by regulating agencies and client organizations are to be perceived by the laboratory staff as learning experiences and opportunities to hear suggestions from knowledgeable persons on how operations might be improved. Consequently, the laboratory staff is to be open and cooperative with external auditors. Formal follow-up using written summaries of external audits is to be carried out to ensure that any suggested improvements are thoroughly evaluated.

Figure 4-1
Documentation System Structure

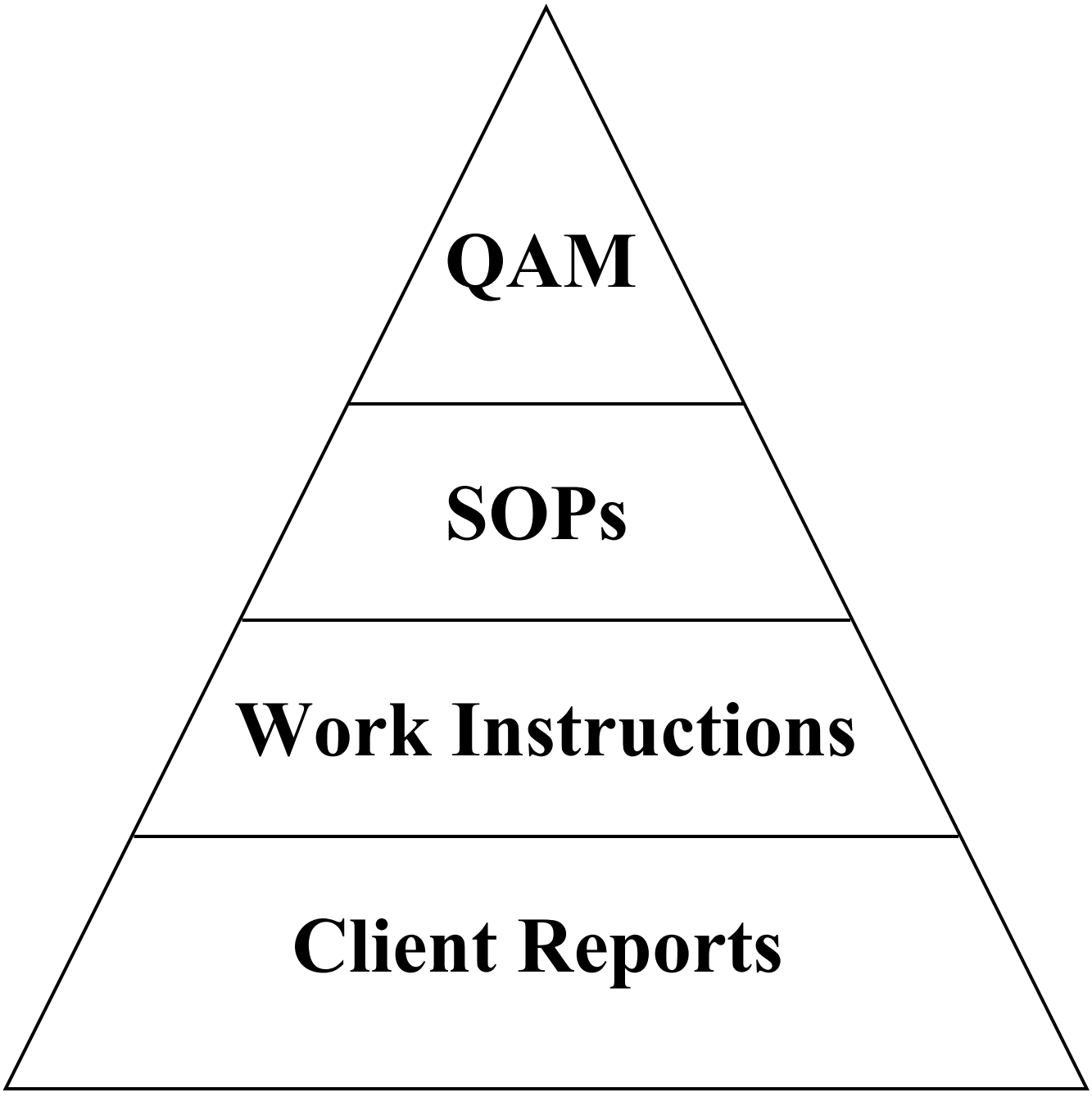


Figure 4-2
Document – Benchsheets/Client Report
Flow Diagram

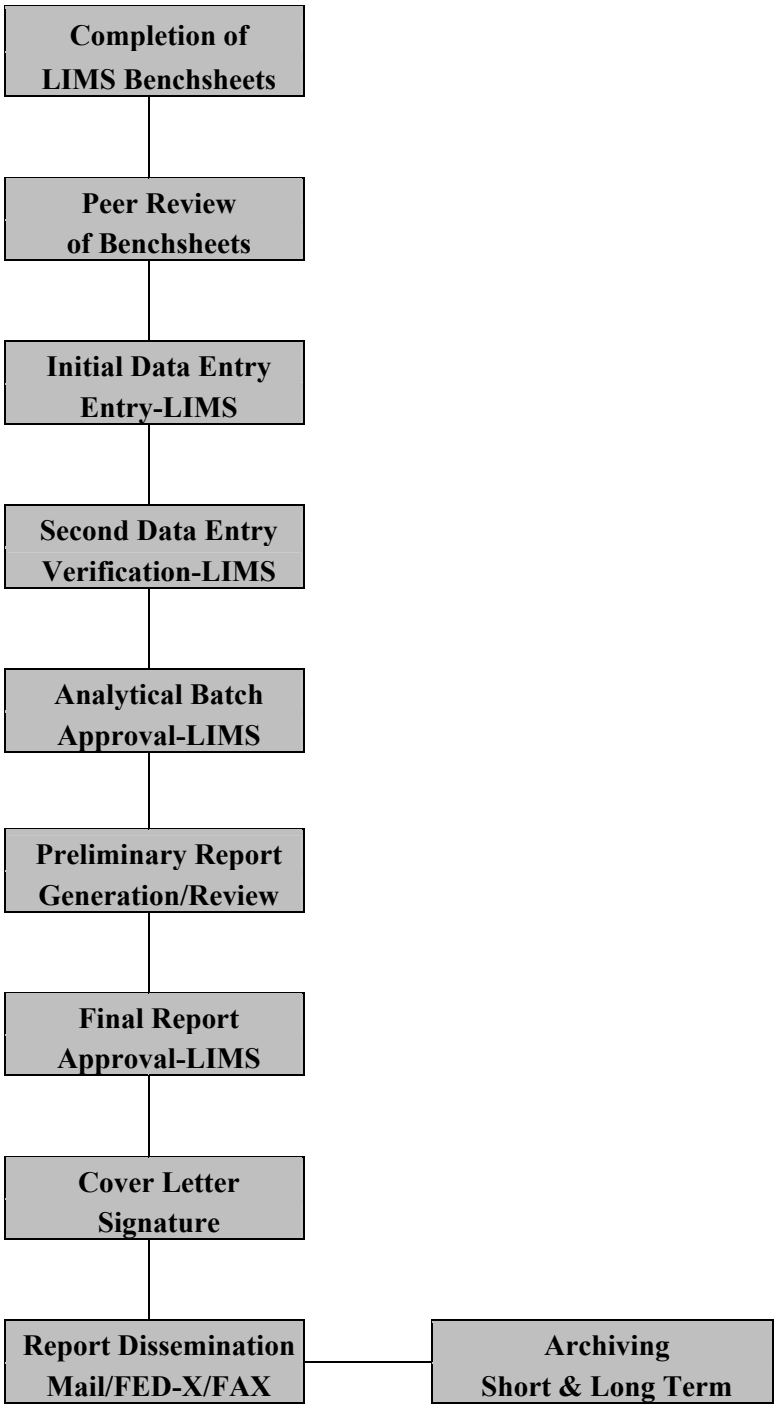
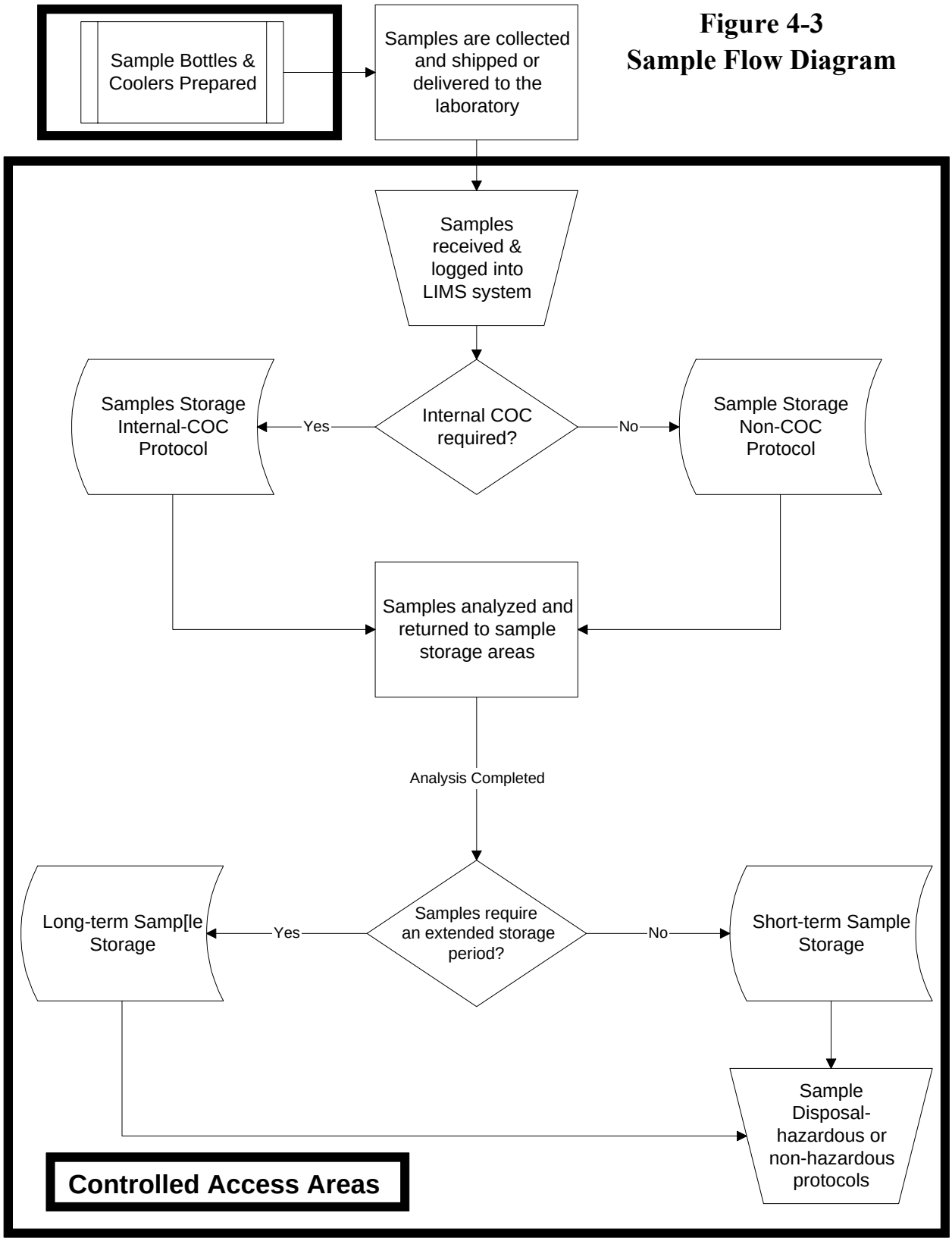


Figure 4-3
Sample Flow Diagram



5.0 REFERENCES

- Methods for Chemical Analysis of Water and Wastes; EPA-600/4-79-020 most current revision.
- Standard Methods for the Evaluation of Water and Wastewater; Current Edition, APHA, AWWA, WPCF.
- Handbook for Analytical Quality Assurance in Water and Wastewater Laboratories; EPA 600/4-79-019, most current revision.
- Physical and Chemical Methods for the Evaluation of Solid Waste; EPA-SW-846, most current revision.
- Guidelines Establishing Text Procedures for the Analysis of Pollutants; 40 CFR; Parts 100 to 149, Current Edition.
- Good Automated Laboratory Practices; USEPA Office of Administration and Resource Management, most current revision.

TABLE 1
Default Data Archiving Systems

Document Archives

Document Description	Storage Location	Storage Duration
Laboratory benchsheets	on-site	1 year
Laboratory benchsheets	off-site	6 years
Instrument Print-Outs (raw data)	on-site	1 year
Instrument Print-Outs (raw data)	off-site	6 years
Laboratory Logs (run, maintenance, analyst)	on-site	1 year
Laboratory Logs (run, maintenance, analyst)	off-site	6 years
Client Files (reports, correspondence, invoices)	on-site	1 year
Client Files (reports, correspondence, invoices)	off-site	6 years
Proposal Files	on-site	5 years
Purchase Agreements	on-site	5 years
SOPs	on-site	5 years

Electronic Archives

File Description	Storage Location	Storage Duration	Storage Media
Instrument Data Files-GC/MS	on-site	1 year	Compact Disk
Instrument Data Files-GC/MS (copy)	off-site	10 years	Compact Disk
Instrument Data files-GC (Turbochrom)	on-site	1 year	Compact Disk
Instrument Data files-GC (Turbochrom) (copy)	off-site	10 years	Compact Disk
Instrument Data files-AA, ICP, ICP/MS	on-site	1 year	Compact Disk
Instrument Data files-AA, ICP, ICP/MS (copy)	off-site	10 years	Compact Disk
Instrument Data files-Auto Analyzer	on-site	1 year	Compact Disk
Instrument Data files-Auto Analyzer (copy)	off-site	10 years	Compact Disk
LIMS daily backup	on-site fire-safe	30 day rotation	DAT-Tape
SOPs	on-site	indefinitely	Compact Disk

TABLE 2
Laboratory SOP Categories

Trace Metals
Gas Chromatograph
Spectrophotometric Procedures
Gravimetric Procedures
Extractions-Organic
Sales and Customer Service
Laboratory Computer Operations
Sample Receiving, Storage, & Disposal
Bottle Prep
Microbiology
Waste Characterization
Instrumental-General
Gas Chromatography/Mass Spectroscopy
Titrimetric Procedures
Electrochemical/Potentiometric Procedures
Quality Assurance
Business and Accounting
Laboratory Safety and Security
Miscellaneous
Inorganic-General

TABLE 3
Field Equipment Calibration

Equipment	Method Reference	Minimum # Standards Initial Calibration	Type of Curve	Frequency of Calibration	Acceptance/ Rejection Criteria Initial Calibration	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
Conductivity Meter	SW-846 Method 9050	2	---	Initial	± 5% of Value	Daily	---
Dissolved Oxygen Meter	Standard Method 4500-O G.	---	---	Initial	± 5% of Value	Daily	---
Temperature Probes	Standard Method 2550 B.	---	---	Initial	± 5% of Value	Daily	---
pH Meter	SW-846 Method 9040	3	Linearity	Initial	Adjust slope to within ±0.05 pH units accuracy	Daily	---

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
Mercury Cold Vapor AA	SW-846 7470/7471 EPA 245.1	5	Correlation coefficient must be ≥0.995	Daily, at the beginning of every analytical batch, and when CCV fails acceptance criteria	Every calibration	90-110% recovery 95-105% recovery	Every 10 samples	90-110% recovery 90-110% recovery
ICP	SW-846 6010 EPA 200.7	3	same as above	same as above	same as above	95-105% recovery	same as above	90-110% recovery
ICP/MS	SW-846 6020 EPA 200.8	6	same as above	same as above	same as above	90-110% recovery	same as above	90-110% recovery
Ion Chromatograph	SW-846 9056 EPA 300.1	6	Correlation coefficient must be ≥0.995	Every 6 months or when CCV fails	Every calibration	90-110% recovery	Every 10 samples	90-110% recovery
Konelab: Sulfate Chloride	EPA 600/4-79-020 Method 375.2 Method 325.2	 10 8	same as above	Every batch	same as above	85-115% recovery	Every 10 samples	85-115% recovery
Phenolics (Total)	SW-846 9065 EPA 420.1	5-7	same as above	same as above	same as above	85-115% recovery	Every 10 samples	85-115% recovery
Cyanide Total and Amenable	SW-846 9012, 9014 EPA 335.1, 335.3, 335.4	7	same as above	same as above	same as above	90-110% recovery	Every 10 samples	90-110% recovery

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
TOC Analyzer-TOC	EPA 415.1	5	same as above	same as above	same as above	85-115% recovery	Every 10 samples	85-115% recovery
GC-PID/ ELCD	SW-846 8021	5 for linear 6 for quadratic	≤20% RSD use average RF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration; ±20% for compounds that boil below 30° C (Bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, and vinyl chloride
	EPA 601/602	3	<10% RSD use average RF or regression, ≥10% must use regression	As needed when CCV fails method Table 2 criteria				Method Table 2 criteria
GC-FID	SW-846 8015	5 for linear 6 for quadratic	≤20% RSD use average CF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
GC-ECD	SW-846 8081 SW-846 8151 SW-846 8082 SW-846 8121 EPA 608 EPA 612	5 for linear 6 for quadratic 3	≤20% RSD use average CF or regression, >20% must use regression <10% RSD use average CF or regression, ≥10% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration; breakdown criteria: DDT <15% Endrin <15%, <20% total
GC-HPLC	SW-846 8310	5 for linear 6 for quadratic	≤20% RSD use average CF or regression, >20% must use regression	As needed, when CCV >15% expected response or concentration	As needed, with analysis of each curve	80-120% recovery	Before and after every 10 samples and at end of each analytical batch	±15% expected response or concentration

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
GC/MS-Volatiles	SW-846 8260	5 for linear 6 for quadratic	CCCs – %RSD ≤30% 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene ethyl benzene, vinyl chloride, all other target analytes ≤15% use average RF for quantitation, otherwise regression SPCCs – average RF ≥ 0.10 for chloromethane, 1,1-dichloroethane and bromoform; ≥ 0.30 for 1,1,2,2-tetrachloroethene and chlorobenzene	As needed, when CCV fails	As needed, with analysis of each curve	80-120% recovery	12 hours	8260: CCCs – % Difference or drift ≤20%, all other target analytes within 20% expected value, high recovery acceptable when analyte not present in sample; SPCCs same criteria as initial calibration
	EPA 624	3	<35% RSD for all compounds use average RF, otherwise use regression				24 hours	Recovery of all analytes must meet recoveries specified in Table 5

TABLE 4
Instrument Calibration

Instrument	Method Reference	Minimum Number Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Second Source Calibration Verification	Acceptance/ Rejection Criteria Second Source Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
GC/MS-Semi-volatiles	SW-846-8270	5 for linear 6 for quadratic	CCCs – %RSD ≤30% acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-diphenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol, 2,4,6-trichlorophenol, all other target analytes ≤15% use average RF for quantitation, otherwise regression SPCCs – average RF ≥0.05 N-nitrosodi-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, 4-nitrophenol	As needed, when CCV fails	As needed, with analysis of each curve	80-120% recovery	12 hours	8270: CCCs % Difference or drift ≤20%; all other target analytes within 20% expected value, high recovery acceptable when analyte not present in sample; SPCCs same criteria as initial calibration
	EPA 625	3	<35% RSD for all compounds use average RF, otherwise use regression				24 hours	80-120% recovery

TABLE 5
Quality Control Sample Types

Blank Type	Abbreviation	Description	Frequency of Use
Method Preparation Blank	MPB	This blank has been carried through the entire analytical process including any pretreatment procedures. The MPB will monitor any contaminants that may affect the sample results. General acceptance limits for the MPB are less than the test reporting Limit. If contamination is detected in the MPB above the reporting limit, all samples with analyte concentrations within 10x that found in the MPB must be flagged for re-extraction or digestion. If it is not possible to re-prep the samples then all analyses for that batch must be qualified.	One per analytical batch

TABLE 5
Quality Control Sample Types

Blank Type	Abbreviation	Description	Frequency of Use
Continuing Calibration Blank	CCB	The continuing calibration blank is a reagent blank that is analyzed as a sample, generally after 10 samples have been tested. The CCB must be run prior to re-zeroing an instrument, unless this practice was performed for each previous sample. The CCB will verify whether significant instrument drift has occurred during the analytical run near the test method detection limit. General acceptance limits are ± the test reporting limit. If the CCB falls outside the acceptance limits, the instrument must be recalibrated and the previous 10 samples reanalyzed. For automated tests where run data is generated after all analyses are completed, 10 samples before and after the unacceptable CCB must be reanalyzed, i.e., all sample results must be encased in acceptable CCB. The reanalysis must also include the ICB and ICV QC samples.	Every ten samples/or as specified in the analytical method.

TABLE 5
Quality Control Sample Types

Blank Type	Abbreviation	Description	Frequency of Use
Field Trip Blank	FTB	These are used with VOA vials where there is the possibility that organic contaminants may diffuse through the PTFE-faced silicone rubber septum of the sample vial. A field trip blank vial filled with organic-free water accompanies the sample containers to and from a client location, at the discretion of the client, may be analyzed along with the samples.	One per sample shipping container
Storage Blank	STB	Reagent-grade water (40 mL aliquot) is stored with samples in a client set. Per the discretion of the client, it may be analyzed after all samples in that set are analyzed. The purpose is to determine the level of contamination acquired during storage.	One per sample storage refrigerator or client sample set (if required)

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Laboratory Fortified Blank or Blank Spike	LFB or BS	This is a fortified method preparation blank in which an aliquot of de-ionized water has been spiked with a known amount of a stock reference standard or spiking solution. A blank spike is required for each digestion or distillation batch. The purpose of the blank spike is to verify the analyst's spiking procedure and assure that any matrix interference shown by the spike and spike duplicate is really matrix induced.	One per analytical batch or as specified in the analytical method

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Second-Source Calibration Verification	SCV	The SCV is identical to the CCV with the exception it must be made from a source dissimilar to that used to prepare the initial calibration curve. The purpose of the SCV is to validate the accuracy both the calibration standards, and the initial calibration curve. Unless otherwise specified by the method, recovery limits for this QC type are typically 80-120%. Sample analysis may not begin prior to the analysis of a successful SCV.	One with every initial calibration

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Continuing Calibration Verification	CCV	The continuing calibration verification standard is generally the standard used as the midpoint of the initial calibration curve. The standard is analyzed and quantitated in the in the same manner as a sample. The CCV will reveal any significant instrument drift. Acceptance limits for this QC type are ± 10%, or as stated in the method. If the CCV falls outside the acceptance window, the instrument must be recalibrated and the previous 10 samples reanalyzed. For automated tests where run data is generated after all analysis is complete, all samples run after the last acceptable CCV must be reanalyzed, i.e. all samples must be bracketed by an acceptable CCV.	Every 10 samples or as specified in the analytical method

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Detection Limit	CRDL	A standard which contains the minimum level of detection acceptable under a contract Statement of Work must be analyzed for particular contract sample sets to demonstrate that detection limit can be met.	One per analytical batch for certain contract sample sets and methods only.
Sample Duplicate	DUP	The sample duplicate is a replicate analysis of a particular sample that has been analyzed previously during the sample analytical batch. The purpose of the duplicate is to monitor precision within the analytical process.	Every 10 samples for each matrix type

TABLE 5
Quality Control Sample Types

Control Type	Abbreviation	Description	Frequency of Use
Sample Matrix Spike	SPK	The sample matrix spike is an aliquot of a sample that has been spiked with a known amount of a stock reference standard or spiking solution. A the purpose of the SPK is to monitor sample matrix effects on the test. Acceptance limits for this QC type are based on the 95% confidence limits established for a test and matrix.	Every 10 samples for each matrix type, or as specified in the analytical method

TABLE 5
Quality Control Sample Types

Matrix QC Type	Abbreviation	Description	Frequency of Use
Matrix Spike Duplicate	MSD	A matrix spike duplicate is an aliquot of the same sample used for the matrix spike (SPK). A spike duplicate is required for each matrix type within a digestion or distillation batch. A spike duplicate analysis may be required on a non-distilled or non-digested sample if the spike has indicated a matrix interference. The purpose of this duplicate spike is to confirm any matrix effects on the test. Acceptance limits for this QC type are based on the 95% confidence limits established for a test and matrix.	Every 10 samples for each matrix type or as specified in the analytical method

TABLE 5
Quality Control Sample Types

Matrix QC Type	Abbreviation	Description	Frequency of Use
Field Duplicate	FDUP	This may be required to evaluate the uniformity of samples and sampling techniques at a field location. Acceptance limits for this QC type are based on established confidence limits, with generally two levels or ranges. The first range extends from the test reporting limit to 10x the test reporting limit. The second range encompasses any values higher than 10x the MDL.	As required on a project basis
Post-Digestion Spike	PDS	The post-digestion spike may be required, on a project basis, when a matrix precludes the use of pre-digestion spike.	One per analytical batch when required by project

TABLE 5
Quality Control Sample Types

Matrix QC Type	Abbreviation	Description	Frequency of Use
Surrogate Spike	SUR	For almost all organic analyses, the analytical method requires surrogate compounds to be added to every blank, sample, matrix spike, matrix spike duplicate, and standard. Surrogate compounds are used to measure analytical efficiency by measuring percent recovery from the known value. They are generally brominated, fluorinated, or isotopically labeled compounds not typically detected in environmental samples.	Every QC and per batch for semi-volatile, volatile, pesticide, PCB analysis
Internal Standard	IST	These are compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), at a known concentration, prior to analysis. Internal standards are used as the basis of quantitation of the target compounds.	Every QC and client sample per batch for volatiles and semi-volatiles

Appendix A

TriMatrix Laboratories, Inc.

CHEMIST I

General Description

Under direct supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties.

Educational/Background Requirements

- Associates degree and 3 or more years of experience in an environmental or related laboratory setting; or

- BS degree in Chemistry or a related field of science.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist I.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).

- Responsible for the daily operation and routine maintenance of instruments and equipment.

- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.

- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.

- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.

- Follow all laboratory safety procedures.

- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

CHEMIST II

General Description

Under ***general*** supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties.

Educational/Background Requirements

- Associates degree and ***5*** or more years of experience in an ***applicable discipline***; or
- BS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline***; or
- ***MS degree in Chemistry or a related field of science***.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist II.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- ***Assist other chemists and technicians with their professional development***.

- ***Act as company advocate by setting a positive example in work habits and attitude to other staff members***.
- ***Demonstrate ability to work independently with minimal errors***.
- ***Capable of conducting peer review on routine data packages***.
- ***Possess the minimum level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements***.
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

CHEMIST III

General Description

Under ***minimal*** supervision of the area manager and group leader, conducts analyses on samples to determine their chemical and/or physical properties. ***Eligible for consideration of group leader status.***

Educational/Background Requirements

- Associates degree and ***7 or more years of experience in an applicable discipline***; or
- BS degree in Chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist III.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine/***non-routine*** maintenance ***and troubleshooting*** of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Assist other chemists and technicians with their professional development.

- Act as company advocate by setting a positive example in work habits and attitude to other staff members.

- Demonstrate ***increased*** ability to work independently with minimal errors.

- Capable of conducting peer review on routine ***and non-routine*** data packages. ***Has demonstrated knowledge to perform final data review and approval on LIMS.***

- Possess ***an above average*** level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- ***Assist in the development and maintenance of laboratory SOPs.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

CHEMIST IV

General Description

Under minimal supervision of the area manager and/***or the technical director***, conducts ***complex*** analyses on samples to determine their chemical and/or physical properties. Eligible for consideration of group leader status.

Educational/Background Requirements

- Associates degree and ***10*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***7*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- ***Ph.D. in Chemistry or a related field of science and experience in an environmental or related laboratory setting***.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist IV.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation ***of, and assisting other chemists in***, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.

- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other chemists and technicians with their professional development ***and in the integration of new methods and technologies***.

- Act as company advocate by setting a positive example in work habits and attitude to other staff members, ***prospective employees, existing and perspective clientele, and the general public***.

- Demonstrate ***superior*** ability to work independently with minimal errors.

- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.

- Possess ***a superior*** level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- ***Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.***

- ***When appropriate, work with the technical director to develop new methods and technologies***.

- ***Develop, review, and update laboratory SOPs as necessary***.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

CHEMIST V

General Description

Under minimal supervision of the area manager and/or the technical director, conducts complex analyses on samples to determine their chemical and/or physical properties. Eligible for consideration of group leader status. ***May work directly with the technical director to develop new methods and technologies for the laboratory***.

Educational/Background Requirements

- Associates degree and ***13*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and ***6*** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and ***2 or more years of*** experience in an environmental or related laboratory setting.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Chemist V.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, assisting other chemists in, ***and serving as the primary reference for***, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.

- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other chemists and technicians with their professional development and in the integration of new methods and technologies.

- Act as company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.

- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.

- ***Responsible for the study and implementation of*** new methods and technologies.

- Develop, review, and update existing laboratory SOPs as necessary, ***write new SOPs as required to reflect advancements in methods and technologies***.

- ***Work with management team to plan for future equipment acquisitions***.

- ***Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions***.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

SENIOR CHEMIST

General Description

Working independently or under minimal supervision of, ***an*** area manager, technical director, ***or the laboratory president***, conducts ***or supervises analysis of complex non-routine projects*** to determine their chemical and/or physical properties. Eligible for consideration of group leader status.

Educational/Background Requirements

- BS degree in Chemistry or a related field of science and ***15*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and ***7*** or more years of experience in an environmental or related laboratory setting.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Senior Chemist.

- Perform analyses in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, assisting other chemists in, and serving as the primary reference for, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent methods following the guidelines established in the test method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to analyses, including but not limited to, standard preparation logbooks, instrument run logbooks, personal notebooks, and instrument maintenance logbooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other chemists and technicians with their professional development and in the integration of new methods and technologies.

- Act as company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Capable of conducting peer review on routine and non-routine data packages. Has demonstrated knowledge to perform final data review and approval on LIMS.

- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in sample throughput, addition of new methods of analysis, and/or operation of additional instruments.

- Responsible for the study and implementation of new methods and technologies.

- Develop, review, and update existing laboratory SOPs as necessary, write new SOPs as required to reflect advancements in methods and technologies.

- Work with management team to plan for future equipment acquisitions.

- Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST I

General Description

Under direct supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs.

Educational/Background Requirements

- Associates degree and 3 or more years of experience in an environmental or related laboratory setting; or
- BS degree in Chemistry or a related field of science.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist I.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness.
- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.
- Prepare proposal outlines for existing clients.
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST II

General Description

Under ***general*** supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs.

Educational/Background Requirements

- Associates degree and ***5*** or more years of experience in an ***applicable discipline;*** or
- BS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline;*** or
- ***MS degree in Chemistry or a related field of science.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist II.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness.
- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.
- Prepare proposal outlines for existing ***and new*** clients.
- ***Assist other project chemists and technicians with their professional development.***

- ***Act as a company advocate by setting a positive example in work habits and attitude to other staff members.***
- ***Demonstrate ability to work independently with minimal errors.***
- ***Posses the minimum level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.***
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST III

General Description

Under ***minimal*** supervision of the client services manager and project chemist group leader, acts as the primary interface with the client to assure laboratory services are meeting client needs. ***Eligible for consideration of group leader status.***

Educational/Background Requirements

- Associates degree and ***7*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist III.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. ***Assist with the preparation, archiving, and delivery of a CLP or "CLP Like" deliverables package.***
- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".
- Follow all laboratory safety procedures.

- ***Prepare and/or coordinate the preparation of proposals for existing and new clients under direct supervision of the client services manager, sales manager, or laboratory president.***
- Assist other project chemists and technicians with their professional development.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members.
- Demonstrate ***increased*** ability to work independently with minimal errors.
- Posses ***an above average*** level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.
- ***Demonstrate ability to improve productivity as shown by an increase in project workload and throughput.***
- ***Provide data interpretation services to clients.***
- ***Assist in the development and maintenance of laboratory SOPs.***
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST IV

General Description

Under minimal supervision of the client services manager and/***or the sales manager,*** acts as the primary interface with the client to assure laboratory services are meeting client needs. ***May work directly with the sales manager to develop increased business from existing clients.*** Eligible for consideration of group leader status.

Educational/Background Requirements

- Associates degree and ***10*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***7*** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- ***Ph.D. in Chemistry or a related field of science and experience in an environmental or related laboratory setting.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist IV.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, ***and assist other project chemists with,*** incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. ***Coordinate*** the preparation, archiving, and delivery of CLP or "CLP Like" deliverables packages.

- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".

- Follow all laboratory safety procedures.

- Prepare and/or coordinate the preparation of proposals for existing and new clients under ***minimum*** supervision of the client services manager, sales manager, or laboratory president.

- Assist other project chemists and technicians with their professional development ***and in the integration of new methods and technologies.***

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members***, prospective employees, existing and perspective clientele, and the general public.***

- Demonstrate ***superior*** ability to work independently with minimal errors.

- Posses ***a superior*** level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput ***as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or "CLP Like" deliverables package and/or managing projects to specifications outlines in QAPPs.***

- Provide data interpretation services to clients.

- ***Develop, review, and update laboratory SOPs as necessary.***

- ***When appropriate, work with sales manager to develop additional business from existing clients.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

PROJECT CHEMIST V

General Description

Under minimal supervision of the client services manager and/or the sales manager, acts as the primary interface with the client to assure laboratory services are meeting client needs. ***Works*** directly with the sales manager to ***establish relationships with new clients as well as increase*** business from existing clients. Eligible for consideration of group leader status.

Educational/Background Requirements

- Associates degree and ***13*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and ***6*** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and ***2 or more years of*** experience in an environmental or related laboratory setting.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Project Chemist V.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, and assist other project chemists with, incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. Coordinate the preparation, archiving, and delivery of CLP or "CLP Like" deliverables packages.

- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".

- Follow all laboratory safety procedures.

- Prepare and/or coordinate the preparation of proposals for existing and new clients under minimum supervision of the client services manager, sales manager, or laboratory president. ***Take an active and substantial role on the marketing team in the development and coordination of large technical and cost proposals, qualifications packages, and marketing literature.***

- Assist other project chemists and technicians with their professional development and ***serve as the primary reference for*** the integration of new methods and technologies.

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Posses a superior level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or "CLP Like" deliverables package and/or managing projects to specifications outlines in QAPPs. ***Improve the productivity of others through training, assistance and the development and implementation of new, more efficient procedures.***

- Provide data interpretation services to clients. ***Assist clients in developing work plans or QAPPs by providing technical and administrative laboratory documentation and/or writing the laboratory portion of QAPPs.***

- Develop, review, and update laboratory SOPs as necessary. ***Write new SOPs as required to reflect advancements in procedures or technologies.***

- ***Routinely*** work with sales manager to develop additional business from existing clients ***and new clients.***

- ***Responsible for the study and implementation of new procedures and technologies.***

- ***Work with management team to plan for future equipment and software acquisitions.***

- ***Provide input to client services manager, sales manager, and/or laboratory president on personnel issues including performance reviews and staff additions / reductions.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

SENIOR PROJECT CHEMIST

General Description

Working independently or under minimal supervision of the client services manager and/or the sales manager***, or laboratory president,*** acts as the primary interface with the client to assure laboratory services are meeting client needs. Works directly with the sales manager to establish relationships with new clients as well as increase business from existing clients. ***Works directly with the laboratory president to develop the laboratory portion of QAPPs, work plans, and other technical documents.*** Eligible for consideration of group leader status.

Educational/Background Requirements

- BS degree in Chemistry or a related field of science and ***15*** or more years of experience in an applicable discipline; or
- MS degree in chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- Ph.D. in Chemistry or a related field of science and ***7*** or more years of experience in an environmental or related laboratory setting.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Senior Project Chemist.

- Perform duties in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Prepare, and assist other project chemists with, incoming projects for laboratory testing. Required tasks include, but are not limited to, timely submittal of properly completed bottle request forms to bottle prep, verification of the accuracy, completeness, and punctuality of filled bottle requests prior to their shipment, and timely problem solving and creation of submittals for sample delivery groups which are received to the lab.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Review all final reports for accuracy and completeness. Coordinate the preparation, archiving, and delivery of CLP or "CLP Like" deliverables packages.

- Maintain files of all applicable documentation pertinent to projects, including but not limited to, quotations, completed bottle request forms, copies of contracts / purchase orders, and all other documentation listed on the "Project File Outline".

- Follow all laboratory safety procedures.

- Prepare and/or coordinate the preparation of proposals for existing and new clients under minimum supervision of the client services manager, sales manager, or laboratory president. Take an active and substantial role on the marketing team in the development and coordination of large technical and cost proposals, qualifications packages, and marketing literature.

- Assist other project chemists and technicians with their professional development and serve as the primary reference for the integration of new methods and technologies.

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and perspective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Posses a superior level of competence in computer skills (Excel, Word, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in project workload and throughput as well as an increased in the complexity of projects and data packages. This includes, but is not limited to, managing projects requiring a CLP or “CLP Like” deliverables package and/or managing projects to specifications outlines in QAPPs. Improve the productivity of others through training, assistance and the development and implementation of new, more efficient procedures.

- Provide data interpretation services to clients. Assist clients in developing work plans or QAPPs by providing technical and administrative laboratory documentation and/or writing the laboratory portion of QAPPs.

- Develop, review, and update laboratory SOPs as necessary. Write new SOPs as required to reflect advancements in procedures or technologies.

- Routinely work with sales manager to develop additional business from existing clients and new clients.

- Responsible for the study and implementation of new procedures and technologies.

- Work with management team to plan for future equipment and software acquisitions.

- Provide input to client services manager, sales manager, and/or laboratory president on personnel issues including performance reviews and staff additions / reductions.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN I

General Description

Under direct supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory.

Educational/Background Requirements

- High school diploma or equivalent.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician I.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- Become completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN II

General Description

Under ***general*** supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory.

Educational/Background Requirements

- High school diploma or equivalent ***and 2 or more years of experience in an applicable discipline;*** or
- ***Associates degree and 1 or more years of experience in an applicable discipline;*** or
- ***BS degree in Chemistry or a related field of science***.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician II.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine maintenance of instruments and equipment.
- ***Remain*** completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.
- ***Assist other technicians with their professional development.***
- ***Act as a company advocate by setting a positive example in work habits and attitude to other staff members.***

- ***Demonstrate ability to work independently with minimal errors.***
- ***Possess the minimum level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.***
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN III

General Description

Under ***minimal*** supervision of the area manager and group leader, performs tasks necessary for efficient operation of the laboratory. ***Eligible for consideration of group leader status.***

Educational/Background Requirements

- High school diploma or equivalent and ***4*** or more years of experience in an applicable discipline; or
- Associates degree and ***3*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***
- ***MS degree in Chemistry or a related field of science.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician III.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation and routine/***non-routine*** maintenance ***and troubleshooting*** of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other technicians with their professional development.
- Act as a company advocate by setting a positive example in work habits and attitude to other staff members.
- Demonstrate ***increased*** ability to work independently with minimal errors.
- Possess ***an above average*** level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.
- ***Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput.***
- ***Assist in the development and maintenance of laboratory SOPs.***
- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN IV

General Description

Under minimal supervision of the area manager and***/or the technical director***, performs ***complex*** tasks necessary for efficient operation of the laboratory. Eligible for consideration of group leader status.

Educational/Background Requirements

- High school diploma or equivalent and ***7*** or more years of experience in an applicable discipline; or
- Associates degree and ***5*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***4*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or a related field of science ***and 2 or more years of experience in an applicable discipline.***

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician IV.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation ***of, and assisting other technicians in,*** routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other technicians with their professional development ***and in the integration of new procedures and technologies.***

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, ***prospective employees, existing and prospective clientele, and the general public***.

- Demonstrate ***superior*** ability to work independently with minimal errors.

- Possess ***a superior*** level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput***, addition of new procedures/technologies and/or operation of additional equipment/instruments.***

- ***When appropriate, work with the technical director, laboratory president, or sales manager to develop new procedures and technologies.***

- ***Develop, review, and update laboratory SOPs as necessary.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

TECHNICIAN V

General Description

Under minimal supervision of the area manager and/or the technical director, performs complex tasks necessary for efficient operation of the laboratory. Eligible for consideration of group leader status. ***May work directly with the technical director, laboratory president, or sales manager to develop methods, procedures, and technologies for the laboratory.***

Educational/Background Requirements

- High school diploma or equivalent and ***10*** or more years of experience in an applicable discipline; or
- Associates degree and ***8*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***6*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or related field of science and ***4*** or more years of experience in an applicable discipline.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Technician V.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, and assisting other technicians in, ***and serving as the primary reference for,*** routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.

- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other technicians with their professional development and in the integration of new procedures and technologies.

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and prospective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput, addition of new procedures/technologies and/or operation of additional equipment/instruments.

- ***Responsible for the study and implementation of*** new procedures and technologies.

- Develop, review, and update laboratory SOPs as necessary***, write new SOPs as required to reflect advancement in procedures and technologies.***

- ***Work with management team to plan for future equipment acquisitions.***

- ***Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.***

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

SENIOR TECHNICIAN

General Description

Working independently or under minimal supervision of***, an*** area manager, technical director, ***or the laboratory president,*** performs ***or supervises tasks related to complex non-routine projects*** necessary for efficient operation of the laboratory. Eligible for consideration of group leader status.

Educational/Background Requirements

- High school diploma or equivalent and ***15*** or more years of experience in an applicable discipline; or
- Associates degree and ***13*** or more years of experience in an applicable discipline; or
- BS degree in Chemistry or a related field of science and ***10*** or more years of experience in an applicable discipline; or
- MS degree in Chemistry or related field of science and ***7*** or more years of experience in an applicable discipline.

Minimum Required Skills and Responsibilities

The following are the minimum skills and responsibilities required of a Senior Technician.

- Perform tasks in an ethical and acceptable manner, as outlined in the TriMatrix Laboratory Code of Ethics, and each applicable Standard Operating Procedure (SOP).
- Responsible for the daily operation of, and assisting other technicians in, and serving as the primary reference for, routine/non-routine maintenance and troubleshooting of instruments and equipment.
- Remain completely familiar with all aspects of the laboratory Quality Assurance Manual. Perform all QA/QC procedures outlined in the laboratory Quality Assurance Manual and the laboratory specific SOPs.
- Perform Demonstration of Capabilities (DOC) for all pertinent procedures following the guidelines established in the method or Quality Assurance Manual.
- Maintain all applicable documentation pertinent to procedures, including but not limited to, procedural and maintenance logbooks and personal notebooks.
- Follow all laboratory safety procedures.
- Maintain adequate supply of all spare parts and consumable supplies to ensure efficient, uninterrupted operation of the laboratory area.

- Assist other technicians with their professional development and in the integration of new procedures and technologies.

- Act as a company advocate by setting a positive example in work habits and attitude to other staff members, prospective employees, existing and prospective clientele, and the general public.

- Demonstrate superior ability to work independently with minimal errors.

- Possess a superior level of competence in computer skills (Excel, Word, instrument software, LIMS, etc.) required to carry out job requirements.

- Demonstrate ability to improve productivity as shown by an increase in process/data/sample throughput, addition of new procedures/technologies and/or operation of additional equipment/instruments.

- Responsible for the study and implementation of new procedures and technologies.

- Develop, review, and update laboratory SOPs as necessary, write new SOPs as required to reflect advancement in procedures and technologies.

- Work with management team to plan for future equipment acquisitions.

- Provide input to area manager/technical director/laboratory president on personnel issues including performance reviews and staff additions/reductions.

- Perform all other activities deemed necessary to management.

TriMatrix Laboratories, Inc.

GROUP LEADER

General Description

In addition to the duties associated with the current chemist level, a group leader also takes on administrative responsibilities involved with the operation of the laboratory area.

Educational/Background Requirements

- Minimum of those specified with a Chemist III.

Minimum Required Skills and Responsibilities

Consistent with current Chemist Level, with additional or increased emphasis on the following requirements.

- Act as the area manager when the area manager is absent, filling such duties as supervision of employees and review and approval of data.
- Act as an additional source of information for management and others regarding laboratory area analysis capabilities.
- Responsible for the scheduling of work and the monitoring of workload for such items as hold times and due dates.
- Provide leadership, guidance, and training to other laboratory personnel on methods, equipment, and quality control.
- Develop, review and update laboratory SOPs as necessary.
- Assure that new methods, policies, and procedures are integrated into the laboratory area.
- Assume a primary responsibility for verifying that sample analyses are adhering to all method and laboratory specified quality assurance parameters.

Appendix B

TriMatrix Laboratories, Inc.

Inorganic Analyses

Parameter	Reference Citation
ACIDITY AS $CaCO_3$	SDM 2310 B
ALKALINITY, BICARBONATE	SDM 2320 B
ALKALINITY, CARBONATE	SDM 2320 B
ALKALINITY, HYDROXIDE	SDM 2320 B
ALKALINITY, PHENOLPHTHALEIN	SDM 2320 B
ALKALINITY, TOTAL	SDM 2320 B
BOD, (5-DAY)	SDM 5210 B
BOD, (5-DAY), DISSOLVED	SDM 5210 B
BOD, CARBONACEOUS (5-DAY)	SDM 5210 B
BROMIDE	USEPA 9056, ASTM D1246-88
CARBON DIOXIDE	SDM 4500-CO_2 C
CARBON, DISSOLVED ORGANIC	USEPA 9060, SDM 5310 D
CARBON, PURGEABLE ORGANIC	USEPA 9060
CARBON, TOTAL INORGANIC	USEPA 9060
CARBON, TOTAL ORGANIC	USEPA 9060, MSA 29.3.5.2, SDM 5310 D
CARBON,ORGANIC(NON-PURGE)	USEPA 9060
CATION EXCHANGE CAPACITY	USEPA-9081
CHEMICAL OXYGEN DEMAND	SDM 5220 D
CHLORIDE	SDM 4500-Cl B, USEPA 300.0/9056
CHLORINE, TOTAL RESIDUAL	HACH-8167
CHROMIUM, HEXAVALENT	SDM 3500-Cr D/USEPA 7196A
COLIFORM, FECAL	SDM 9222 D
COLIFORM, TOTAL	SDM 9223 B
COLOR (APPARENT)	SDM 2120 B
CONDUCTIVITY @ 25*C	USEPA-120.1/9050A, SDM 2510 B
CORROSION TOWARD STEEL	USEPA-1110
CYANIDE, AMENABLE	USEPA-9012A, SDM 4500-CN G
CYANIDE, FREE	USEPA-9014
CYANIDE, WEAK ACID DIS.	SDM-4500-CN I
CYANIDE,TOTAL	USEPA-335.4/9012A
DENSITY	SDM 2710 F
EXTRACTABLE ORGANIC HALIDES-EOX	USEPA-9023
FLUORIDE	USEPA-300.0/9056, SDM 4500-F C
FORMALDEHYDE	USEPA-8315A
GROUNDWATER DEPTH	USGS
GROUNDWATER LEVEL	USGS
HARDNESS, TOTAL	SDM 2340 C
HEM; OIL & GREASE	USEPA-1664/9070A/9071B
HETEROTROPHIC PLATE COUNT	SDM 9215 B
IGNITABILITY, SETAFLASH CLOSED-CUP	USEPA-1020A
IRON, FERRIC BY CALCULATION	SDM 3500-Fe D
IRON, FERROUS	SDM 3500-Fe D
NITROCELLULOSE	USARMY BR&D Lab
NITROGEN, AMMONIA	SDM 4500-NH_3 G
NITROGEN, INORGANIC (NH4)	SDM 4500-NH_3 G
NITROGEN, INORGANIC (NO3+NO2)	USEPA-353.2, SDM 4500-NO_3 F
NITROGEN, INORGANIC	USEPA-350.1 + 353.2
NITROGEN, NITRATE	USEPA-300.0/353.2/9056, SDM 4500-NO_3 F

TriMatrix Laboratories, Inc.

Inorganic Analyses

Parameter	Reference Citation
NITROGEN, NITRATE+NITRITE	USEPA-353.2, SDM 4500-NO_3 F
NITROGEN, NITRITE	USEPA-300.0/353.2/9056, SDM 4500-NO_2 B
NITROGEN, ORG. (NH4)	USEPA-350.1
NITROGEN, ORGANIC	USEPA-351.2
NITROGEN, TOTAL KJELDAHL	USEPA-351.2
ODOR	SDM 2150 B
OXYGEN, DISSOLVED	SDM 4500-O G
PAINT FILTER LIQUIDS TEST	USEPA-9095
PERCENT ASH	USEPA-160.4
PERCENT MOISTURE	SDM 2540 B
PERCENT SOLIDS	SDM 2540 B
PERCENT VOLATILE SOLIDS	USEPA-160.4, SDM 2540 G
PH	USEPA-150.1/9040B/9045C
PHENOLICS, TOTAL	USEPA-420.1/B17420.2/9066
PHOSPHORUS, ORTHO	SDM 4500-P E
PHOSPHORUS, TOTAL	USEPA-365.1, SDM 4500-P F
PHOSPHORUS, TOTAL-SOLUBLE	USEPA-365.1, SDM 4500-P F
RESIDUE, DISSOLVED @ 180C	SDM 2540 C
RESIDUE, DISSOLVED-VOL.	USEPA-160.4
RESIDUE, SUSPENDED	SDM 2540 D
RESIDUE, SUSPENDED-VOL.	USEPA-160.4
RESIDUE, TOTAL	SDM 2540 B
RESIDUE, TOTAL-VOLATILE	USEPA-160.4, SDM 2540 G
SGT-HEM; NON-POLAR MATERIAL	USEPA-1664/9070A/9071B
SILICA, DISSOLVED	SDM 4500-$Si0_2$ D
SODIUM HEXAMETAPHOSPHATE	USEPA-365.1
SPECIFIC GRAVITY	ASTM-D 1429-79, SDM 2710 F
STATIC WATER LEVEL	USGS
SULFATE	USEPA-300.0/375.2/9056/9038, SDM 4500-$S0_4$ F
SULFIDE	USEPA-9034, SDM 4500-S_2 F
SULFIDES, ACID VOLATILE	ET&C VOL 12
SULFITE	SDM 4500-SO_3 B
SURFACTANTS, MBAS	SDM 5540 C
TEMPERATURE	SDM 2550 B
THIOCYANATE	SDM 4500-CN M
TOTAL ORGANIC HALIDES	USEPA-9020B/9023
TURBIDITY	SDM 2130 B

TriMatrix Laboratories, Inc.

Metals Analyses

Parameter	Reference Citation
ALUMINUM, ICP	USEPA-200.7/6010B
ANTIMONY, ICP	USEPA-200.7/6010B
ANTIMONY, MS	USEPA-200.8/6020
ARSENIC, ICP	USEPA-200.7/6010B
ARSENIC, MS	USEPA-200.8/6020
BARIUM, ICP	USEPA-200.7/6010B
BARIUM, MS	USEPA-200.8/6020
BERYLLIUM, ICP	USEPA-200.7/6010B
BERYLLIUM, MS	USEPA-200.8/6020
BORON, ICP	USEPA-200.7/6010B
BORON, MS	USEPA-200.8/6020
CADMIUM, ICP	USEPA-200.7/6010B
CADMIUM, MS	USEPA-200.8/6020
CALCIUM AS $CaCO_3$	USEPA-200.7/6010B
CALCIUM, ICP	USEPA-200.7/6010B
CHROMIUM, ICP	USEPA-200.7/6010B
CHROMIUM, MS	USEPA-200.8/6020
COBALT, ICP	USEPA-200.7/6010B
COBALT, MS	USEPA-200.8/6020
COPPER, ICP	USEPA-200.7/6010B
COPPER, MS	USEPA-200.8/6020
HARDNESS BY CALCULATION, ICP	USEPA-200.7/6010B
IRON, ICP	USEPA-200.7/6010B
LEAD, ICP	USEPA-200.7/6010B
LEAD, MS	USEPA-200.8/6020
LITHIUM, ICP	USEPA-200.7/6010B
MAGNESIUM AS $CaCO_3$, ICP	USEPA-200.7/6010B
MAGNESIUM, ICP	USEPA-200.7/6010B
MANGANESE, ICP	USEPA-200.7/6010B
MANGANESE, MS	USEPA-200.8/6020
MERCURY, COLD VAPOR	USEPA-245.1/7470A/7471A
MOLYBDENUM, ICP	USEPA-200.7/6010B
MOLYBDENUM, MS	USEPA-200.8/6020
NICKEL, ICP	USEPA-200.7/6010B
NICKEL, MS	USEPA-200.8/6020
PHOSPHORUS, ICP	USEPA-200.7/6010B
POTASSIUM, ICP	USEPA-200.7/6010B
SELENIUM, ICP	USEPA-200.7/6010B
SELENIUM, MS	USEPA-200.8/6020
SILICON, ICP	USEPA-200.7/6010B
SILVER, ICP	USEPA-200.7/6010B
SILVER, MS	USEPA-200.8/6020
SODIUM, ICP	USEPA-200.7/6010B
STRONTIUM, TOTAL	USEPA-200.7/6010B
THALLIUM, ICP	USEPA-200.7/6010B
THALLIUM, MS	USEPA-200.8/6020
TIN, ICP	USEPA-200.7/6010B
TIN, MS	USEPA-200.8/6020
TITANIUM, ICP	USEPA-200.7/6010B

TriMatrix Laboratories, Inc.

Metals Analyses

Parameter	Reference Citation
VANADIUM, ICP	USEPA-200.7/6010B
VANADIUM, MS	USEPA-200.8/6020
ZINC, ICP	USEPA-200.7/6010B
ZINC, MS	USEPA-200.8/6020

TriMatrix Laboratories, Inc.

Semi-Volatile Organic Analyses

Parameter	Reference Citation
HPLC ACRYLAMIDE	EPA-8316
GC ORGANOCHLORINE PESTICIDES	USEPA-608/8081A
GC METHOXYCHLOR	USEPA-608.2
HPLC POLYNUCLEAR AROMATIC HYDROCARBONS	USEPA-610/8310
GC/MS BASE/NEUTRAL/ACIDS	USEPA-625/8270C
GC ANALYSIS OF 1,2-DIBROMOMETHANE/ 1,2-DIBROMO-3-CHLOROPROPANE/ 1,2,3-TRICHLOROPROPANE BY MICROEXTRACTION	USEPA-8011
GC DIESEL RANGE ORGANICS	USEPA-8015B, CALIFORNIA LUFT METHOD, WISCONSIN METHOD PUBL-SW-141
GC GLYCOLS	USEPA-8015B
GC POLYCHLORINATED BIPHENYLS	USEPA-8082
GC CHLORINATED HYDROCARBONS	USEPA-8121
GC HERBICIDES	USEPA-8151A
HPLC ALDEHYDES	USEPA-8315A
HPLC NITROAROMATICS AND NITRAMINES	USEPA-8330
HPLC NITROGLYCERINE	USEPA-8332

TriMatrix Laboratories, Inc.

Volatile Organic Analyses

Parameter	Reference Citation
GC GASOLINE RANGE ORGANICS	USEPA-8015B, CALIFORNIA DHS LUFT, IOWA-PA1, WISCONSIN METHOD PUBL-SW-140
GC AIR ANALYSIS	40CFR METHOD 18
GC DISSOLVED HEADSPACE ANALYSIS OF METHANE/ETHANE/ETHYLENE	RSK-175
GC ALCOHOLS	USEPA-8015B
GC VOLATILE ORGANICS	USEPA-601/602/8021B
GC/MS VOLATILE ORGANICS	USEPA-524.2/624/8260B

Appendix C

TriMatrix Laboratories, Inc.

Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
190	Administration	Sonicator - Fisher	F550,# F1520			
200	Administration	Sonicator - Fisher	F550,# F1309			
209	Administration	Mettler PC4400 Toploading Balance				
212	Administration	Mettler AB204 Analytical Balance				
215	Administration	Denver Instrument P-4002 Toploading Balance				
225	Administration	Dionex Accelerated Solvent Extractor	Model ASE 300	8/2003	3070313	New
300	Administration	Fisher Ultrasonic Cleaner	FS110			
301	Administration	Fisher Heated Ultrasonic Cleaner	FS21H			
307	Administration	Denver Instruments Top Loading Balance	Model P-2002	1/2007	P2K2126010	New
317	Client Services	YSI Multi-Parameter Meter	Model 556MPS	2005	05G1614AM	New
318	Client Services	YSI Multi-Parameter Meter	Model 556MPS	2004	04C2296AE	New
319	Client Services	HACH Turbidimeter	Model 2100P		30300030404	New
320	Client Services	Fisher Accumet Multi-Parameter Meter	Model AP84		274436	New
100	Wet Chemistry	Orion pH/ISE Meter	8102			
120	Wet Chemistry	Ultraviolet Spectrophotometer Shimadzu	1601			
161	Wet Chemistry	Auto-analyzer Lachat Quick Chem				
164	Wet Chemistry	pH/ISE meter Orion	710A			
165	Wet Chemistry	Expandable Ion Analyzer Orion Research	EA920			
167	Wet Chemistry	Spectrophotometer (UV-VIS) Shimadzu	1201			
171	Wet Chemistry	Polarograph EG&G Princeton Applied Research	384B			
176	Wet Chemistry	Field Meter				
177	Wet Chemistry	Mettler AE200 Analytical Balance				
178	Wet Chemistry	Turbidimeter Hach	2100N			
186	Wet Chemistry	Koehler Rapid Tester Flashpoint Tester	RT-1			
187	Wet Chemistry	Mettler DL12 Auto-Titrator				
188	Wet Chemistry	YSI Conductivity Meter	3200			
189	Wet Chemistry	Lachat	FIA-8000			
194	Wet Chemistry	Total Organic Halogen: ThermoGlass	1200			
196	Wet Chemistry	Lachat IC	8000			
198	Wet Chemistry	Total Organic Carbon Analyzer, OI Analytical	1010			
205	Wet Chemistry	OHAUS TP4KD Toploading Balance				
206	Wet Chemistry	Mettler BB600 Toploading Balance				
207	Wet Chemistry	Denver Instrument A-250 Analytical Balance				
208	Wet Chemistry	Mettler AE163 Analytical Balance				

TriMatrix Laboratories, Inc.

Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
210	Wet Chemistry	Mettler AE163 Analytical Balance				
298	Wet Chemistry	Konelab Automated Ultraviolet Spectrophotometer	Model Aqua 20			
299	Wet Chemistry	OIC Available Cyanide Analyzer				
303	Wet Chemistry	Konelab Automated Ultraviolet Spectrophotometer	Model 20	1/2006	24618583	Refurb
305	Wet Chemistry	Orion 3-Star Benchtop DO Meter	Model 1113000	2/2006	7383	New
306	Wet Chemistry	Dionex Ion Chromatograph	Model ICS-2000	3/2006	6020239	New
309	Wet Chemistry	pH Meter, Fisher Accumet Basic	Model AB15	3/28/2007	AB92325491	New
310	Wet Chemistry	Market Forge Autoclave	Model STM-E	3/30/2007	226071	New
313	Wet Chemistry	HACH Turbidimeter	Model 2100N	8/1/2008	07060C022389	New
314	Wet Chemistry	VWR Forced Air Oven	Model 1370FM	10/3/2007	4104307	New
315	Wet Chemistry	Thermo Scientific TOX Analyzer	Model ECS 1200	10/18/2007	2003.481	New
321	Wet Chemistry	BW Technologies	Gas Alert Micro		Propoerty of Kent Couty DPW	
322	Wet Chemistry	Chemetrics VVR	Photometer		5121	
324	Wet Chemistry	OI Analytical TOC Analyzer	Aurora Model 1030	2/2008	E750730372E	New
326	Wet Chemistry	OI Analytical Automated Chemistry Analyzer	Flow Solution 3100 (322689/323898)	9/2008	821831887/826833549	New
198T	Wet Chemistry					
305a	Wet Chemistry	Orion DO Probe	Model 081010MD	2/2006	Lot Number RJS16	New
312a	Wet Chemistry	HACH Portable Multi-meter (pH/Cond/Sal/TDS/LDO)	Model HQ40d	7/1/2007	70700010664	New
312b	Wet Chemistry	HACH Portable Multi-meter (pH/Cond/Sal/TDS/LDO)	Model HQ40d	7/1/2007	70700010664	New
324T	Wet Chemistry	OI Analytical TOC Analyzer	Aurora Model 1030	6/17/2008		Loaner
101	Metals	ICP Spectrophotometer PE	Optima 3000			
106	Metals	Atomic Absorption Spectrophotometer Furnace	3			
114	Metals	ICP Mass Spectrometer	ELAN 6000			
116	Metals	Perkin Elmer Optima Trace ICP	3300 DV			
201	Metals	ICP Mass Spectrometer	ELAN 6100			
202	Metals	PSA Low-Level Mercury Analyzer	Millenium System			
203	Metals	A&D FX-2000 Toploading Balance				
211	Metals	Mettler PB1502 Toploading Balance				
216	Metals	PSA Cold Vapor AA Mercury Analyzer	Millenium System			
217	Metals	Env. Express Hotblock, CS154		2000	424CEC0564	
218	Metals	Env. Express Hotblock, CS154		2000	944CEC1008	
219	Metals	Env. Express Hotblock, CS154		2002	1423CEC1147	
220	Metals	Env. Express Hotblock, CS154		2002	1423CEC1113	
311	Metals	Perkin Elmer Optima ICP-OES	Model 5300DV	5/7/2007	077C7032601	New

TriMatrix Laboratories, Inc.

Equipment List

Inst. #	Department	Description	Model Number	Date Purchased	Serial Number	Condition When Purchased
316	Metals	Mettler Analytical Balance	Model XS204	12/21/2007	1128261601	New
144	Semivolatiles GC	Gas Chromatograph (Dual ECD)	HP-5890A			
151	Semivolatiles GC	Liquid Chromatograph Perkin Elmer	PDA 235/240 HPLC			
157	Semivolatiles GC	Gas Chromatograph HP	5890A (FID)			
158	Semivolatiles GC	Gas Chromatograph HP	5890A (ECD)			
159	Semivolatiles GC	Gas Chromatograph Varian	3400 (FID)-SV			
174	Semivolatiles GC	Gas Chromatograph HP	5890 (ECD/FID)			
199	Semivolatiles GC	Gas Chromatograph HP-6890 Dual ECD				
221	Semivolatiles GC	Perkin-Elmer 200 LC Plus HPLC				
222	Semivolatiles GC	Agilent 6890 Dual ECD				
325	Semivolatiles GC	Clarus Gas Chromatograph	Model 500	7/2008	5564	New
133	Semivolatiles MS	GC/MS Varian Ion Trap	Saturn II			
138	Semivolatiles MS	GC/MS Varian Ion Trap	Saturn II			
195	Semivolatiles MS	HP 5973 Quadrupole Mass Spectrometer				
304	Semivolatiles MS	MS = Agilent MSD	Model 5975B	9/7/2006	MS = US60522528 GC = HP 6890N SN US10626080	New
308	Semivolatiles MS	MS = Agilent MSD	Model 5975B	2/2007	US65125179 GC = HP 6890N SN CN10703067	New
117	Volatiles GC	Agilent PID/ELCD GC	6890			
140	Volatiles GC	HP GC	5890 Series II			
142	Volatiles GC	HP GC	5890 Series II			
132	Volatiles MS	GC/MS Varian Ion Trap	Saturn II			
139	Volatiles MS	HP Quadrupole GC/MS	5971			
145	Volatiles MS	HP Quadrupole GC/MS	GC-5890/MS-5971			
197	Volatiles MS	HP 5973 Quadrupole GC/MS				
204	Volatiles MS	Mettler BB2440 Toploading Balance				
224	Volatiles MS	Agilent 5973 Inert MSD				
302	Volatiles MS	Branson Heated Ultrasonic Cleaner	3210R-MTH			
323	Volatiles MS	Agilent GC/MS	Model 6890/5973 Inert	2/21/2008	CN10426060/US35120404	New

Appendix D

TriMatrix Laboratories, Inc.

New Employee Orientation Checklist

Name: _____ Employee #: _____ Date of Hire: ____/____/____

I. Personnel Information Review (Human Resources Manager)

Reviewed (☑)	Item
	Employee Information Sheet Completed
	I-9 Employment Eligibility Verification Form Completed
	W-4 Forms Completed
	Employee Benefits Reviewed
	Direct Deposit Forms Initiated
	Details of Compensation Reviewed
	Key Fob to the Facility Provided (Number _____)
	Employee Handbook Distributed
	Code of Ethics / Data Integrity Policy Agreement Form Signed and Collected. Violation of Ethics Policy Explained.

Signatures below attest that all the information or items described above have been discussed/provided:

____/____/____ _____ ____/____/____ _____

Human Resources Manager Signature Employee Signature

TriMatrix Laboratories, Inc.

New Employee Orientation Checklist

II. Quality Assurance Training (Quality Assurance Officer)

Reviewed (☑)	Item
	Initial and Continuing Demonstration of Capability Requirements Reviewed
	Corrective Action (Non-Conformance) Investigation Procedure Reviewed
	Error Correction Policy Reviewed
	Code of Ethics/Data Integrity Policies Explained
	Initials Added to the Initials Logbook
	Training Forms Initiated for the Following Documents: QA Manual Corrective Action SOP, GR-10-106 or GR-03-101 or GR-03-124 Manual Integration SOP, GR-10-115 General Guidelines for Data Validation and Reporting, GR-10-103 Internal Chain-of-Custody, GR-10-104 Data Confidentiality, GR-10-118

Signatures below attest that all the information or items described above have been discussed/provided:

____/____/____ _____ ____/____/____ _____

Quality Assurance Officer Signature Employee Signature

TriMatrix Laboratories, Inc.

New Employee Orientation Checklist

III. Safety Training (Health and Safety Officer)

Reviewed (☑)	Item
	MSDS Location Discussed
	Safety Walk/Safety Equipment Review, First-Aid Cabinet Locations Identified
	Safety Exam Explained-First two of thirteen videos completed (others to be completed on own during normal working hours)
	Training Forms Initiated for the Following Documents: Chemical Hygiene Plan Safety Manual Copy Emergency Action Plan Copy
	Safety Glasses Ordered or Distributed

Signatures below attest that all the information or items described above have been discussed/provided:

____/____/____ _____ ____/____/____

Appendix E

TriMatrix Laboratories, Inc.

CODE OF ETHICS / DATA INTEGRITY AGREEMENT

All full time, part time and contracted employees working for TriMatrix Laboratories, Inc. are required to make every effort to conduct quality work with data integrity, ethical practices and professionalism. To ensure strength in the individual, in the laboratory organization and in client relationships, each employee must be aware of the following company policies:

I. Each TriMatrix employee is responsible for the propriety and consequences of his or her actions when representing the laboratory through sample analysis, data review, adherence to policies and procedures, client /vender relationships, other employees and/or visitors.

II. All aspects of company business must be conducted in an ethical, legal and professional manner, and in compliance with all applicable federal, state and local laws and regulations.

III. Under no circumstances must client confidentiality be compromised or any information regarding the client be revealed to another agency without the client's prior written permission.

IV. Gratuities, gifts and/or rewards provided by clients or vendors are laboratory property and may not be kept for personal use without written approval.

V. Reporting of data integrity issues is encouraged. Reporting shall be kept confidential when anonymity is requested and/or required.

Additionally, violations of the data integrity/code of ethics policy may result in immediate termination of employment with TriMatrix Laboratories, Inc. Such violations include the following:

A. Intentionally misrepresenting laboratory data in any manner.

B. Intentionally misapplying any date and/or time.

C. Intentional representation of another employee without written approval.

D. Intentional omission of any information, fact or datum.

E. Intentional deviation from or shortcut through a procedure without written approval.

A highly ethical approach to laboratory analysis/reporting is a key component of the TriMatrix laboratory objective. This approach is backed by management in providing the facilities, equipment and time necessary minimize undue pressures to make compromises, whether such pressures be internal or external.

AGREEMENT STATEMENT

I have read and understood the Code of Ethics/Data Integrity Agreement, and agree to abide by all policies stated. I understand that violation of these policies may result in severe consequences up to and including termination of my employment with TriMatrix Laboratories, Inc.

Employee (print name)	Signature	Date

Appendix F

TriMatrix Laboratories, Inc.

New Instrument Accuracy Study

Instrument Number: ________________ **Method Reference:** __________________________ **Analyst:** _________________ **Date Analyzed:** _________________

Compound	Analysis #1		Analysis #2		Analysis #3		Analysis #4		Analysis #5		Analysis #6		Analysis #7	
	Conc.:		Conc.:		Conc.:		Conc.:		Conc.:		Conc.:		Conc.:	
	Units:		Units:		Units:		Units:		Units:		Units:		Units:	
	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery	Amount Found	Percent Recovery

TriMatrix Laboratories, Inc.

New Instrument Accuracy Study

Instrument Number: _____ **Method Reference:** _____ **Analyst:** _____ **Date Analyzed:** _____

Compound	Standard Deviation of Percent Recoveries	Standard Deviation Window	Pass/ Fail Standard Deviation	Average Percent Recovery	Percent Recovery Window	Pass/ Fail Percent Recovery	Overall Pass/ Fail

Appendix G

TriMatrix Laboratories, Inc.

New Instrument Information and Initial Demonstration of Capability

Item:		**Serial Number:**	
Manufacturer:		**Date Received:**	
Model:		**Location:**	

Initial Demonstration of Capability Passed: Yes / No / NA

Date Initial Demonstration of Capability Completed:

Initial Demonstration of Capability Data Attached: Yes / No / NA

Adequate Sensitivity Achieved (LFB or MDL Completed): Yes / No / NA

LFB or MDL Documentation Attached: Yes / No / NA

Date LFB or MDL Completed:

Linear Range Developed and Demonstrated Yes / No / NA

Linear Range Development Information Attached: Yes / No / NA

Notes:

Approvals and Assigned Instrument Number

Quality Assurance Manager	**Laboratory Area Manager**

TriMatrix Instrument Number: **Date In Service:**

Appendix H

TriMatrix Laboratories, Inc.

********** LABORATORY
DEMONSTRATION OF CAPABILITY FOR **********

Parameter	Date Analyzed	Method	Inst. Number	Units	Amount Spiked	Cert. #1 Amount Found	Cert. #2 Amount Found	Cert. #3 Amount Found	Cert. #4 Amount Found	Average Percent Recovery	Percent Recovery Window	Pass/Fail Percent Recovery	Percent RSD	Percent RSD Window	Percent RSD Pass/Fail	Overall Pass/ Fail

Appendix I

TriMatrix Laboratories, Inc.

INORGANIC LABORATORY
DEMONSTRATION OF CAPABILITY

Parameter: Percent Solids

Trainer: John Doe

Method: SW-846 3550B/GR-07-115

Trainee: John Smith

Analyst	Date	Run #1	Run #2	Run #3	Run #4	Units	Inst. #	Standard Deviation	Average	Degrees of Freedom D	Experimental Student's t Value	Tabular Student's t Value	Are the Two Sets of Results Statistically the Same AND RSDs<20?
John Doe	12/31/02	48.3	55.6	44.2	47.5	%	117	4.81	48.9	5.48	0.227	4.032	YES(PASS)
John Smith	12/31/02	45.9	50.2	52.1	44.7	%	117	3.50	48.2				

Appendix J

TriMatrix Laboratories, Inc.

NELAC Demonstration of Capability Certification Statement

Employee Name:_____ **Date:**_____

Method Name(s), Number(s), and Revision(s):

_____

Matrix:_____ **Analyte(s) or Parameter(s):**_____

SOP Number:_____ **Revision Number:**_____

We, the undersigned, CERTIFY that:

Yes / NA

☐☐ 1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

☐☐ 2. The test method(s) was performed by the analyst identified on this certification.

☐☐ 3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

☐☐ 4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

> With *true* meaning consistent with supporting data; *accurate* meaning based on good laboratory practices consistent with sound scientific principles/practices; *complete* meaning includes the results of all supporting performance testing; and *self explanatory* meaning data properly labeled and stored so that the results are clear and require no additional explanation.

☐☐ 5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

This certification form must be completed each time an Initial Demonstration of Capability study is performed, or when a Continuing Demonstration of Capability study is performed in conjunction with a revised SOP.

_____ _____

Area Supervisor
Heather L. Brady — Date

_____ _____

Quality Assurance Department
Tom C. Boocher — Date

Appendix K

TriMatrix Laboratories, Inc.

LABORATORY TRAINING CHECKLIST

Employee Name: _____

Instructor Name: _____

Method Number(s) and Revision(s): _____

SOP Name, Number, and Revision: _____

Applicable Matrices: _____

n/a	Trainer/Trainee Initials	CheckPoint Item
❑		1) The employee has read the method and the standard operating procedure.
❑		2) The instructor has reviewed the method and the procedure with the employee.
❑		3) The instructor has performed a manual demonstration of the procedure.
❑		4) The employee has correctly performed the procedure under direct supervision.
❑		5) The employee has correctly performed the procedure without direct supervision.
❑		6) The employee has successfully and exclusively completed an Initial Demonstration of Capability (IDC).
❑		7) The DoC spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
☒	------- -------	8) If applicable, or a MDL study does not yet exist, the employee has successfully completed a MDL study for all applicable matrixes.
☒	------- -------	9) The MDL study spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
❑		10) The employee has been instructed in the QA/QC requirements of this procedure.
❑		11) The employee has been instructed in the proper procedure governing paperflow, benchsheet completion, and other relevant documentation requirements.
❑		12) NELAC Demonstration of Capability Certification Statement is Attached.

The required CheckPoints have been successfully completed, and in my opinion this employee has been adequately trained to correctly perform this procedure.

Instructor: _____ **Date:** _____

I have read and understand the SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.

Employee: _____ **Date:** _____

Appendix L

TriMatrix Laboratories, Inc.

INORGANIC/METALS/SEMI-VOLATILE/VOLATILE LABORATORY
2008 WATER/SOIL
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Instrument Number	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1 Amount Found	Rep. #2 Amount Found	Rep. #3 Amount Found	Rep. #4 Amount Found	Rep. #5 Amount Found	Rep. #6 Amount Found	Rep. #7 Amount Found	Average Amount Found	Average % Recovery	Standard Deviation	MDL

file: MDL 2008 version 10.XLS

revision: 2008.10

INORGANIC/METALS/SEMI-VOLATILE/VOLATILE LABORATORY
2008 WATER/SOIL
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Average Amount Found	Average % Recovery	Standard Deviation	MDL	Amount Spiked	MDL Window	Pass / Fail	Average % Recovery Check	Minimum Report Limit
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
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							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		
							Missing Parameter / Compound		

TriMatrix Laboratories, Inc.

INORGANIC/METALS/SEMI-VOLATILE/VOLATILE LABORATORY
2008 WATER/SOIL
METHOD DETECTION LIMIT VERIFICATION STUDY

Parameter / Compound	Instrument Number	Reference Citation	MDL Result	Units	Date	Analyst	Verification Concentration	MDL/MDL Verification Concentration Difference	MPB Result	MDL Verification Result	MPB/MDL Verification Response Increase	Acceptable (≥3x MPB Response)?

file: MDL 2008 version 10.XLS

revision: 2008.10

Appendix M

TriMatrix Laboratories, Inc.

SOP MAJOR REVISION
LABORATORY TRAINING CHECKLIST

Employee Name: _____

Method Number(s) and SOP Name, Number, and Revision: _____

Applicable Matrices: _____

n/a	Employee Initials	CheckPoint Item
❑		1) I have read the updated method and/or the revised Standard Operating Procedure.
❑		2) I have successfully completed an Initial Demonstration of Capability (IDC).
❑		3) The DoC spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
❑		4) If applicable, or a MDL study does not yet exist, I have successfully completed a MDL study for all applicable matrixes.
❑		5) The MDL study spreadsheet has been completed. The spreadsheet and all supporting analytical data have been attached.
❑		6) I have been instructed in any new QA/QC requirements of this procedure.
❑		7) NELAC Demonstration of Capability Certification Statement is Attached.

The required CheckPoints have been successfully completed.

Date: _____ **Quality Assurance:** _____

I have read and understand the revised SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.

Date: _____ **Employee Signature:** _____

TriMatrix Laboratories, Inc.

SOP MINOR REVISION
LABORATORY TRAINING CHECKLIST

Employee Name: _____

Method Number(s) and Revision(s): _____

SOP Name, Number, and Revision: _____

Applicable Matrices: _____

n/a	Employee Initials	CheckPoint Item
❑		1) I have read and understood the updated method and/or the revised Standard Operating Procedure.
❑		2) I have read and understood any new QA/QC requirements of this procedure.
❑		3) NELAC Demonstration of Capability Certification Statement is Attached.

I have read and understand the revised SOP, understand what is required, and agree to follow it as instructed. I understand that I may not deviate from the SOP without prior approval from management.

Date:_____ **Employee Signature:**_____

The SOP revision has been successfully implemented.

Date:_____ **QA/QC Signature:**_____

Appendix N

TriMatrix Laboratories, Inc.

Container Packing List

For any questions regarding these containers, contact a Project Chemist at (616) 975-4500.

Client: Project:

#	Sets	Sample Locations	Sample Container Types and Quantities Requested																									
			0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26
1																													
2																													
3																													
4																													
5																													
6																													
7																													
8																													
9																													
10																													
11																													
12																													
13																													
14																													
15																													
16																													
17																													
18																													
19																													
20																													
Total Containers																													

This container type requires field-filtering

MATRIX	#	TEST	SIZE (mL) / TYPE CONTAINER	OPTIONS	PRESERVATIVE	TAG COLOR
WATER	0	Unpreserved Purgeable Organics	40 mL Clear Glass Vial	40	Cool to 4° C	Yellow & Black Stripe
	1	Preserved Purgeable Organics	40 mL Clear Glass Vial (pre-preserved)	40	HCl; Cool to 4° C	Yellow
	2	Non-Purgeable Organics	1000 mL Amber Glass	1000	Cool to 4° C	Salmon
	3	General Short Hold	Plastic	125, 250, 500, 1000	Cool to 4° C	Green
	4	Nutrients	Plastic	125, 250, 500, 1000	pH <2 w/ H_2SO_4	Dark Blue
	5	Cyanides	500 mL Amber Plastic	500	pH >12 w/ NaOH	Light Blue
	6	Total Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red
	7	Oil & Grease/TPH	Clear Glass	1000WM, 1000NM	pH <2 w/ H_2SO_4	Dark Blue
	8	Bacteria	125 mL Plastic (pre-preserved)	125	$Na_2S_2O_3$; Cool to 4° C	Pre-Labeled (White)
	9	Sulfide	500 mL Amber Glass + NaOH ampule	500	Zinc Acetate at Lab; NaOH in Field	Light Green
	10	TOX	250 mL Amber Glass **w/ Septa Lid**	250	pH <2 w/ H_2SO_4	Lilac
	11	TOC	40 mL Amber Vial	40	pH <2 w/ H_2SO_4	Pink
	12	DRO	1000 mL Amber Glass	1000	pH <2 w/ HCl	Gray
	13	Phenols	500 mL Amber Glass	500	pH <2 w/ H_2SO_4	Brown
	14	Formaldehyde	250 mL Amber Glass	250	Cool to 4° C	Orange
	15	Dissolved Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red & White Stripe
SOIL	16	Inorganics/Metals	WM Plastic	125, 250, 500, 1000	Cool to 4° C	White
	17	Non Purgeable Organics	WM Clear Glass	125, 250, 500, 1000	Cool to 4° C	Manila
	18	Purgeable Organics - Bulk	60 mL WM Clear Glass	60	Cool to 4° C	Light Yellow
	19	TCLP Volatiles	125 mL Clear Glass Vial	125	Cool to 4° C	Yellow & Black Stripe
	20	% Solids	125 mL WM Plastic	125	Cool to 4° C	Yellow & White Stripe
	21	Purgeable Organics	Encore Sampler	5g, 25g	Cool to 4° C	Label on Bag
	22	Purgeable Organics - PrePres.	40 mL Pre-Tared Clear Glass Vial + 10 mL MeOH ampule	40	MeOH in field; Cool to 4° C	Pre-Labeled (Light Yellow added at Lab)
MISC	23					
	24					
	25	Pesticide WWs by Method 608	1000 mL Amber Glass	1000	pH 5-9; Cool to 4° C	Yellow & White Stripe
	26	Drinking Water Volatiles	40 mL Clear Glass Vial	40	Ascorbic Acid at Lab; HCl in Field	Yellow

Notes:

DI Water for Equipment Blanks	Container Type and Size	Qty.
VOC Free		
Millipore		
ASTM Metals Free		

TriMatrix Laboratories, Inc.

Project Chemist Initials	Added to Calendar & Folders (initials/date)	Revision:	Revised By/Date:

Client: _____ Project Manager: _____

Project: _____ Contact: _____

TriMatrix Project No: _____ Date of Request: _____

Type of Order: ○ One-Time ⇒ Due to Client: _____ ○ AM ◉ PM

or

◉ Calendar ⇒ Frequency: ○ Weekly ○ Semi-Annually ○ Monthly ○ Annually ○ Quarterly ◉ Daily

Prepare Containers For:

Months	☐ Jan	☐ Feb	☐ Mar	☐ Apr	☐ May	☐ Jun
	☐ Jul	☐ Aug	☐ Sep	☐ Oct	☐ Nov	☐ Dec
Weeks	☐ 1	☐ 2	☐ 3	☐ 4	☐ 5	
Days	☐ M	☐ T	☐ W	☐ TH	☐ F	

Containers will be ◉ Picked Up or ○ Shipped via: ○ First Overnight ◉ Standard Overnight

Pick up/Ship Date: _____ ○ Priority Overnight ○ Express Saver

Ship Containers to: ○ 2-Day ○ Ground

_____ ○ Saturday Delivery ○ TriMatrix Courier

_____ ○ Other: _____

_____

_____ ☐ Shipment to be billed to FedEx Account No.:

Telephone No: _____

Shipment to include: ☐ COCs (Qty) _____ ☐ Custody Seals ☐ Temperature Blanks

☐ MSDS Sheets for all preservatives used ☐ WB TM#? ○ Y ◉ N

Comments: ☐ Cooler Banding Required

_____

_____

_____

Assembled by/Date:	Checked by/Date:	Shipped by/Date:

Cooler Number(s) Used:	Coolers Sealed With Tape	Coolers Sealed With Banding Strap	Tracking Number Label(s):
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	

Appendix O

Sample Receipt Record

TriMatrix Laboratories, Inc.

Date: _____

Delivery Method A: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method B: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method C: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Delivery Method D: _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

TriMatrix Courier (TC): _____ **No. of Sample Boxes:** _____ **Number of Coolers:** _____ **Signed for By:** _____ **Time:** _____

Page/ Line Number	Client	Quantity of Coolers OR TriMatrix Cooler Number	Arrived in Laboratory					Submittal Number (Project Chemist)	Folder Prepared (Log-In ✓)
			Time	AM	PM	Received By	Delivery Method Letter		
1-1									
1-2									
1-3									
1-4									
1-5									
1-6									
1-7									
1-8									
1-9									
1-10									
1-11									
1-12									
1-13									
1-14									
1-15									
1-16									
1-17									
1-18									
1-19									
1-20									
1-21									

Appendix P

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST

Client	Project-Submittal No. new / add to
Receipt Record Page/Line No.	Project Chemist / Sample Nos.

Coolers Received

Recorded by (initials/date)	☐ Cooler ☐ Box ☐ Other_____	Qty Received	Thermometer Used: ☐ IR Gun (#202) ☐ Digital Thermometer (#54) ☐ Other (#_____)	☐ See Additional Cooler Information Form

Cooler No. / Time	Cooler No. / Time	Cooler No. / Time	Cooler No. / Time
Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact
Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom
Coolant/Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ☐ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ☐ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ☐ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ☐ none / avg 2-3 containers
Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container
Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C
tb	tb	tb	tb
tb location: representative / in ice	tb location: representative / in ice	tb location: representative / in ice	tb location: representative / in ice
1	1	1	1
2	2	2	2
3	3	3	3
Average °C	Average °C	Average °C	Average °C
☐ Cooler ID on COC?	☐ Cooler ID on COC?	☐ Cooler ID on COC?	☐ Cooler ID on COC?
☐ VOC trip blank received?	☐ VOC trip blank received?	☐ VOC trip blank received?	☐ VOC trip blank received?

If any shaded areas checked, complete Sample Receiving Non-Conformance Form

Paperwork Received

☐ **No COC received**

N/A	Yes	No	
	☐	☐	**Chain of Custody Record(s)?**
	☐		If **No**, COC initiated by _____
	☐		Rec'd for Lab signed/date/time?
☐	☐		Shipping Document?
☐	☐		Other _____

COC ID Nos.

☐ TriMatrix

☐ Other (name or ID#)_____

Check COC for Accuracy

☐ **No analysis requested**

Yes	No	
☐	☐	Sample ID matches COC?
☐	☐	Sample date and time matches COC?
☐		Container type completed on COC?
☐	☐	All container types indicated are received?

Sample Condition Summary

☐ **Non-TriMatrix containers, see Notes**

N/A	Yes	No	
	☐	☐	Broken containers/lids?
	☐	☐	Missing or incomplete labels?
	☐	☐	Illegible information on labels?
	☐	☐	Low volume received?
	☐	☐	Inappropriate containers received?
☐	☐	☐	VOC vials / TOX containers have headspace?
☐	☐	☐	Extra sample locations / containers not listed on COC?

Check Sample Preservation

N/A	Yes	No	
	☐	☐	Average sample temperature ≤6° C?
☐	☐		Completed Sample Preservation Verification Form?
☐	☐	☐	Samples preserved correctly?
☐	☐		If "No", added orange tag?
☐	☐		Received pre-preserved VOC soils?

☐ MeOH ☐ Na_2SO_4

Check for Short Hold-Time Prep/Analyses

☐ Bacteriological
☐ Air Bags
☐ EnCores / Methanol Pre-Preserved
☐ Formaldehyde/Aldehyde
☐ Green-tagged Containers
☐ Yellow/White-tagged 1L Ambers (SV Prep-Lab)

AFTER HOURS ONLY:
COPIES OF COC TO LAB AREA(S)
☐ NONE RECEIVED
☐ RECEIVED, COCs TO LAB(S)

Notes

☐ Trip blank received ☐ Trip blank not listed on COC
☐ No COC received, Proj. Chemist reviewed (init./date) _____
☐ No analysis requested, Proj. Chemist completed (init./date) _____

Cooler Received (Date/Time)	Paperwork Delivered (Date/Time)	≤1 Hour Goal Met?
		Yes / No

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST - page 2

Project Chemist Use

Notify Laboratory Personnel of Short Hold-Times and/or Rush Work ☐ **NONE**

(Lab personnel notified/date)

- ☐ Inorganics _____
- ☐ Microbiology (bacteria) _____
- ☐ Metals Prep _____
- ☐ Metals _____
- ☐ GC-Volatiles _____
- ☐ MS-Volatiles _____
- ☐ Semi-Vol. Prep _____
- ☐ GC-Semi-Volatiles _____
- ☐ MS-Semi-Volatiles _____

Log-In Priority ☐ RUSH ☐ Standard

Project Chemist Notes to Log-In Personnel

Trip Blank: ☐ Log-in ☐ Do not log-in

☐ Prep Storage Blank for Client (VOCs)

☐ Sub-Contracting required ☐ Coolant required

☐ Non-TriMatrix or non-standard container type(s) received
Check pH of container type _____
Expected pH:_____ ☐ Adjust if needed

☐ Adjust pH of orange-tagged containers

☐ Lab-filter samples and document on Preservation Form

Sample Narratives to be added at Log-in

Log-In Use

Log Samples into LIMS Sample Nos.

N/A	Yes	
	☐	Receive samples in LIMS
	☐	Date/Time received entered in LIMS match COC
	☐	Read project and submittal narratives
☐	☐	Enter VOC rack/tray number into submittal narrative
	☐	Enter sample information into LIMS
☐	☐	Add any sample narratives
☐	☐	If non-conformance issues, add sample qualifiers
	☐	Print sample number labels

Log-in Analyst (initials/date/time)

Label Sample containers

N/A	Yes	No	
	☐	☐	LIMS label matches tag?
☐	☐		**DISCREPANCIES CORRECTED IN LIMS**
			Initials/Date: _____
☐	☐		Applicable stickers applied to labels?
			☐ MS/MSD sample
			☐ Composite before analysis
☐	☐		Applicable stickers applied to containers?
			☐ Waste sample
			☐ PT sample
			☐ USDA regulated
	☐	☐	Orange-tagged containers present?
☐	☐	☐	Adjust pH per Project Chemist
☐	☐		Initials and Date/Time Adjusted on orange tag?
☐	☐		Initials and Date/Time Adjusted on Preservation Form?

Verify Label Accuracy

	☐	Second analyst checked labels for accuracy?
☐	☐	Verify that Orange-tagged containers adjusted/initialed?

Labeled by (initials/date)	Verified by (initials/date)

Sample Storage Check all that apply

bacteria……	☐ bacteria refrigerator
non-volatiles……	☐ walk-in cooler
volatiles…….….	☐ volatile lab refrigerator
waste……...…	☐ waste cabinet
waste VOCs.....…	☐ log-in hood refrigerator
low-level Hg ….	☐ metals lab - DO NOT STORE IN WALK-IN

Paperwork

N/A	Yes	
	☐	original COC (white)
	☐	copy of COC (yellow)
	☐	receiving/log-in checklist
☐	☐	additional cooler information form
☐	☐	sample preservation verification
☐	☐	sample receiving non-conformance form
☐	☐	shipping documents
☐	☐	custody seals
	☐	arrival log
☐	☐	other (note)

Appendix Q

TriMatrix
Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST
ADDITIONAL COOLER INFORMATION

Recorded by (initials/date)	Client		Project-Submittal No.
	Receipt Log No.	Sample Nos.	Project Chemist

Cooler No. | Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant/Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No. | Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No. | Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No. | Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No. | Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant/Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No. | Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No. | Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Cooler No. | Time

Custody Seals
☐ none
☐ present / intact
☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
☐ loose ice / avg 2-3 containers
☐ bagged ice / avg 2-3 containers
☐ blue ice / avg 2-3 containers
☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
☐ temperature blank (tb)
☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

☐ Cooler ID on COC?
☐ VOC trip blank received?

Comments

Appendix R

TriMatrix Laboratories, Inc.

SAMPLE PRESERVATION VERIFICATION FORM

page ___ of ____

Client		Project-Submittal No.
Receipt Log No.	Completed By (initials/date)	Project Chemist

COC ID No.			Adjusted by:_____ Date:_____		DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
Container Type	5	4	13		3	6	15	
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe	
Preservative	NaOH	H_2SO_4	H_2SO_4		None	HNO_3	HNO_3	
Expected pH	**>12**	**<2**	**<2**		**~7**	**<2**	**<2**	
COC Line No. 1								
COC Line No. 2								
COC Line No. 3								
COC Line No. 4								
COC Line No. 5								
COC Line No. 6								
COC Line No. 7								
COC Line No. 8								
COC Line No. 9								
COC Line No. 10								

Comments

COC ID No.			Adjusted by:_____ Date:_____		DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
Container Type	5	4	13		3	6	15	
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe	
Preservative	NaOH	H_2SO_4	H_2SO_4		None	HNO_3	HNO_3	
Expected pH	**>12**	**<2**	**<2**		**~7**	**<2**	**<2**	
COC Line No. 1								
COC Line No. 2								
COC Line No. 3								
COC Line No. 4								
COC Line No. 5								
COC Line No. 6								
COC Line No. 7								
COC Line No. 8								
COC Line No. 9								
COC Line No. 10								

Comments

pH strip lot No.

☐ **HC896537**

☐ _____

Aqueous Samples: For each sample and container type, check the box if pH is acceptable. **If pH is not acceptable for any sample container, record pH in box, and note on Sample Receiving Checklist and on Sample Receiving Non-Conformance Form.** If approved by Project Chemist, add acid or base to the sample to achieve the correct pH. Add up to, but do not exceed 2x the volume initially added at container prep (see table below for initial volumes used). **Add orange pH tag to sample container and record information requested. Record adjusted pH on this form. Do not adjust pH for container types 3, 6, and 15.**

Container Size (mL)	Original Vol. of Preservative (mL)
Container Type 5:	NaOH
500	2.5
1000	5.0
Container Type 4:	H_2SO_4
125	0.5
250	1.0
500	2.0
1000	4.0
Container Type 13	H_2SO_4
500	2.5

Appendix S

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING NON-CONFORMANCE REPORT

Client		Project-Submittal No.
Receipt Log No.	Completed By (initials/date)	Project Chemist

List non-conformance issues associated with this submittal in the chart below/left. Identify discrepancies between the COC and sample tags in the chart below/right. Add comments as needed. Give to Project Chemist for immediate action.

		Type of Problem									
COC ID No.	Line No.	Discrepancy	Missing Container	Broken Container	Label Missing / Incomplete	Label Illegible	Low Volume	Inappropriate Container	Headspace	Not Listed on COC	Preservation

COC					Sample Tag					Line Item Comments
Sample Field ID	Date Sampled	Time Sampled	Container Type	Qty	Sample Field ID	Date Sampled	Time Sampled	Container Type	Qty	

General Comments:

Project Chemist (initials/date)

Appendix T

Client:	Project Manager: **Rick D. Wilburn**
Project: **TCLP Semi-Volatiles**	Project Number: **[none]**
Work Order: **TCLP October 2008**	SDG:

Report To:

R
T
2
L
Phone:
Fax:

Invoice To:

TriMatrix Laboratories
Mr. Rick D. Wilburn
5560 Corporate Exchange Court SE
Grand Rapids, MI 49512-5503
Phone: 616-975-4500 x4
Fax: 616-942-7463

Client Due Date:	**Nov-24-08 23:00 (21 day TAT)**	Report Level:	**3MD**
Date Received:	Oct-24-08 12:00	Received By:	Rick D. Wilburn
Date Logged In:	Oct-27-08 08:15	Logged In By:	William D. Cole
W.O. Comments:	*QC is 3MD.*		

Analysis	Lab Due Date	TAT	Expires	Analysis Comments
0810557-01 TCLP Semi-Volatiles [Soil] ***Sampled Oct-23-08 08:00 Eastern by***				
8270C TCLP Herbs	Nov-24-08 17:00	10	Oct-30-08 08:00	
8270C TCLP SVOC/Pest	Nov-24-08 17:00	10	Oct-30-08 08:00	
TCLP Organics Extraction	Nov-24-08 17:00	10	Nov-06-08 08:00	
0810557-02 TCLP Analytes in Soil [Soil] ***Sampled Oct-23-08 08:00 Eastern by***				
8151A Herbicides CLP [dual-col]	Nov-24-08 17:00	10	Nov-06-08 08:00	
8270C Standard SVOCs	Nov-24-08 17:00	10	Nov-06-08 08:00	
Solids, Total 3550B (%)	Nov-24-08 17:00	10	Nov-06-08 08:00	

Client:		Project Manager:	**Rick D. Wilburn**
Project:	**TCLP Semi-Volatiles**	Project Number:	**[none]**
Work Order:	**TCLP October 2008**	SDG:	

Inorganic - Wet Chemistry Analysis Detail

* indicates custom

Matrix	Analysis	Unit	MDL	RL
Soil	Solids, Total 3550B (%)	%	0.1	0.1

Semivolatiles GC Analysis Detail

* indicates custom

	Analyte	CLrept?	QCrept?	MDL	RL
Soil	**8151A Herbicides CLP [dual-col]**	**mg/kg**			
	2,4-D	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex)	Y	Y	0.0051	0.05
	2,4-D [2C]	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex) [2C]	Y	Y	0.0051	0.05

Semivolatiles MS Analysis Detail

* indicates custom

	Analyte	CLrept?	QCrept?	MDL	RL
Soil	**TCLP Organics Extraction**				
Soil	**8270C Standard SVOCs**	**mg/kg**			
	1,4-Dichlorobenzene	Y	Y	0.000906	0.0167
	2,4-Dinitrotoluene	Y	Y	0.00406	0.0167
	Hexachlorobenzene	Y	Y	0.00337	0.0167
	Hexachlorobutadiene	Y	Y	0.00107	0.0167
	Hexachloroethane	Y	Y	0.000778	0.0167
	3+4-Methylphenol	Y	Y	0.00129	0.0167
	2-Methylphenol	Y	Y	0.00223	0.0167
	Nitrobenzene	Y	Y	0.00199	0.0167
	Pentachlorophenol	Y	Y	0.00368	0.0167
	Pyridine	Y	Y	0.00566	0.0167
	2,4,5-Trichlorophenol	Y	Y	0.00329	0.0167
	2,4,6-Trichlorophenol	Y	Y	0.00132	0.0167
Soil	**8270C TCLP Herbs**	**mg/L**			
	2,4-D	Y	Y	0.00409	0.1
	2,4,5-TP (Silvex)	Y	Y	0.00381	0.1
Soil	**8270C TCLP SVOC/Pest**	**mg/L**			
	1,4-Dichlorobenzene	Y	Y	0.0000148	0.005
	2,4-Dinitrotoluene	Y	Y	0.000214	0.005
	Hexachlorobenzene	Y	Y	0.0000117	0.005
	Hexachlorobutadiene	Y	Y	0.000125	0.005
	Hexachloroethane	Y	Y	0.0000378	0.005
	Nitrobenzene	Y	Y	0.0000257	0.005
	Pyridine	Y	Y	0.000385	0.05
	Pentachlorophenol	Y	Y	0.000187	0.005
	2,4,6-Trichlorophenol	Y	Y	0.0000267	0.005
	2,4,5-Trichlorophenol	Y	Y	0.000109	0.005

Client:	Project Manager: **Rick D. Wilburn**
Project: **TCLP Semi-Volatiles**	Project Number: **[none]**
Work Order: **TCLP October 2008**	SDG:

Semivolatiles MS Analysis Detail

Analyte	CLrept?	QCrept?	* indicates custom MDL	RL
2-Methylphenol	Y	Y	0.000144	0.005
3-Methylphenol	Y	Y	0.0000157	0.005
4-Methylphenol	Y	Y	0.0000157	0.005
gamma-BHC (Lindane)	Y	Y	0.0000566	0.005
Endrin	Y	Y	0.000284	0.005
Methoxychlor	Y	Y	0.0000723	0.005
Technical Chlordane	Y	Y	0.000124	0.005
Heptachlor	Y	Y	0.0000907	0.005
Heptachlor Epoxide	Y	Y	0.0000758	0.005
Toxaphene	Y	Y	0.000293	0.5

Appendix U

Semivolatiles GC Sample Receipt Notice

Client: **C**
Project: **TCLP Semi-Volatiles**
Client Due Date: **Nov-24-08 23:00 (21 day TAT)**
W.O. Comments: *QC is 3MD.*

Project Manager: **Rick D. Wilburn**
Project Number: **[none]**
Report Level: **3MD**

Lab Number	Sample Name / Analysis	Matrix	Sampled Date / TAT	Expire Date	Sample Comments / Lab Due Date	Comments
0810557-02	**TCLP Analytes in Soil**	**Soil**	**Oct-23-08 08:00 Eastern**			
	8151A Herbicides CLP [dual-col]		10	Nov-06-08 08:00	Nov-24-08 17:00	

Semivolatiles GC Analysis Detail

* indicates custom

	Analyte	CLrept?	QCrept?	MDL	RL
Soil	**8151A Herbicides CLP [dual-col]**	**mg/kg**			
	2,4-D	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex)	Y	Y	0.0051	0.05
	2,4-D [2C]	Y	Y	0.0602	0.2
	2,4,5-TP (Silvex) [2C]	Y	Y	0.0051	0.05

Semivolatiles MS Sample Receipt Notice

Client: **T**
Project: **TCLP Semi-Volatiles**
Client Due Date: **Nov-24-08 23:00 (21 day TAT)**
W.O. Comments: *QC is 3MD.*

Project Manager: **Rick D. Wilburn**
Project Number: **[none]**
Report Level: **3MD**

Lab Number	Sample Name / Analysis	Matrix	Sampled Date / TAT	Expire Date	Sample Comments / Lab Due Date	Comments
0810557-01	**TCLP Semi-Volatiles**	**Soil**	**Oct-23-08 08:00 Eastern**			
	8270C TCLP Herbs		10	Oct-30-08 08:00	Nov-24-08 17:00	
	8270C TCLP SVOC/Pest		10	Oct-30-08 08:00	Nov-24-08 17:00	
	TCLP Organics Extraction		10	Nov-06-08 08:00	Nov-24-08 17:00	
0810557-02	**TCLP Analytes in Soil**	**Soil**	**Oct-23-08 08:00 Eastern**			
	8270C Standard SVOCs		10	Nov-06-08 08:00	Nov-24-08 17:00	

Semivolatiles MS Analysis Detail

	Analyte	CLrept?	QCrept?	* indicates custom MDL	RL
Soil	**TCLP Organics Extraction**				
Soil	**8270C Standard SVOCs**	**mg/kg**			
	1,4-Dichlorobenzene	Y	Y	0.000906	0.0167
	2,4-Dinitrotoluene	Y	Y	0.00406	0.0167
	Hexachlorobenzene	Y	Y	0.00337	0.0167
	Hexachlorobutadiene	Y	Y	0.00107	0.0167
	Hexachloroethane	Y	Y	0.000778	0.0167
	3+4-Methylphenol	Y	Y	0.00129	0.0167
	2-Methylphenol	Y	Y	0.00223	0.0167
	Nitrobenzene	Y	Y	0.00199	0.0167
	Pentachlorophenol	Y	Y	0.00368	0.0167
	Pyridine	Y	Y	0.00566	0.0167
	2,4,5-Trichlorophenol	Y	Y	0.00329	0.0167
	2,4,6-Trichlorophenol	Y	Y	0.00132	0.0167
Soil	**8270C TCLP Herbs**	**mg/L**			
	2,4-D	Y	Y	0.00409	0.1
	2,4,5-TP (Silvex)	Y	Y	0.00381	0.1
Soil	**8270C TCLP SVOC/Pest**	**mg/L**			
	1,4-Dichlorobenzene	Y	Y	0.0000148	0.005
	2,4-Dinitrotoluene	Y	Y	0.000214	0.005
	Hexachlorobenzene	Y	Y	0.0000117	0.005
	Hexachlorobutadiene	Y	Y	0.000125	0.005
	Hexachloroethane	Y	Y	0.0000378	0.005
	Nitrobenzene	Y	Y	0.0000257	0.005
	Pyridine	Y	Y	0.000385	0.05
	Pentachlorophenol	Y	Y	0.000187	0.005
	2,4,6-Trichlorophenol	Y	Y	0.0000267	0.005
	2,4,5-Trichlorophenol	Y	Y	0.000109	0.005
	2-Methylphenol	Y	Y	0.000144	0.005
	3-Methylphenol	Y	Y	0.0000157	0.005
	4-Methylphenol	Y	Y	0.0000157	0.005
	gamma-BHC (Lindane)	Y	Y	0.0000566	0.005
	Endrin	Y	Y	0.000284	0.005
	Methoxychlor	Y	Y	0.0000723	0.005
	Technical Chlordane	Y	Y	0.000124	0.005
	Heptachlor	Y	Y	0.0000907	0.005
	Heptachlor Epoxide	Y	Y	0.0000758	0.005
	Toxaphene	Y	Y	0.000293	0.5

Semivolatiles MS, Soil, 3550B Sonication Extraction

Surrogate #1 = 8110251 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0810557	8270C Standard SVOCs	0810557	8270C MDEQ BNA	0810557	8270C MDEQ Base/Neutrals
0810648	8270C MDEQ BNA	0810665	8270C MDEQ Base/Neutrals	0811070	8270C Standard SVOCs
0811070	8270C MDEQ BNA	0811070	8270C MDEQ Base/Neutrals	0811154	8270C MDEQ BNA

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0813151-BLK1		Nov-10-08 07:09	BJH	30	1	100				BLANK	
0813151-BLK2		Nov-10-08 07:09	JLB	30	1	100				BLANK	mdeq base/neutrals
0813151-BLK3		Nov-10-08 07:09		30	1	100				BLANK	
0813151-BLK4		Nov-10-08 07:09		30	1	100				BLANK	
0813151-DUP1		Nov-10-08 07:09	BJH	30	1	100	0810557-02			DUPLICATE	
0813151-BS1		Nov-10-08 07:09	BJH	30	1	100		8110206	100	LCS	
0813151-BS2		Nov-10-08 07:09	BJH	30	1	100		8100124	100	LCS	
0813151-BS3		Nov-10-08 07:09	JLB	30	1	100		8110206	100	LCS	mdeq base/neutrals
0813151-BS4		Nov-10-08 07:09		30	1	100		8110206	100	LCS	
0813151-BS5		Nov-10-08 07:09		30	1	100		8110206	100	LCS	
0813151-MS1		Nov-10-08 07:09	BJH	30	1	100	0811070-06	8110206	100	MATRIX SPIKE	
0813151-MSD1		Nov-10-08 07:09	BJH	30	1	100	0811070-06	8110206	100	MATRIX SPIKE DUP	
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					Added for BatchQC in: 0813151
0810557-02	A	Nov-10-08 07:09	BJH	30	1	100					Added for BatchQC in: 0813151
0810648-19	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-20	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-21	A	Nov-10-08 07:09	BJH	30	1	100					
0810648-24	A	Nov-10-08 07:09	BJH	30	5	100					stopped at 5 mL
0810648-25	A	Nov-10-08 07:09	BJH	30	5	100					stopped at 5 mL
0810665-01	A	Nov-10-08 07:09	BJH	30	1	100					2,4-Dinitrotoluene only
0810665-02	A	Nov-10-08 07:09	BJH	30	1	100					2,4-Dinitrotoluene only

Comments:

Analyst Initials:

Semivolatiles MS, Soil, Nov-12-08

Instrument = 308, Calibration = 8K05005

Sequence Analyses:
8270C MDEQ BNA

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client / QC Type*	*Extraction Comments*
8111315-TUN1	QC		8100195	8060452	MS TUNE	
8111315-CCV1	QC		8110293	8060452	CALIBRATION CHECK	
8111315-CCV2	QC		8110085	8060452	CALIBRATION CHECK	
0813151-BLK4	QC			8060452	BLANK	
0813151-BS5	QC			8060452	LCS	
0811154-04	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-05	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-06	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-07	8270C MDEQ BNA	A 02		8060452	Engineering	
0811154-09	8270C MDEQ BNA	A 02		8060452	Engineering	

Comments:

Analyst Initials:

Lab PM (Rick D. Wilburn) Nov-11-08 - Dec-09-08

yzed,Available,Batched,Cancelled,Entered,Hold,Invoiced,Leached,Prepared,Received,Reported,Reviewed,Subcontr

Lab Number	Analysis	Matrix	RptLev	RTAT	Due	Expires	Status	Client	Project	Sample [Analysis] Comments
0810557-01	8270C TCLP Herbs	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	8270C TCLP SVOC/P	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	TCLP Organics Extrac	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8151A Herbicides CLI	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8270C Standard SVOC	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810558-01	DRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-02-08	Reported	RTC	Minnesota DRO/GRO	
0810558-01	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	GRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	

Appendix V

TriMatrix Laboratories, Inc. - Department

Work Orders Received Sep-01-08 to Sep-30-08 - Printed Dec-08-08 10:26 by TCB

Department	Samples	Analyses	Price	Surcharge	Total
Inorganic - Wet Chemistry	1622	5878	$120,471.90	$333.75	$120,805.60
Metals	997	10085	$83,255.61	$829.00	$84,084.61
Semivolatiles GC	495	638	$60,893.00	$110.00	$61,003.00
Semivolatiles MS	468	548	$78,247.00	$58.15	$78,305.15
Volatiles GC	116	117	$5,438.50	$14.40	$5,452.90
Volatiles MS	1382	1400	$135,669.00	$297.75	$135,966.80
****TOTALS****	**5080**	**18666**	**$483,975.01**	**$1,643.05**	**$485,618.06**

TriMatrix Laboratories, Inc. - % On-Time by Department [Sep-01-08 to Sep-30-08]

Printed Dec-08-08 10:36 by TCB

Department: [All]

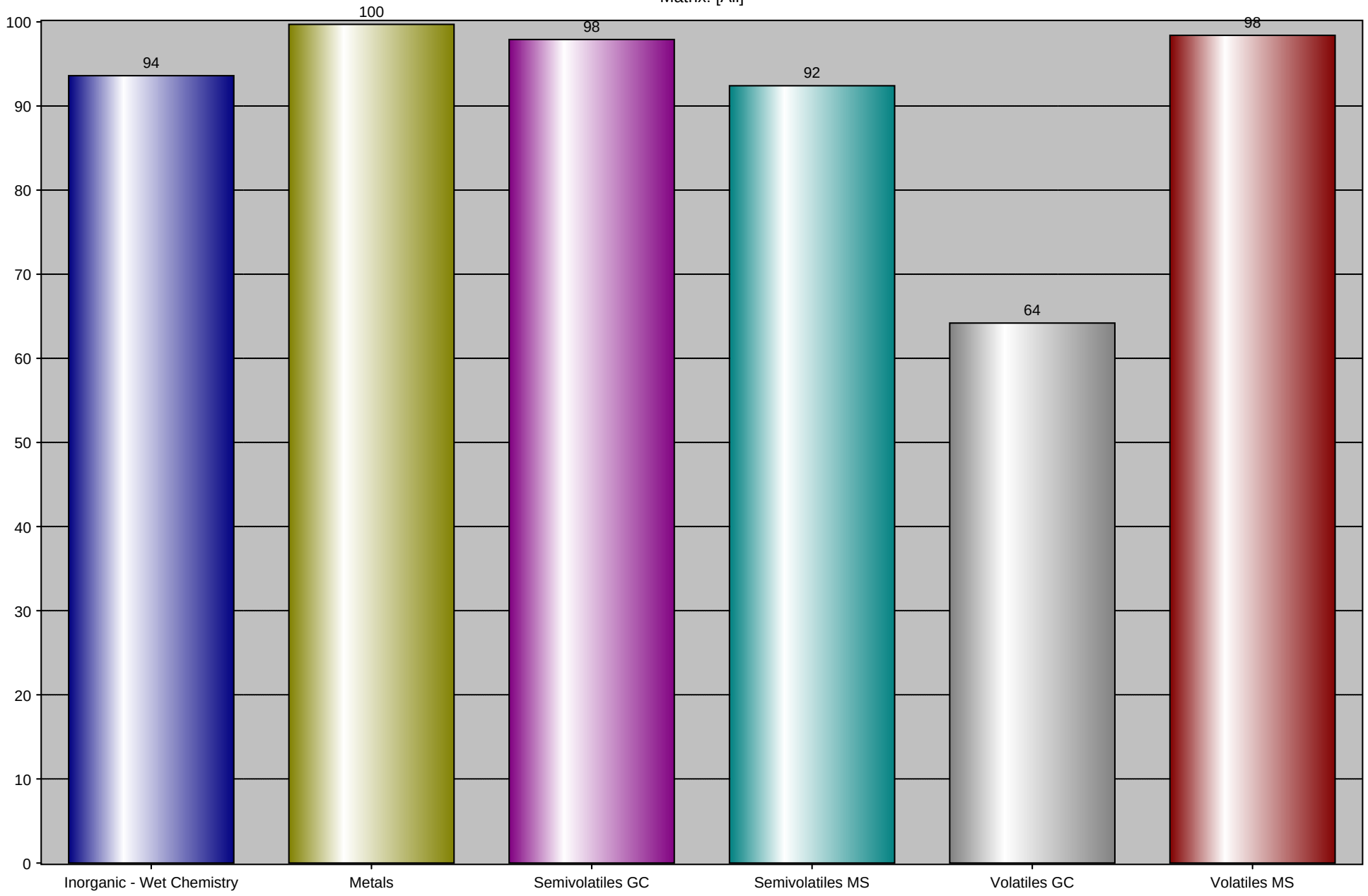
Analysis: [All]

Matrix: [All]

Department	On-Time	Total	%
Inorganic - Wet Chemistry	4117	4399	93.6
Metals	6889	6907	99.7
Semivolatiles GC	644	658	97.9
Semivolatiles MS	390	422	92.4
Volatiles GC	147	229	64.2
Volatiles MS	1415	1438	98.4

TriMatrix Laboratories, Inc. - % On-Time by Department [Sep-01-08 to Sep-30-08]

Printed Dec-08-08 10:34 by TCB
Department: [All]
Analysis: [All]
Matrix: [All]



Lab PM (Rick D. Wilburn) Jan-01-08 - Dec-09-08

Available,Cancelled,Completed,Invoiced,Preliminary,Received,Reported

Work Order	Done	RptLvl	Pending	Status	Client	Project Name (Number)	PMgr	TAT	Received	Due
0707274	64/64	3MD		Completed	Environmental Resource Associates	ERA WS PT Samples Summer (35005)	RDW	22	Jul-17-07	Aug-16-07
0801455	2/2	3MD		Completed	Environmental Resource Associates	ERA WP PT Samples (35005)	RDW	19	Jan-25-08	Feb-21-08
0801456	120/120	3FL		Completed	Environmental Resource Associates	Semi-Annual Solid PE Study (35338)	RDW	21	Jan-25-08	Feb-25-08
0801501	136/136	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	18	Jan-30-08	Feb-25-08
0802130	60/60	3MD		Completed	Environmental Resource Associates	ERA WS PT Samples Winter (35005)	RDW	16	Feb-08-08	Mar-03-08
0802188	4/4	3MD		Completed	Environmental Resource Associates	Micro Analyst Cert (34110)	RDW	10	Feb-12-08	Feb-26-08
0803447	4/4	3MD		Completed	Environmental Resource Associates	Micro Analyst Cert (34110)	RDW	10	Mar-27-08	Apr-10-08
0804113	116/116	3MD		Completed	Analytical Products Group	WP Performance Testing Program Spring (35508)	RDW	10	Apr-07-08	Apr-21-08
0805464	1/1	3MD		Completed	Analytical Products Group	WP Performance Testing Program Spring Re-Do (35508)	RDW	5	May-21-08	May-29-08
0805489	70/70	3MD		Completed	Analytical Products Group	DMRQA Testing (36330)	RDW	26	May-21-08	Jun-27-08
0806220	4/4	3MD		Completed	Analytical Products Group	WP Performance Testing Program Quick Turn (35508)	RDW	10	Jun-11-08	Jun-25-08
0806250	75/75	3MD		Completed	TriMatrix Laboratories	pH Strip Testing (36236)	RDW	10	Jun-12-08	Jun-26-08
0807484	126/126	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	18	Jul-23-08	Aug-18-08
0807485	120/120	3MD		Completed	Environmental Resource Associates	Semi-Annual Solid PE Study (35338)	RDW	22	Jul-24-08	Aug-25-08
0807486	2/2	3MD		Completed	Environmental Resource Associates	ERA WP PT Samples (35005)	RDW	17	Jul-24-08	Aug-18-08
0808052	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-04-08	Aug-18-08
0808059	2/2	3MD		Completed	Analytical Products Group	DMRQA Testing Micro (36330)	RDW	10	Aug-05-08	Aug-19-08
0808244	48/48	2RLM		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-13-08	Aug-27-08
0808571	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Aug-28-08	Sep-12-08
0809070	10/10	3MD		Completed	State of New York	Department of Health PT Samples (36229)	RDW	17	Sep-04-08	Sep-29-08
0809419	48/48	2RL		Completed	TriMatrix Laboratories	Stericup Filter Certification ([none])	RDW	10	Sep-22-08	Oct-06-08
0810124	110/110	3MD		Completed	Analytical Products Group	WP Performance Testing Program Fall (35508)	RDW	18	Oct-08-08	Nov-03-08
0810557	6/6	3MD		Completed	RTC	TCLP Semi-Volatiles ([none])	RDW	21	Oct-24-08	Nov-24-08

Lab PM (Rick D. Wilburn) Jan-01-08 - Dec-09-08

Available,Cancelled,Completed,Invoiced,Preliminary,Received,Reported

Work Order	Done	RptLvl	Pending	Status	Client	Project Name (Number)	PMgr	TAT	Received	Due
0810558	4/4	3MD		Completed	RTC	Minnesota DRO/GRO ([none])	RDW	21	Oct-24-08	Nov-24-08

Lab PM (Rick D. Wilburn) Nov-01-08 - Nov-30-08

yzed,Available,Batched,Cancelled,Entered,Hold,Invoiced,Leached,Prepared,Received,Reported,Reviewed,Subcontr

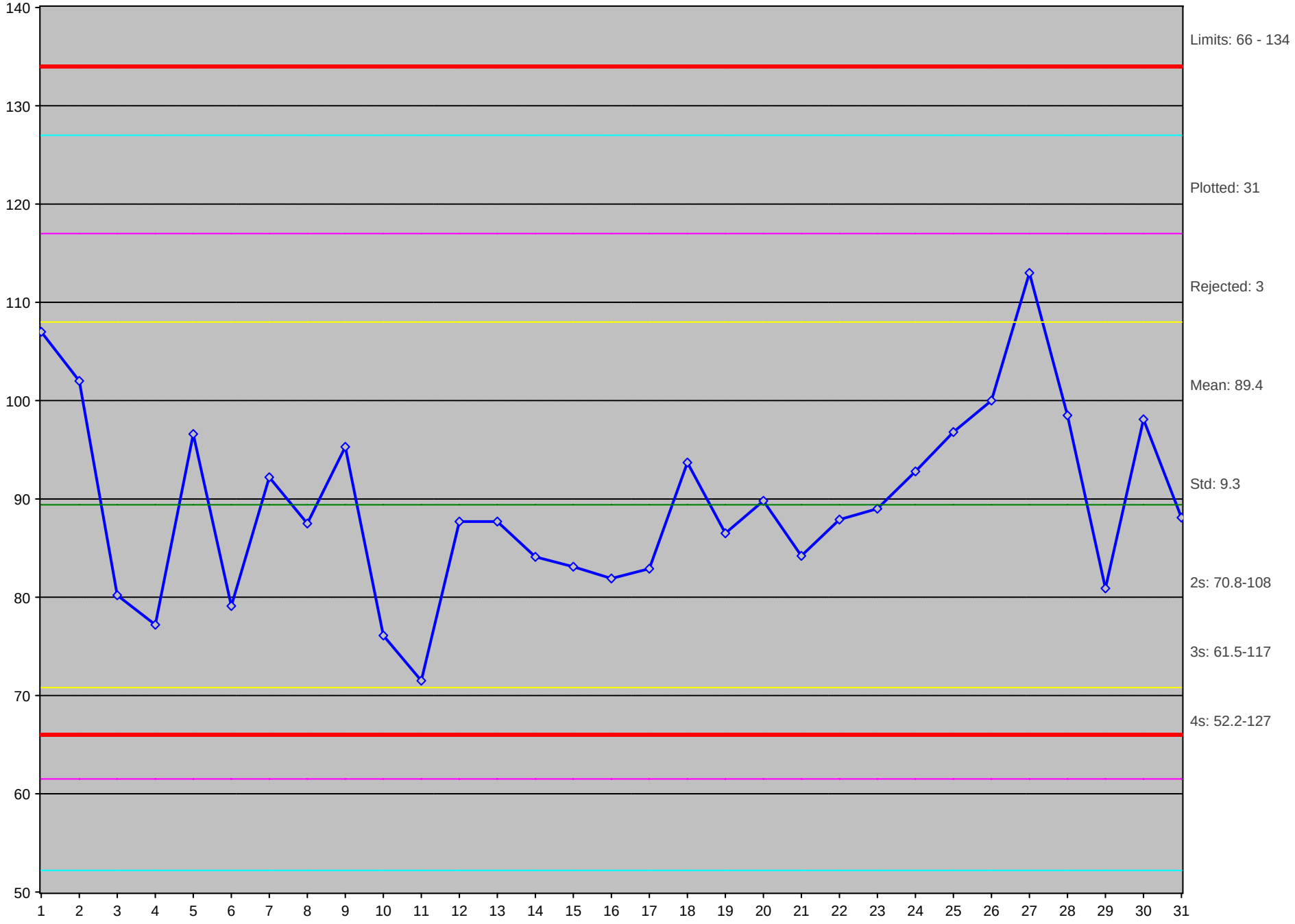
Lab Number	Analysis	Matrix	RptLev	RTAT	Due	Expires	Status	Client	Project	Sample [Analysis] Comments
0810557-01	8270C TCLP Herbs	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	8270C TCLP SVOC/P	Soil	3MD	10	Nov-24-08	Oct-30-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-01	TCLP Organics Extrac	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8151A Herbicides CLI	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	8270C Standard SVOC	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810557-02	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	TCLP Semi-Volatiles	
0810558-01	DRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-02-08	Reported	RTC	Minnesota DRO/GRO	
0810558-01	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	GRO - Wisconsin Metl	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	
0810558-02	Solids, Total 3550B (%	Soil	3MD	10	Nov-24-08	Nov-06-08	Reported	RTC	Minnesota DRO/GRO	

Appendix W

TriMatrix Laboratories, Inc. - LCS %R for CHRYSENE

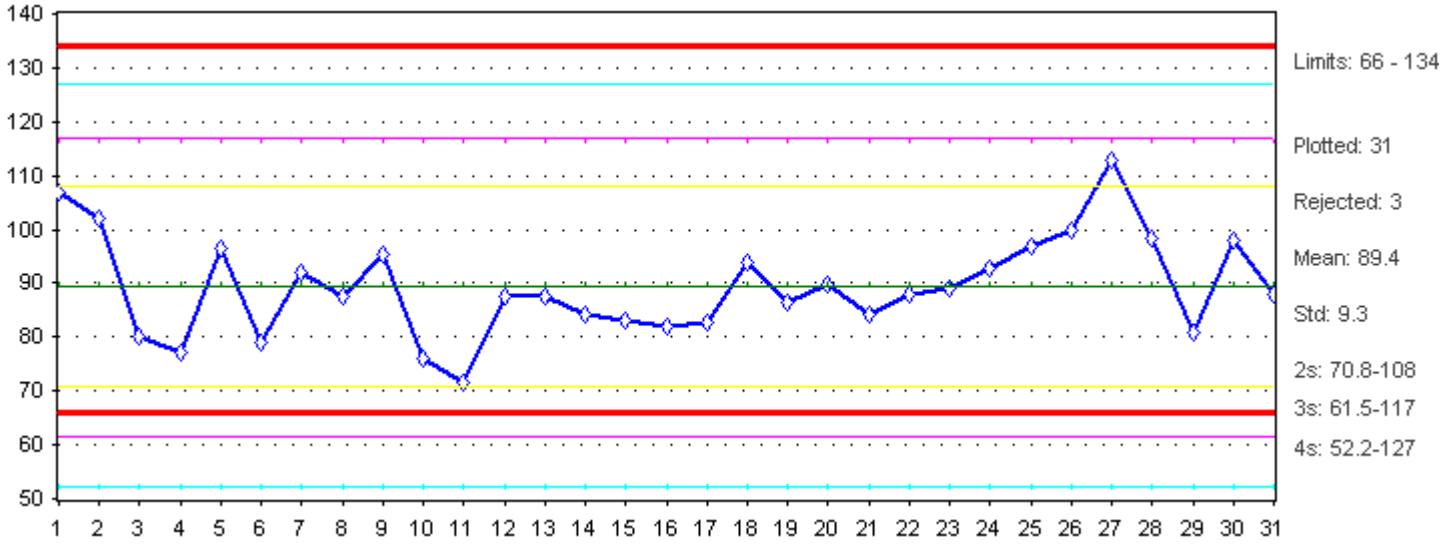
8270C Standard SVOCs IN Water Printed: Dec-08-08 11:04 by TCB

All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]



Printed:	Dec-08-08 11:09	Matrices:	Water
Client:	All Clients	Instruments:	All Instruments
Project:	All Projects	Prepared By:	All Extractionists
Analyses:	8270C Standard SVOCs	Analyzed By:	All Analysts
		Extractions:	All Extractions

Chrysene ***LCS %R***

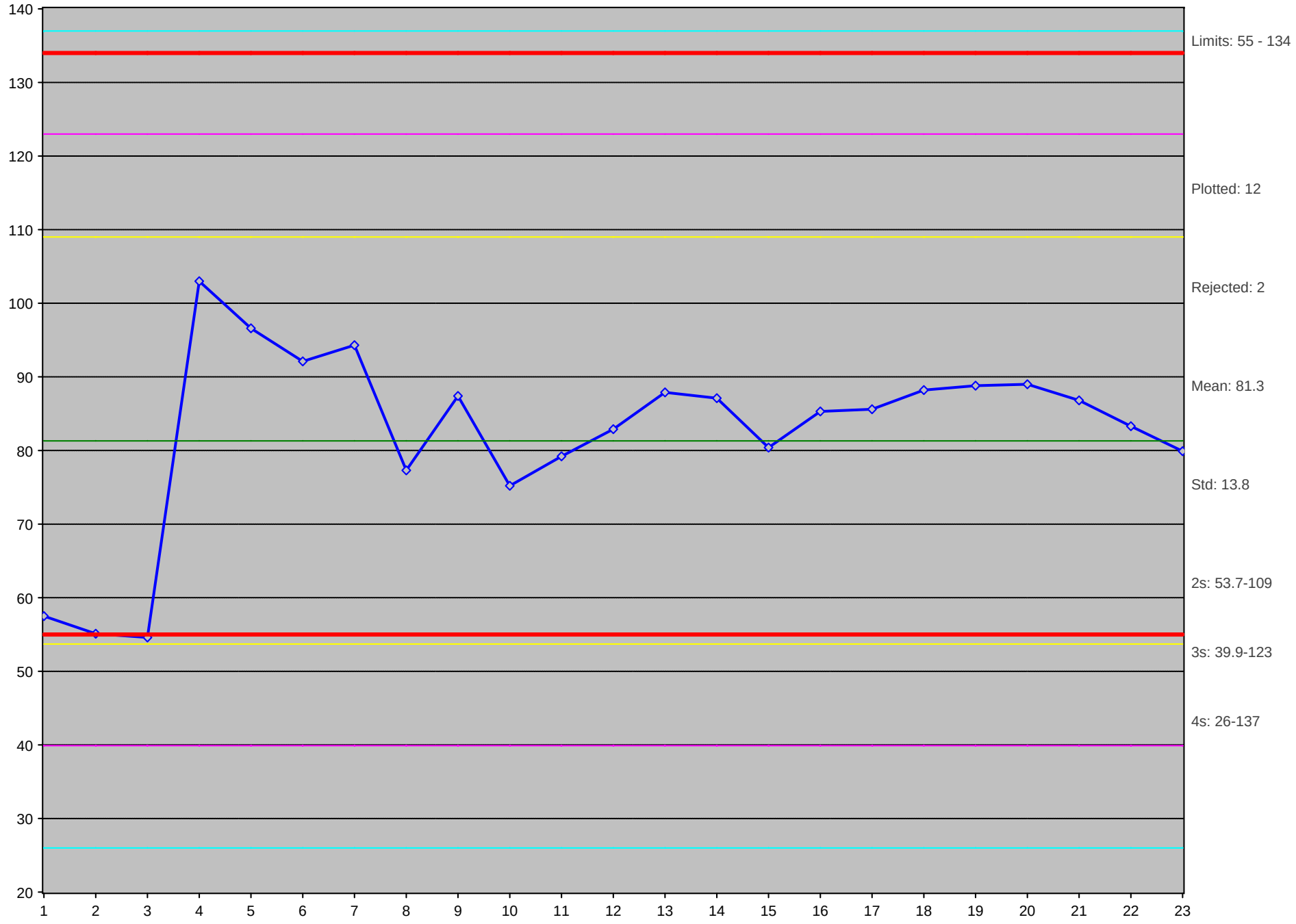


Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	0809978-BS1	8/29/08	9/2/08	9.6 ug/L	10.25	106.7708	66-134	
	0810083-BS2	9/2/08	9/3/08	9.6 ug/L	9.832	102.4167	66-134	
X	0810084-BS1	9/2/08	9/4/08	9.6 ug/L	0		66-134	
	0810561-BS1	9/12/08	9/17/08	96 ug/L	76.98	80.1875	66-134	
	0810624-BS1	9/15/08	9/16/08	9.6 ug/L	7.41	77.1875	66-134	
	0810739-BS1	9/17/08	9/18/08	96 ug/L	92.76	96.625	66-134	
X	0810740-BS1	9/17/08	9/22/08	9.6 ug/L	0		66-134	
	0810938-BS1	9/22/08	9/26/08	9.6 ug/L	7.59	79.0625	66-134	
	0810561-BS2	9/22/08	9/23/08	9.6 ug/L	8.852	92.20833	66-134	GN020
	0810967-BS1	9/23/08	9/26/08	96 ug/L	83.96	87.45833	66-134	
	0811030-BS1	9/24/08	9/27/08	96 ug/L	91.5	95.3125	66-134	
	0811107-BS1	9/25/08	9/27/08	9.6 ug/L	7.31	76.14583	66-134	
X	0811107-BS2	9/25/08	10/3/08	9.6 ug/L	0		66-134	
	0811170-BS1	9/26/08	9/27/08	9.6 ug/L	6.86	71.45833	66-134	
	0811170-BS2	9/29/08	10/2/08	9.6 ug/L	8.419	87.69791	66-134	
	0811170-BS3	9/29/08	10/2/08	9.6 ug/L	8.419	87.69791	66-134	
	0811030-BS2	10/1/08	10/1/08	9.6 ug/L	8.07	84.06249	66-134	
	0811661-BS1	10/9/08	10/10/08	9.6 ug/L	7.98	83.125	66-134	
	0811711-BS1	10/13/08	10/13/08	9.6 ug/L	7.866	81.9375	66-134	
	0811661-BS2	10/14/08	10/15/08	9.6 ug/L	7.96	82.91666	66-134	
	0811858-BS2	10/15/08	10/16/08	96 ug/L	89.94	93.6875	66-134	
	0811858-BS1	10/15/08	10/20/08	9.6 ug/L	8.3	86.45833	66-134	
	0812029-BS1	10/20/08	10/25/08	9.6 ug/L	8.62	89.79166	66-134	
	0812131-BS1	10/21/08	10/21/08	9.6 ug/L	8.08	84.16666	66-134	
	0812166-BS1	10/21/08	10/22/08	9.6 ug/L	8.44	87.91666	66-134	
	0812461-BS1	10/27/08	10/30/08	9.6 ug/L	8.54	88.95833	66-134	
	0812462-BS1	10/27/08	10/29/08	9.6 ug/L	8.91	92.81249	66-134	

TriMatrix Laboratories, Inc. - MS %R for CHRYSENE

8270C Standard SVOCs IN Water Printed: Dec-08-08 11:12 by TCB

All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]



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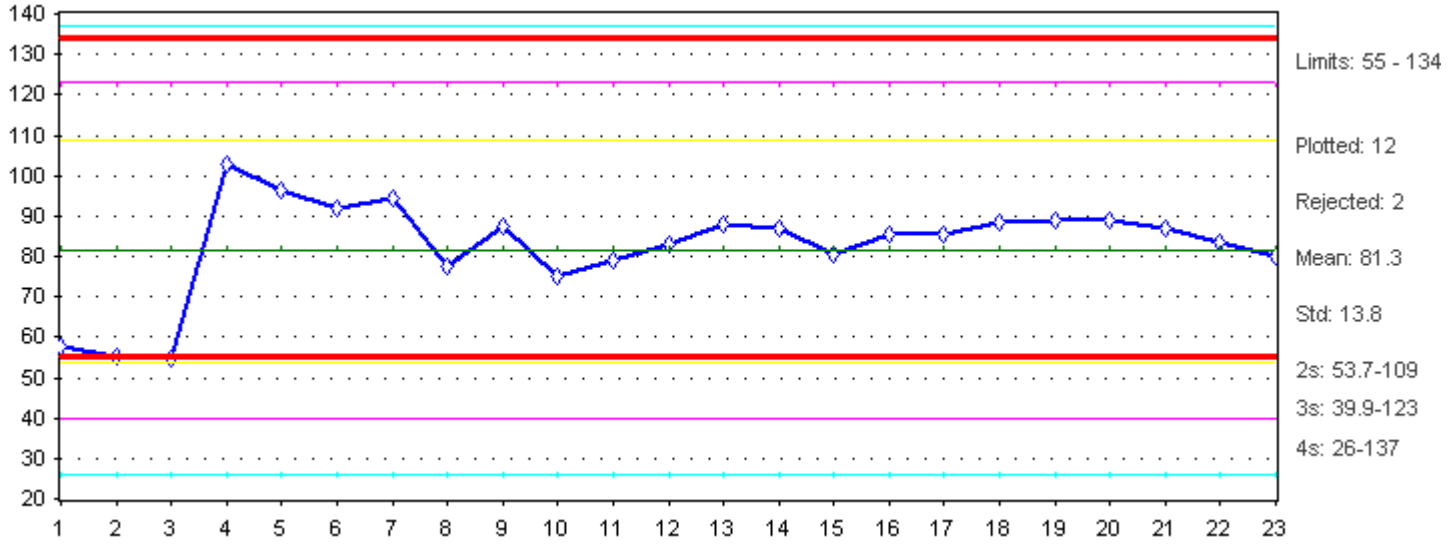
Project:	All Projects	Prepared By:	All Extractionists
Analyses:	8270C Standard SVOCs	Analyzed By:	All Analysts
		Extractions:	All Extractions

Chrysene ***MS %R***

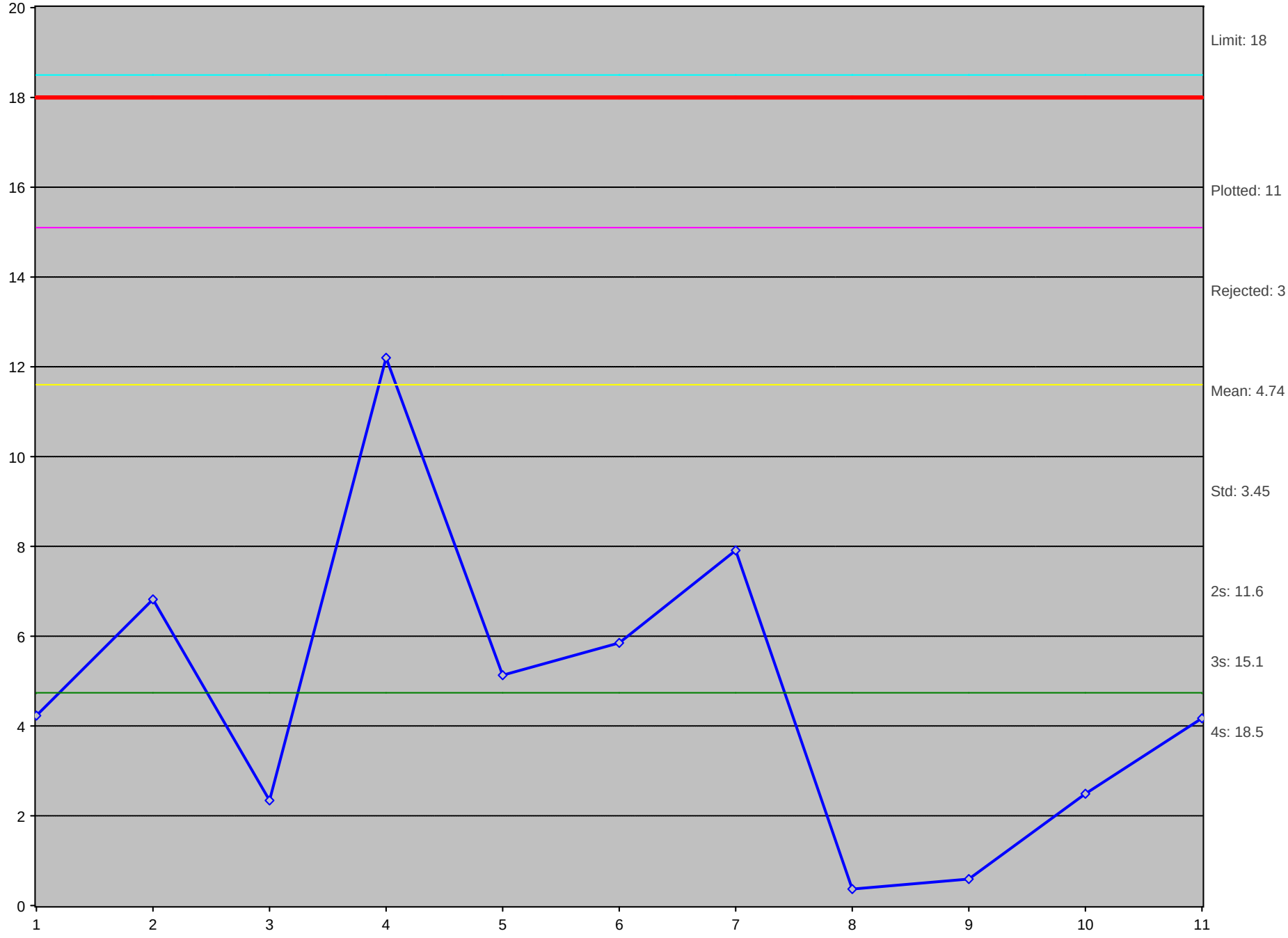


Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Source	Limits	Qualifiers
	0809978-MS1	8/29/08	9/2/08	9.69697 ug/L	5.606061	57.5	808570-08 (3.030303E-0	55-134	
	0809978-MSD1	8/29/08	9/2/08	9.69697 ug/L	5.373738	55.10417	808570-08 (3.030303E-0	55-134	
X	0810084-MSD1	9/2/08	9/4/08	9.6 ug/L	0.01	0.520833	0808570-29 (0.05)	55-134	
	0810084-MS1	9/2/08	9/5/08	9.6 ug/L	5.29	54.58333	0808570-29 (0.05)	55-134	
	0810561-MS1	9/12/08	9/18/08	96.9697 ug/L	100.2828	103.4166	0809136-03 (ND)	55-134	
	0810561-MSD1	9/12/08	9/18/08	96.9697 ug/L	93.66666	96.59373	0809136-03 (ND)	55-134	
	0810561-MS2	9/12/08	9/18/08	97.95918 ug/L	90.2347	92.11459	0809136-05 (ND)	55-134	
	0810561-MSD2	9/12/08	9/18/08	97.95918 ug/L	92.36735	94.29167	0809136-05 (ND)	55-134	
	0810967-MS1	9/23/08	9/27/08	97.95918 ug/L	75.7551	77.33333	0809311-06 (ND)	55-134	
	0810967-MSD1	9/23/08	9/27/08	97.95918 ug/L	85.59183	87.37499	0809311-06 (ND)	55-134	
	0811107-MS1	9/25/08	9/27/08	9.795918 ug/L	7.367347	75.20833	0809435-05 (ND)	55-134	
	0811107-MSD1	9/25/08	9/27/08	9.795918 ug/L	7.755102	79.16667	0809435-05 (ND)	55-134	
	0811661-MS1	10/9/08	10/10/08	10.90909 ug/L	9.045455	82.91668	0810133-04 (ND)	55-134	
	0811661-MSD1	10/9/08	10/10/08	10.90909 ug/L	9.590909	87.91667	0810133-04 (ND)	55-134	
	0811711-MS1	10/13/08	10/13/08	9.795918 ug/L	8.529592	87.07291	0810180-03	55-134	
	0811711-MSD1	10/13/08	10/14/08	9.795918 ug/L	7.880612	80.44791	0810180-03	55-134	
	0811858-MS1	10/15/08	10/16/08	9.6 ug/L	8.19	85.31249	0810220-33 (ND)	55-134	
	0811858-MSD1	10/15/08	10/16/08	9.6 ug/L	8.22	85.625	0810220-33 (ND)	55-134	
	0812029-MS1	10/20/08	10/25/08	9.69697 ug/L	8.555556	88.22917	0810321-06	55-134	
	0812029-MSD1	10/20/08	10/25/08	9.69697 ug/L	8.606061	88.75	0810321-06	55-134	
	0812461-MS1	10/27/08	10/29/08	9.795918 ug/L	8.714286	88.95833	0810435-08 (ND)	55-134	
	0812461-MSD1	10/27/08	10/29/08	9.795918 ug/L	8.5	86.77083	0810435-08 (ND)	55-134	
X	0812462-MS1	10/27/08	10/28/08	ug/L	0		0810452-01 (ND)	55-134	
X	0812462-MSD1	10/27/08	10/28/08	ug/L	0		0810452-01 (ND)	55-134	
X	0813364-MS1	11/13/08	11/17/08	ug/L	0		0811231-07	55-134	
X	0813364-MSD1	11/13/08	11/17/08	ug/L	0		0811231-07	55-134	
	0813840-MS1	11/24/08	12/2/08	9.494949 ug/L	7.909091	83.29787	0811481-04 (ND)	55-134	
	0813840-MSD1	11/24/08	12/2/08	9.494949 ug/L	7.585859	79.89362	0811481-04 (ND)	55-134	

TriMatrix Laboratories, Inc. - MS/MSD RPD for CHRYSENE

8270C Standard SVOCs IN Water Printed: Dec-08-08 11:15 by TCB

All Clients/Projects [8/25/2008 to 11/26/2008 11:59:59 PM]



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Project:	All Projects	Prepared By:	All Extractionists
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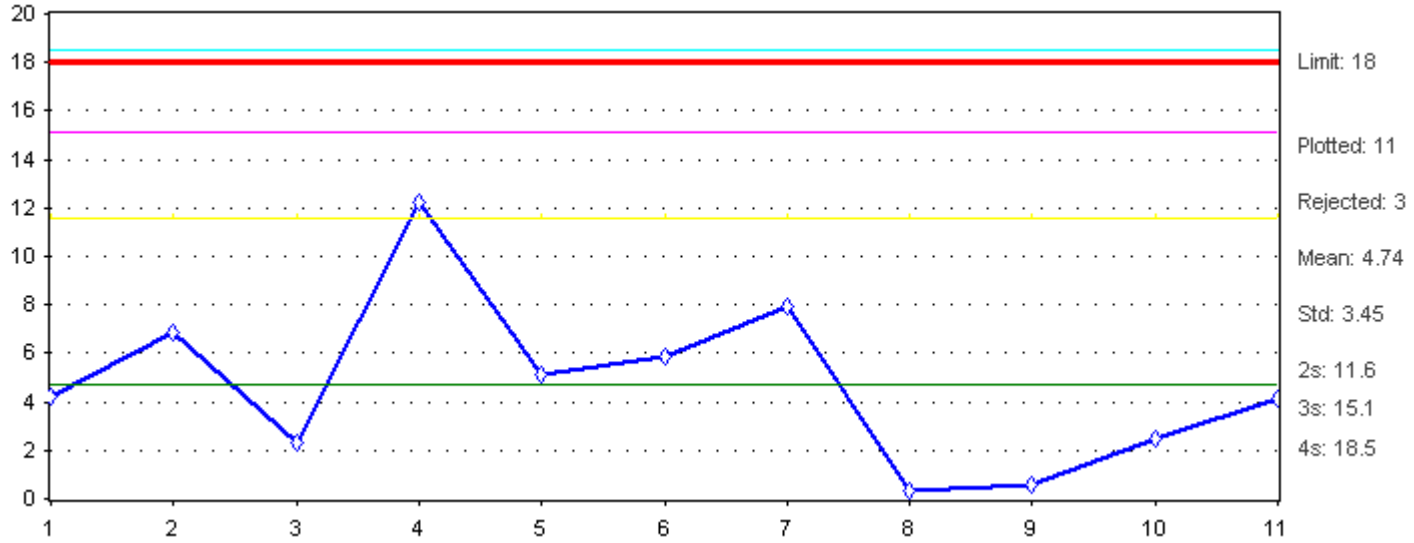
		Extractions:	All Extractions

Chrysene ***MS/MSD RPD***



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Dup	Dup %R	Source	RPD	Limit	Qualifier
	0809978-MSD1	8/29/08	9/2/08	9.69697 ug/L	5.373738	55.10417	5.606061	57.5	08570-08 (3.030303E-0	4.231829	18	
X	0810084-MSD1	9/2/08	9/4/08	9.6 ug/L	0.01	-0.5208333	5.29	54.58333	0808570-29 (0.05)		18	
	0810561-MSD1	9/12/08	9/18/08	96.9697 ug/L	93.66666	96.59373	100.2828	103.4166	0809136-03 (ND)	6.822543	18	
	0810561-MSD2	9/12/08	9/18/08	97.95918 ug/L	92.36735	94.29167	90.2347	92.11459	0809136-05 (ND)	2.335839	18	
	0810967-MSD1	9/23/08	9/27/08	97.95918 ug/L	85.59183	87.37499	75.7551	77.33333	0809311-06 (ND)	12.19327	18	
	0811107-MSD1	9/25/08	9/27/08	9.795918 ug/L	7.755102	79.16667	7.367347	75.20833	0809435-05 (ND)	5.128209	18	
	0811661-MSD1	10/9/08	10/10/08	10.90909 ug/L	9.590909	87.91667	9.045455	82.91668	0810133-04 (ND)	5.853653	18	
	0811711-MSD1	10/13/08	10/14/08	9.795918 ug/L	7.880612	80.44791	8.529592	87.07291	0810180-03	7.909465	18	
	0811858-MSD1	10/15/08	10/16/08	9.6 ug/L	8.22	85.625	8.19	85.31249	0810220-33 (ND)	.365639	18	
	0812029-MSD1	10/20/08	10/25/08	9.69697 ug/L	8.606061	88.75	8.555556	88.22917	0810321-06	.588577	18	
	0812461-MSD1	10/27/08	10/29/08	9.795918 ug/L	8.5	86.77083	8.714286	88.95833	0810435-08 (ND)	2.489628	18	
X	0812462-MSD1	10/27/08	10/28/08	ug/L	0		0		0810452-01 (ND)		18	
X	0813364-MSD1	11/13/08	11/17/08	ug/L	0		0		0811231-07		18	
	0813840-MSD1	11/24/08	12/2/08	9.494949 ug/L	7.585859	79.89362	7.909091	83.29787	0811481-04 (ND)	4.172097	18	

Appendix X

TriMatrix Laboratories, Inc.

Controlled Temperature Unit #55
Daily Log Sheet

Description: Fisher Isotemp Freezer
Model Number: 13-986-148
Serial #: 2017080504449
Location: Volatile Organic Laboratory

Purpose: Volatile Low-Level Soil Samples
Control Windows: Low: ≥ -20° C High: ≤ -7° C
Thermometer #: 184
Thermometer Serial #: 1353

Date	Initials	Time of First Reading	First Reading (°C)	Time of Second Reading	Second Reading (°C)	Weekend Minimum (°C)	Weekend Maximum (°C)	Adjustments/Observations/Comments

Appendix Y

TriMatrix Laboratories, Inc.

Balance Calibration Verification Acceptance Window Calculations

Balance ID: 204
Manufacturer: Mettler
Serial Number: J58563
Model Number: BB2440

Calibration Source: External
Calibration Weight (g): 1000
Calibration Weight Error (g): 0.00171811
Location: Volatiles Laboratory; South Bench Top

I. Calibration Weight Correction Calculations

Calibration Verification Weight Nominal Mass (g)	Calibration Verification Weight Correction Factor (g)	Calibration Verification Weight Actual Mass (g)	Linear Error of Balance (g)	Calibration Verification Weight Expected Mass (g)	Calibration Weight Combinations Used For Verification	
					Nominal	Actual
0.5000	0.00000190	0.5000	-0.00000086	0.5000	0.50	0.5000
5.0000	0.00000910	5.0000	-0.00000859	5.0000		
20.0000	0.00003540	20.0000	-0.00003436	20.0000	0.50 + 5 + 20	25.5000
100.0000	0.00006020	100.0001	-0.00017181	99.9999	0.50 + 100	100.4999

II. 20 Measurements Using Each Calibration Verification Mass

Date	Replicate Number	Mass 1 (g) 0.50	Mass 2 (g) 25.50	Mass 3 (g) 100.50			
1/29/2008	1	0.49	25.48	100.49			
1/29/2008	2	0.49	25.49	100.48			
1/29/2008	3	0.48	25.47	100.48			
1/29/2008	4	0.49	25.48	100.49			
1/29/2008	5	0.48	25.48	100.48			
1/29/2008	6	0.49	25.51	100.49			
1/29/2008	7	0.52	25.49	100.49			
1/30/2008	8	0.52	25.49	100.49			
1/30/2008	9	0.50	25.48	100.48			
1/30/2008	10	0.48	25.50	100.49			
1/30/2008	11	0.50	25.48	100.48			
1/30/2008	12	0.49	25.49	100.49			
1/30/2008	13	0.48	25.50	100.50			
1/30/2008	14	0.49	25.49	100.49			
1/31/2008	15	0.51	25.50	100.50			
1/31/2008	16	0.51	25.49	100.49			
1/31/2008	17	0.48	25.52	100.51			
1/31/2008	18	0.52	25.51	100.49			
1/31/2008	19	0.50	25.52	100.48			
1/31/2008	20	0.51	25.51	100.49			

III. Calibration Verification Acceptance Window Calculations

Standard Deviation:	0.01424411	0.01429022	0.00788069			
Random Error:	0.04273234	0.04287067	0.02364207			
Acceptance Window Low:	0.46	25.46	100.48			
Acceptance Window High:	0.54	25.54	100.52			

TriMatrix Laboratories, Inc.

Daily Balance Calibration Logbook

Balance ID: 204 **Serial #:** J58563 **Calibration Source:** External **Location:** Volatiles Laboratory;
Manufacturer: Mettler **Model #:** BB2440 **Calibration Weight (g):** 1000 South Bench Top

Date	Analyst	Calibration Verification Expected Mass 0.50	Window Pass / Fail 0.46 -0.54	Calibration Verification Expected Mass 25.50	Window Pass / Fail 25.46 -25.54	Calibration Verification Expected Mass 100.50	Window Pass / Fail 100.48 -100.52	Calibration Verification Expected Mass	Window Pass / Fail	Calibration Verification Expected Mass	Window Pass / Fail

Appendix Z

Analytical Standard Record

TriMatrix Laboratories, Inc.

7120763

Description:	8260 1 UG/L 12-28-07	Expires:	Feb-15-08
Standard Type:	Calibration Standard	Prepared:	Dec-28-07
Solvent:	WTR	Prepared By:	Diane L. VanMale
Final Volume (mls):	100	Department:	Volatiles MS
Vials:	1	Last Edit:	Dec-28-07 15:42 by DLV

Analyte	CAS Number	Concentration	Units
4-Methyl-2-pentanone (MIBK)	108-10-1	0.001	ppm
1-Chlorohexane	544-10-5	0.001	ppm
2,2-Dichloropropane	594-20-7	0.001	ppm
2-Butanone (MEK)	78-93-3	0.001	ppm
2-Chloroethyl Vinyl Ether	110-75-8	0.001	ppm
2-Chlorotoluene	95-49-8	0.001	ppm
2-Hexanone	591-78-6	0.001	ppm
2-Methylnaphthalene	91-57-6	0.001	ppm
4-Bromofluorobenzene	460-00-4	0.04	ppm
Carbon Disulfide	75-15-0	0.001	ppm
4-Isopropyltoluene	99-87-6	0.001	ppm
1,3-Dichloropropane	142-28-9	0.001	ppm
Acetone	67-64-1	0.001	ppm
Acrolein	107-02-8	0.001	ppm
Acrylonitrile	107-13-1	0.001	ppm
Benzene	71-43-2	0.001	ppm
Bromobenzene	108-86-1	0.001	ppm
Bromochloromethane	74-97-5	0.001	ppm
Bromodichloromethane	75-27-4	0.001	ppm
Bromoform	75-25-2	0.001	ppm
1,1,1,2-Tetrachloroethane	630-20-6	0.001	ppm
4-Chlorotoluene	106-43-4	0.001	ppm
1,2-Dibromo-3-chloropropane	96-12-8	0.001	ppm
1,1,1-Trichloroethane	71-55-6	0.001	ppm
1,1,2,2-Tetrachloroethane	79-34-5	0.001	ppm
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.001	ppm
1,1,2-Trichloroethane	79-00-5	0.001	ppm
1,1-Dichloroethane	75-34-3	0.001	ppm
1,1-Dichloroethene	75-35-4	0.001	ppm
1,1-Dichloropropene	563-58-6	0.001	ppm

Reviewed By Date

1,2,3-Trichlorobenzene	87-61-6	0.001	ppm
1,2,3-Trichloropropane	96-18-4	0.001	ppm
1,4-Dichlorobenzene	106-46-7	0.001	ppm
1,2,4-Trimethylbenzene	95-63-6	0.001	ppm
1,3-Dichloropropene (Total)	542-75-6	0.002	ppm
1,2-Dibromoethane	106-93-4	0.001	ppm
1,2-Dichlorobenzene	95-50-1	0.001	ppm
1,2-Dichloroethane	107-06-2	0.001	ppm
1,2-Dichloroethane-d4	17060-07-0	0.04	ppm
1,2-Dichloroethene (Total)	540-59-0	0.002	ppm
1,2-Dichloropropane	78-87-5	0.001	ppm
1,3,5-Trimethylbenzene	108-67-8	0.001	ppm
1,3-Dichlorobenzene	541-73-1	0.001	ppm
Carbon Tetrachloride	56-23-5	0.001	ppm
1,2,4-Trichlorobenzene	120-82-1	0.001	ppm
Total Trihalomethanes		0.004	ppm
n-Butylbenzene	104-51-8	0.001	ppm
n-Propylbenzene	103-65-1	0.001	ppm
Naphthalene	91-20-3	0.001	ppm
sec-Butylbenzene	135-98-8	0.001	ppm
Styrene	100-42-5	0.001	ppm
tert-Butylbenzene	98-06-6	0.001	ppm
Tetrachloroethene	127-18-4	0.001	ppm
Tetrahydrofuran	109-99-9	0.001	ppm
Bromomethane	74-83-9	0.001	ppm
Toluene-d8	2037-26-5	0.04	ppm
Methyl tert-Butyl Ether	1634-04-4	0.001	ppm
trans-1,2-Dichloroethene	156-60-5	0.001	ppm
trans-1,3-Dichloropropene	10061-02-6	0.001	ppm
trans-1,4-Dichloro-2-butene	110-57-6	0.001	ppm
Trichloroethene	79-01-6	0.001	ppm
Trichlorofluoromethane	75-69-4	0.001	ppm
Vinyl Acetate	108-05-4	0.001	ppm
Vinyl Chloride	75-01-4	0.001	ppm
Xylene (Total)	1330-20-7	0.003	ppm
Xylene, Meta + Para	136777-61-2	0.002	ppm
Toluene	108-88-3	0.001	ppm
Dichlorofluoromethane	75-43-4	0.001	ppm
Chlorobenzene	108-90-7	0.001	ppm

Reviewed By Date

Analytical Standard Record

TriMatrix Laboratories, Inc.

7120763

Chloroethane	75-00-3	0.001	ppm
Chloroform	67-66-3	0.001	ppm
Chloromethane	74-87-3	0.001	ppm
cis-1,2-Dichloroethene	156-59-2	0.001	ppm
cis-1,3-Dichloropropene	10061-01-5	0.001	ppm
Cyclohexane	110-82-7	0.001	ppm
Dibromochloromethane	124-48-1	0.001	ppm
Dibromofluoromethane	1868-53-7	0.04	ppm
Methylene Chloride	75-09-2	0.001	ppm
Dichlorodifluoromethane	75-71-8	0.001	ppm
Methylcyclohexane	108-87-2	0.001	ppm
Ethyl Ether	60-29-7	0.001	ppm
Ethylbenzene	100-41-4	0.001	ppm
Heptane	142-82-5	0.001	ppm
Hexachlorobutadiene	87-68-3	0.001	ppm
Hexachloroethane	67-72-1	0.001	ppm
Iodomethane	74-88-4	0.001	ppm
Isopropylbenzene	98-82-8	0.001	ppm
Methyl Acetate	79-20-9	0.001	ppm
Xylene, Ortho	95-47-6	0.001	ppm
Dibromomethane	74-95-3	0.001	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
7090571	8260 Centurian Working Surrogate	Sep-13-07	Diane L. VanMale	Aug-31-08	Sep-18-07 10:47 by DLV	0.1
7120760	8260 Working Standard B 12-28-07	Dec-28-07	Diane L. VanMale	Feb-15-08	Dec-28-07 15:20 by DLV	0.001

Reviewed By Date

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By
1	VO7. -1													
2	VO7. -2													
3	VO7. -3													
4	VO7. -4													
5	VO7. -5													
6	VO7. -6													
7	VO7. -7													
8	VO7. -8													
9	VO7. -9													
10	VO7. -10													
11	VO7. -11													
12	VO7. -12													
13	VO7. -13													
14	VO7. -14													
15	VO7. -15													
16	VO7. -16													
17	VO7. -17													
18	VO7. -18													

Appendix AA

TriMatrix Laboratories, Inc.

Pipet Calibration Verification Acceptance Window Calculations

Pipet ID:	SPK-15	**Balance Used:**	IN-1
Manufacturer:	Socorex	**Manufacturer:**	Mettler
Model Number:	Calibra 822.1000	**Model Number:**	AE-163
Serial Number:	10111410	**Serial Number:**	B86211

I. 20 Weight (g) Measurements Using Each Pipet Calibration Mass

Date	Replicate Number	Volume 1 uL 100	Volume 2 uL 200	Volume 3 uL 250	Volume 4 uL 300	Volume 5 uL 500	Volume 6 uL 1000
05/04/01	1	0.0975	0.1978	0.2455	0.2924	0.4904	0.9819
05/04/01	2	0.0981	0.1978	0.2461	0.2944	0.5077	0.9958
05/04/01	3	0.0987	0.1980	0.2458	0.2945	0.5028	1.0019
05/04/01	4	0.0983	0.1973	0.2475	0.2933	0.5041	1.0011
05/04/01	5	0.1002	0.1986	0.2479	0.2936	0.4993	1.0030
05/04/01	6	0.0997	0.1984	0.2474	0.2945	0.5001	1.0006
05/04/01	7	0.1000	0.1973	0.2471	0.2942	0.5005	1.0034
05/07/01	8	0.0983	0.1965	0.2449	0.2940	0.4950	0.9930
05/07/01	9	0.0975	0.1971	0.2451	0.2936	0.4939	0.9922
05/07/01	10	0.0970	0.1933	0.2440	0.2948	0.4943	0.9943
05/07/01	11	0.0972	0.1970	0.2447	0.2927	0.4939	0.9938
05/07/01	12	0.0973	0.1963	0.2452	0.2935	0.4935	0.9928
05/07/01	13	0.0966	0.1970	0.2445	0.2939	0.4934	0.9935
05/07/01	14	0.0977	0.1961	0.2438	0.2937	0.4935	0.9920
05/08/01	15	0.0992	0.1969	0.2464	0.2973	0.4937	0.9884
05/08/01	16	0.0990	0.1970	0.2463	0.2953	0.4913	0.9918
05/08/01	17	0.0989	0.1959	0.2479	0.2977	0.4924	0.9841
05/08/01	18	0.0981	0.2012	0.2474	0.2963	0.4932	0.9858
05/08/01	19	0.0985	0.1954	0.2469	0.2962	0.4948	0.9856
05/08/01	20	0.0990	0.1976	0.2462	0.2975	0.4930	0.9865

II. Pipet Calibration Acceptance Window Calculations

Standard Deviation:	0.00100755	0.00151653	0.00128600	0.00156309	0.00469383	0.00647504
Random Error:	0.00302265	0.00454960	0.00385799	0.00468928	0.01408148	0.01942513
Average Percent Recovery	98.3%	98.6%	98.4%	98.2%	99.2%	99.3%
Acceptance Window Low:	0.0970	0.1955	0.2461	0.2953	0.4859	0.9806
Acceptance Window High:	0.1030	0.2045	0.2539	0.3047	0.5141	1.0194

TriMatrix Laboratories, Inc.

Metals Laboratory Spiking Pipet Calibration Logbook

Pipet ID	Calibration Volume	Acceptance Window (g)	Date: Initials:		Date: Initials:		Date: Initials:		Date: Initials:		Date: Initials:	
			g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail	g Found	Pass/Fail
B-8	20 uL	0.0192-0.0208										
	50 uL	0.0495-0.0505										
	100 uL	0.0981-0.1019										
SPK-5	10 uL	0.0096-0.0104										
	25 uL	0.0245-0.0255										
	50 uL	0.0485-0.0515										
	100 uL	0.0982-0.1018										
SPK-12	4.00 mL	3.91-4.09										
	8.00 mL	7.84-8.16										
	9.00 mL	8.84-9.16										
	10.00 mL	9.85-10.15										
SPK-15	100 uL	0.0970-0.1030										
	200 uL	0.1955-0.2045										
	250 uL	0.2461-0.2539										
	300 uL	0.2953-0.3047										
	500 uL	0.4859-0.5141										
	1000 uL	0.9806-1.0194										
SPK-16	100 uL	0.0953-0.1047										
	200 uL	0.1944-0.2056										
	250 uL	0.2457-0.2543										
	300 uL	0.2918-0.3082										
	500 uL	0.4922-0.5078										
	1000 uL	0.9641-1.0359										

Appendix AB

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Digestion of Mercury in Water, Wastewater and Aqueous Waste

EPA Method 245.1
SW-846 Method 7470A

APPROVALS:

Area Supervisor: _____ Date: 9-12-08
Denise S. Coffey

QA Officer: _____ Date: 9-11-08
Tom C. Boocher

Operations Manager: _____ Date: 9/12/08
Jeff P. Glaser

Procedure Number: GR-01-140
Revision Number: 0.3

Date Initiated: 2/19/03
Effective Date: 10/15/08

Date Revised: 8/4/08
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 21

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the digestion of total mercury (inorganic and organic) in samples of groundwater, potable water, surface water, saline water, mobility leachate, and in aqueous domestic and industrial waste.

1.2 The minimum reporting limit is 0.2 ug/L.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

2.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, all client samples and quality control must be digested to convert organo-mercury complexes to inorganic mercury.

3.2 A measured sample aliquot, acids and potassium permanganate-potassium persulfate are transferred to a block digestion vessel and refluxed for 2 hours at 90-95° C.

3.3 The digestate is then prepared for analysis by semi-automated cold vapor atomic absorption spectrometry with the addition of hydroxylamine hydrochloride to reduce excess permanganate.

3.4 Inorganic mercury is converted to mercury in the Hg^{2+} state during the digestion, for detection and quantitation.

4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision

5.3 TriMatrix SOP GR-01-123, *Mercury in Aqueous and Solid Samples by Semi-Automated Cold Vapor Atomic Absorption Spectrometry*, latest revision

Appendix AC

TriMatrix Laboratories, Inc.

Sample Collection, Packing and Return

All supplied containers are pre-cleaned, no additional cleaning is required. Some containers have preservatives present in them. Please do not rinse or overfill. Removal of some or all of the preservative may result in qualified data. Most of the chemicals used as preservatives are hazardous. Use caution when handling. Do not breathe or come in physical contact with these chemicals. For your safety, please read the enclosed Material Safety Data Sheets.

When conducting soil sampling, please clean off any residual soil from the outside of the containers. This will help prevent cross contamination of other samples in the cooler.

Please fill out all sample identification tags as completely as possible.

Please fill out the enclosed Chain of Custody form for adequate sample tracking.

The temperature requirement for the receipt of most environmental samples is 4 ±2° C. Temperatures that exceed this range are subject to qualification and data rejection by regulatory agencies. Following the instructions below provides the best chance of achieving and maintaining this temperature and avoiding qualified data.

- Samples should be collected and placed on ice as soon as possible. It is much more difficult to cool down warm samples.
- When possible, sample containers should be sealed in zip-lock containers. This prevents cross contamination and protects the sample labels from moisture that could render them illegible.
- Do not overfill the cooler with samples. Overfilling the cooler limits the space available for ice.
- Surround the sides and the tops of the sample containers with loose, cubed, ice. Surrounding the samples with ice is the most efficient way of cooling them. Do not use individual small bags of ice. Do not simply lay a bag of ice on top of the samples.
- Place the temperature blank in a representative location in the cooler, not in the middle of a bag of ice.
- Secure all paperwork in a zip-lock bag and place in the cooler. Seal the cooler closed.
- When shipping the coolers back to TriMatrix, complete the enclosed FedEx Airbill and attach it to the cooler. Samples shipped during the week for standard overnight delivery typically arrive the next day between 9:00 and 10:00 a.m. Saturday deliveries must be approved by your project chemist. When shipping samples for a Saturday delivery, select Priority Overnight and Saturday Delivery on the FedEx Airbill.

Please call your TriMatrix project chemist at 1-616-975-4500 if you require any further instructions, or to notify them of the pending arrival of any non-scheduled samples.

Thank You,
TriMatrix Laboratories, Inc.

MATERIAL SAFETY DATA SHEET

Sodium hydroxide, 50 wt% solution in water

Section 1 - Chemical Product and Company Identification

MSDS Name: Sodium hydroxide, 50 wt% solution in water

Catalog Numbers: 25986-0000, 25986-0025, 25986-0050, 38021-0000, 38021-0025, 38021-5000

Synonyms: Caustic soda

Company Identification:
Acros Organics BVBA
Janssen Pharmaceuticalaan 3a
2440 Geel, Belgium

Company Identification: (USA)
Acros Organics
One Reagent Lane
Fair Lawn, NJ 07410

For information in the US, call: 800-ACROS-01

For information in Europe, call: +32 14 57 52 11

Emergency Number, Europe: +32 14 57 52 99

Emergency Number US: 201-796-7100

CHEMTREC Phone Number, US: 800-424-9300

CHEMTREC Phone Number, Europe: 703-527-3887

Section 2 - Composition, Information on Ingredients

CAS#	Chemical Name:	%	EINECS#	Hazard Symbols:	Risk Phrases:
1310-73-2	Sodium hydroxide	50	215-185-5	C	35
7732-18-5	Water	50	231-791-2		

Text for R-phrases: see Section 16

Hazard Symbols: C

Risk Phrases: 35

Section 3 - Hazards Identification

EMERGENCY OVERVIEW

Causes severe burns.

Potential Health Effects

Eye: Causes severe eye burns.

Skin: Causes skin burns. May cause deep, penetrating ulcers of the skin.

Ingestion: Causes gastrointestinal tract burns. Causes severe pain, nausea, vomiting, diarrhea, and shock. May cause corrosion and permanent tissue destruction of the esophagus and digestive tract.

Inhalation: Irritation may lead to chemical pneumonitis and pulmonary edema. Causes severe irritation of upper respiratory tract with coughing, burns, breathing difficulty, and possible coma. Causes chemical burns to the respiratory tract.

Chronic: Prolonged or repeated skin contact may cause dermatitis.

Section 4 - First Aid Measures

Eyes: Immediately flush eyes with plenty of water for at least 15 minutes, occasionally lifting the upper and lower eyelids. Get medical aid immediately.

Skin: Get medical aid immediately. Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Discard contaminated clothing in a manner which limits further exposure.

Ingestion: Do not induce vomiting. Get medical aid immediately.

Inhalation: Get medical aid immediately. Remove from exposure and move to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

Notes to Physician: Treat symptomatically and supportively.

Section 5 - Fire Fighting Measures

General Information: As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear. May react with metals and lead to the formation of flammable hydrogen gas.

Extinguishing Media: Use foam, dry chemical, or carbon dioxide.

Section 6 - Accidental Release Measures

General Information: Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks: Absorb spill with inert material (e.g. vermiculite, sand or earth), then place in suitable container.

Section 7 - Handling and Storage

Handling: Wash thoroughly after handling. Use with adequate ventilation. Do not allow water to get into the container because of violent reaction. Do not breathe dust, vapor, mist, or gas. Do not get in eyes, on skin, or on clothing. Use only in a chemical fume hood.

Storage: Store in a cool, dry place. Store in a tightly closed container. Store in a cool, dry, well-ventilated area away from incompatible substances. Corrosives area. Store under an inert atmosphere.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls:

Facilities storing or utilizing this material should be equipped with an eyewash facility and a safety shower. Use adequate ventilation to keep airborne concentrations low. Use adequate general or local exhaust ventilation to keep airborne concentrations below the permissible exposure limits.

Exposure Limits

CAS# 1310-73-2:

United Kingdom, WEL - STEL: 2 mg/m3 STEL

United States OSHA: 2 mg/m3 TWA

Belgium - TWA: 2 mg/m3 VLE

France - VME: 2 mg/m3 VME

Germany: 2 mg/m3 TWA (inhalable fraction)

Japan: 2 mg/m3 Ceiling

Malaysia: 2 mg/m3 Ceiling

Spain: 2 mg/m3 VLA-EC

CAS# 7732-18-5:

Personal Protective Equipment

Eyes: Wear chemical splash goggles.

Skin: Wear appropriate protective gloves to prevent skin exposure.

Clothing: Wear appropriate protective clothing to prevent skin exposure.

Respirators: Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

Section 9 - Physical and Chemical Properties

Physical State: Viscous liquid

Color: clear colorless

Odor: Not available

pH: >13

Vapor Pressure: Not available

Viscosity: Not available

Boiling Point: 145 deg C (293.00°F)

Freezing/Melting Point: 12 deg C (53.60°F)

Autoignition Temperature: Not available

Flash Point: Not available

Explosion Limits: Lower: Not available

Explosion Limits: Upper: Not available

Decomposition Temperature: Not available

Solubility in water: Soluble

Specific Gravity/Density: 1.525

Molecular Formula: HNaO

Molecular Weight: 40

Section 10 - Stability and Reactivity

Chemical Stability: Stable at room temperature in closed containers under normal storage and handling conditions. Absorbs carbon dioxide from the air.

Conditions to Avoid: Incompatible materials, exposure to air.

Incompatibilities with Water, acids, aluminum, chlorinated solvents,

Other Materials	copper, copper alloys, magnesium, phosphorus, zinc, tin, organic materials.
Hazardous Decomposition Products	Sodium oxide.
Hazardous Polymerization	Will not occur.

Section 11 - Toxicological Information

RTECS#:

CAS# 1310-73-2: WB4900000
CAS# 7732-18-5: ZC0110000

LD50/LC50:

RTECS:
CAS# 1310-73-2: Draize test, rabbit, eye: 400 ug Mild;
Draize test, rabbit, eye: 1% Severe;
Draize test, rabbit, eye: 50 ug/24H Severe;
Draize test, rabbit, eye: 1 mg/24H Severe;
Draize test, rabbit, skin: 500 mg/24H Severe;
.
RTECS:
CAS# 7732-18-5: Oral, rat: LD50 = >90 mL/kg;
.
Other:

Carcinogenicity: Sodium hydroxide - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.
Water - Not listed as a carcinogen by ACGIH, IARC, NTP, or CA Prop 65.

Other: See actual entry in RTECS for complete information.

Section 12 - Ecological Information

Not available

Section 13 - Disposal Considerations

Dispose of in a manner consistent with federal, state, and local regulations.

Section 14 - Transport Information

	IATA	IMO	RID/ADR
Shipping Name:	SODIUM HYDROXIDE SOLUTION	SODIUM HYDROXIDE SOLUTION	SODIUM HYDROXIDE SOLUTION
Hazard Class:	8	8	8
UN			

Number:	1824	1824	1824
Packing Group:	II	II	II

USA RQ: CAS# 1310-73-2: 1000 lb final RQ; 454 kg final RQ

Section 15 - Regulatory Information

European / International Regulations

European Labeling in Accordance with EC Directives

Hazard Symbols: C

Risk Phrases:

R 35 Causes severe burns.

Safety Phrases:

S 26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

S 37/39 Wear suitable gloves and eye/face protection.

S 45 In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

WGK (Water Danger/Protection)

CAS# 1310-73-2: 1

CAS# 7732-18-5: Not available

Canada

CAS# 1310-73-2 is listed on Canada's DSL List

CAS# 7732-18-5 is listed on Canada's DSL List

US Federal

TSCA

CAS# 1310-73-2 is listed on the TSCA Inventory.

CAS# 7732-18-5 is listed on the TSCA Inventory.

Section 16 - Other Information

Text for R-phrases from Section 2

R 35 Causes severe burns.

MSDS Creation Date: 7/16/1996

Revision #1 Date 5/05/2004

Revisions were made in Sections: General revision.

The information above is believed to be accurate and represents the best

information currently available to us. However, we make no warranty of merchantibility or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall the company be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential, or exemplary damages howsoever arising, even if the company has been advised of the possibility of such damages.

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR THE COLLECTION OF VOLATILE ORGANIC DRINKING WATER SAMPLES

Open the water tap and allow the system to flush until the water temperature has stabilized (usually about 10 minutes). Reduce the water flow and carefully collect a set of duplicate samples. It is important that the flow is slow enough that no air bubbles pass through the sample as the vial is being filled. Each 40 mL vial has been pre-preserved with 25 mg of ascorbic acid preservative. Fill sample vials to just overflowing, taking care not to flush out the ascorbic acid.

Prior to sealing the set of vials, each sample must also be preserved with 1:1 hydrochloric acid. Using the supplied eyedropper and vial of HCl, carefully add 2 drops of HCl to each vial. The HCl must be added after the collection of the sample. DO NOT add the HCl to the sample vial prior to collecting the sample.

CAUTION: The 1:1 HCl is very acidic. Handle with care.

NOTE: If the sample foams vigorously when the HCl is added, discard that set of samples. Collect a new set, omitting the addition of the HCl. These samples must be flagged as "not acidified" on the chain of custody.

Seal the vials, invert, and mix for 1 minute. Verify that the sealed and mixed vial is bubble and headspace free. Sample data generated from vials received with headspace will be qualified accordingly.

The samples must be chilled to about 4° C when collected, and maintained at that temperature until analysis. Samples must be packaged for shipment with sufficient ice to ensure they arrive at the laboratory with a substantial amount of ice remaining in the cooler. Do not use Blue Ice. Surrounding the samples with crushed or cubed ice is strongly recommended. Samples received at the laboratory within 6 hours of collection may not have sufficient time to cool to 4° C. Provided that they have been correctly packed in ice, no qualifications will be necessary. Samples received in excess of 6 hours of the time of collection that exceed the required preservation temperature will be qualified accordingly.

Please call 1-616-975-4500 and speak to your project chemist if you have any questions. Thank you.

Dissolved Sulfide Sample Collection and Preservation

To measure dissolved sulfide, insoluble matter in the sample must first be removed. This is accomplished by producing an aluminum hydroxide floc using sodium hydroxide and aluminum chloride. The flocculent is allowed to settle and the supernatant decanted off and preserved with zinc acetate. It is important that there is no headspace present in the bottle after the addition of the aluminum chloride. The vials containing the final decanted sample must also be headspace free. If you have any questions on the treatment procedures described below, please contact your project chemist at 1-616-975-4500.

Supplies

Quantity	*Item*
1 per sample	250 mL amber bottle containing 0.5 mL (10 drops) 6N NaOH
2 per sample	40 mL VOA vials, each containing 0.1 mL (2 drops) 2N Zinc Acetate per sample
2 or 3	eye droppers
1	Container of Aluminum Chloride. Enough has been sent to allow for the addition of 10 drops (0.5 mL) to each 250 mL sample.

Procedure

1.0 Collect the sample in the 250 mL amber bottle containing the NaOH. Completely fill the bottle (must be enough sample so when capped it is headspace free).

2.0 Immediately add 10 drops of the Aluminum Chloride solution.

3.0 Mix the sample by holding the bottle in an upright position and rotating your wrist back and forth for 1 minute.

4.0 Allow the sample to settle for 5 to 15 minutes (long enough to allow the flocculent to settle to the bottom of the bottle but not longer than 15 minutes). Wait only as long as necessary to collect 80 mL of supernatant.

5.0 Carefully decant the supernatant into the (2) 40 mL VOA vials containing the 2N zinc acetate. Completely fill the vials with sample so they are headspace free.

6.0 The sample remaining in the 250 mL amber bottle is caustic. Please return the partially filled bottle to TriMatrix for disposal.

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR SULFIDE SAMPLE COLLECTION

The amber, 500 mL, light green-tagged bottles supplied for sulfide sample collection have been pre-preserved with 1 mL of 2N zinc acetate. Sulfide samples must also be preserved with sodium hydroxide to a pH of ≥9; however, to correctly preserve the sulfide in the sample the addition of the sodium hydroxide must be made *after* the sample has been combined with the zinc acetate. A 4 mL vial containing 2 mL of 10N sodium hydroxide has been included with every 500 mL sulfide sample bottle for this purpose.

With a minimum of aeration, fill a 500 mL bottle up to the neck with sample. Cap and gently swirl to mix the sample and the zinc acetate. Open the sample bottle and transfer all of the sodium hydroxide from one of the 4 mL vials. Carefully add more sample to fill the 500 mL bottle, cap and mix. The filled sample container should be headspace free.

CAUTION: The 10N sodium hydroxide solution is very caustic. Handle with care.

Please call 1-616-975-4500 and speak to your project chemist with any questions. Thank you.

TriMatrix Laboratories, Inc.

IMPORTANT INFORMATION FOR AVAILABLE CYANIDE SAMPLE COLLECTION

Two sample containers must be collected at each sample point. One container will be treated with lead carbonate and sodium hydroxide, and the second with only sodium hydroxide (see below and the attached flowchart). A form titled "Available Cyanide Sample Treatment Record" has been provided to document all field pre-treatment activities. Please complete it as you collect and treat each sample. If you have any questions on the treatment procedures described below, please contact your project chemist at 1-616-975-4500.

IMPORTANT: To avoid analyte loss it is **required** that all sample treatments occur within 15 minutes of sample collection.

CAUTION: All containers labeled as Sodium Hydroxide and Lead Carbonate/Sodium Hydroxide contain 1.3 mL of 10N sodium hydroxide. This solution is very caustic. Avoid skin contact. Handle with care.

CAUTION: All containers labeled as Lead Carbonate contain 0.25 g of solid lead carbonate. Avoid inhalation and skin contact.

1.0 Sample Collection Equipment

Per Sample
- One membrane filter
- One plastic powder funnel
- One sheet of filter paper
- One Lead Carbonate bottle
- One Lead Carbonate/Sodium Hydroxide bottle
- One Sodium Hydroxide bottle

A hand pump (not provided) is also required to perform this procedure

2.0 Collecting a Lead Carbonate/Sodium Hydroxide Pre-Treated Sample

If the sample contains particulates, begin with section 2.1. If the sample is particulate free, begin with section 2.2.

2.1 Sample Contains Particulate Matter

If the sample contains particulate matter that would be removed upon filtration, the sample must be filtered prior to the lead carbonate pre-treatment to avoid the loss of any cyanides associated with the particulate matter. Using a powder funnel and a sheet of filter paper, filter the sample into the bottle labeled Lead Carbonate. Filter enough sample to fill the bottle up to its neck. Place the used filter paper into the bottle labeled Lead Carbonate/Sodium Hydroxide. Cap the Lead Carbonate bottle and gently swirl to mix the sample and the lead carbonate. The sulfide will react with the lead carbonate

and precipitate out as lead sulfide. The sample must now be filtered through a membrane filter to prevent the loss of any cyanide through reaction with the precipitated lead sulfide. Using a new membrane filter apparatus and a hand pump, filter the sample. Transfer the filtrate into the Lead Carbonate/Sodium Hydroxide bottle containing the used filter paper. Do not pre-rinse the container or fill to overflowing, as a loss of the particulate matter and sodium hydroxide will result. Proceed to section 3.0.

2.2 Sample Particulate Free

With a minimum of aeration, fill the 250 mL bottle labeled Lead Carbonate up to the neck with sample. Cap and gently swirl to mix the sample and the lead carbonate. The sulfide will react with the lead carbonate and precipitate out as lead sulfide. The sample must now be filtered through a membrane filter to prevent the loss of any cyanide through reaction with the precipitated lead sulfide. Using a new membrane filter apparatus and a hand pump, filter the sample. Transfer the filtrate collected into the bottle labeled Lead Carbonate/Sodium Hydroxide. Do not pre-rinse the container or fill to overflowing to avoid the loss of the sodium hydroxide.

3.0 Collecting a Sodium Hydroxide Pre-Treated Sample

With a minimum of aeration fill the 250 mL bottle labeled Sodium Hydroxide with sample. Do not pre-rinse the container or fill to overflowing to avoid the loss of the sodium hydroxide.

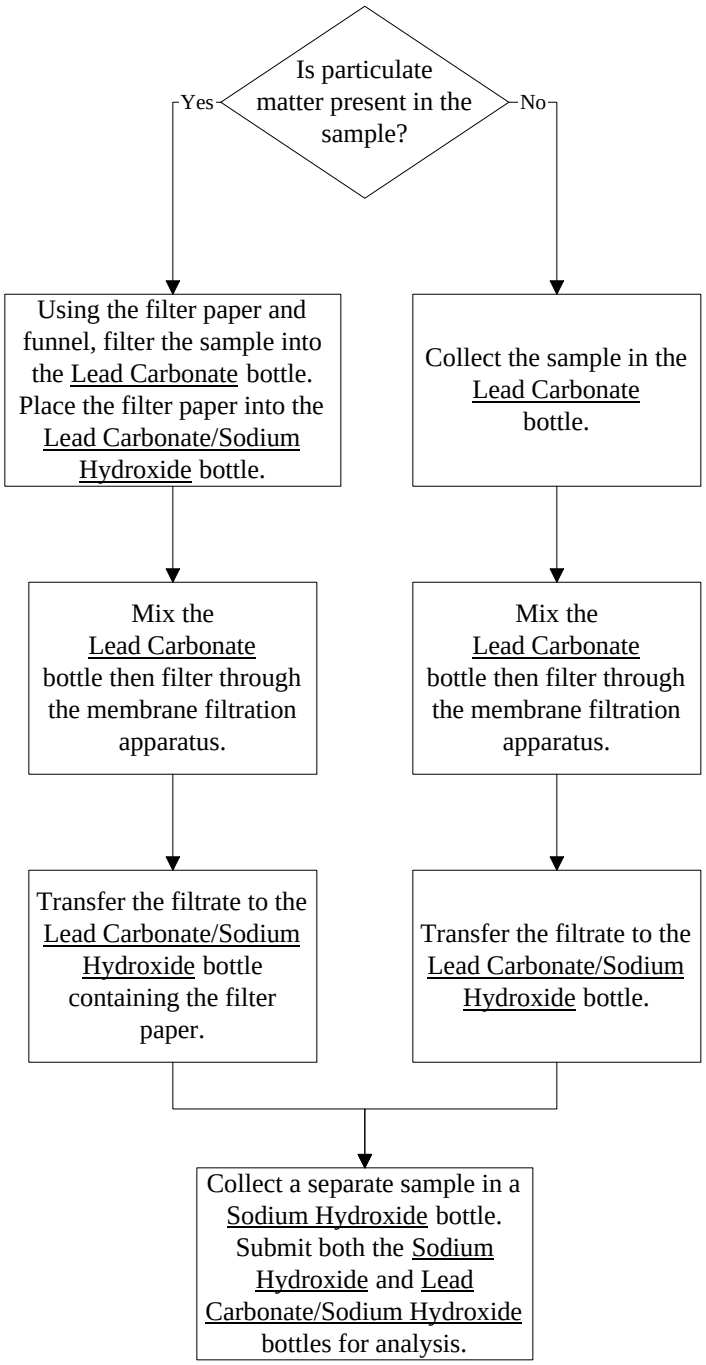
4.0 Collect all Paperwork and Return the Samples to TriMatrix

Place all samples in the cooler. Surround the samples with ice. To avoid data qualification all samples must be received at a temperature of between 0 and 6° C. Seal all paperwork in the resealable bag. Place the sealed bag containing the paperwork. Place all plastic powder funnels and unopened membrane filters in the cooler. Seal the cooler and return it to TriMatrix.

If you have any questions, please call TriMatrix at 1-616-975-4500 and speak with your project chemist. Thank you.

TriMatrix Laboratories, Inc.

Available Cyanide Sample Collection Flowchart



Appendix AD

TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court SE Grand Rapids, MI 49512
Phone (616) 975-4500 Fax (616) 942-7463
www.trimatrixlabs.com

Chain of Custody Record

COC No.

For Lab Use Only

Cart

VOA Rack/Tray

Receipt Log No.

Project Chemist

Work Order No.

Client Name

Address

Phone

Fax

Project Name

Client Project No. / P.O. No.

Invoice To
(●) Client ( ) Other (comments)

Contact/Report To

Analyses Requested

⇦ PRESERVATIVES

A NONE pH~7
B HNO_3 pH<2
C H_2SO_4 pH<2
D 1+1 HCl pH<2
E NaOH pH>12
F ZnAc/NaOH pH>9
G MeOH
H Other (note below)

Container Type (corresponds to Container Packing List)

Number of Containers Submitted

Schedule	Matrix Code	Laboratory Sample Number		Sample ID	Cooler ID	Sample Date	Sample Time	Comp / Grab	Matrix	Total	Sample Comments
			1								
			2								
			3								
			4								
			5								
			6								
			7								
			8								
			9								
			10								

Sampled By (print)

Sampler's Signature

Company

How Shipped?

Tracking No.

Comments

1. Relinquished By Date Time

1. Received By Date Time

2. Relinquished By Date Time

2. Received By Date Time

3. Relinquished By Date Time

3. Received For Lab By Date Time

Appendix AE

TriMatrix Laboratories, Inc.

pH Strip Calibration Logbook

Date	Lot #	pH 4	pH 7	pH 10	Area

pH STRIP CALIBRATION CRITERIA CORRECTIVE ACTION

1. The acceptance range for the strips is to read the exact pH of the buffer being checked. The wide range strips must pass this criteria at all three levels, 4, 7, and 10. The narrow range pH 5-7 strips are only checked at a pH of 7.

2. If the pH strips do NOT read at their appropriate levels, that lot number must NOT be used. Return them to purchasing.

Appendix AF

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

The collection of the sample is the starting point for the generation of quality data. It is the responsibility of TriMatrix to provide the client who collects the sample with sample collection instructions, which ensure sample integrity. Also, where applicable TriMatrix also supplies the client with appropriate clean sample containers and preservative chemicals; these glass containers are purchased new and certified as clean and vendors such as I-Chem Research and Fischer Scientific.

Sampling and Preservation Requirements for certain common environmental analyses are listed in the following table: (NOTE: Holding times are based on EPA guidelines for CLP, NPDES, and RCRA).

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
ORGANICS							
Volatile	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	8015, 8021, 8260	Yellow/Black
Halocarbons*	Water	14 days	4° C	2-40 mL VOA vials	40 mL each	601	Yellow/Black
	Water	14 days	4° C/HCl to pH <2	2-40 mL VOA vials	40 mL each	601, 8015, 8021, 8260	Yellow
	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar	fill the jar	8015, 8021, 8260	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8015, 8021, 8260	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8015, 8021, 8260	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8015, 8021, 8260	Light Yellow
Volatile	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	602	Yellow/Black
Aromatics*	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	602, 8021, 8260	Yellow
	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar or	fill the jar	8021, 8260	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8021, 8260	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8021, 8260	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8021, 8260	Light Yellow
Acrolein*	Water	3 days	4° C	2-40 mL VOA vials	40 mL each	624	Yellow/Black
	Water	14 days	4° C/HCl to pH 4-5	2-40 mL VOA vials	40 mL each	624	Yellow
Acrylonitrile*	Water	14 days	4° C	2-40 mL VOA vials	40 mL each	624	Yellow/Black
	Water	14 days	4°C/HCl to pH 4-5	2-40 mL VOA vials	40 mL each	624	Yellow
TPH-GRO	Water	7 days	4° C	2-40 mL VOA vials	40 mL each	8015	Yellow/Black
	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	8015	Yellow
TPH-GRO/PVOC	Water	14 days	4° C/HCl to pH <2.0	2-40 mL VOA vials	40 mL each	Wisconsin PUBL-SW-140	Yellow
TPH-GRO	Soil/Waste (High Level Bulk)	14 days	4° C	60 mL glass jar or	fill the jar	8015	Light Yellow
	Soil (Low Level Bisulfate)	14 days	4° C/5 mL sodium bisulfate	2-pre-tared 40 mL VOA vials each containing 5 mL of 20% sodium bisulfate and a stir bar	5 g each	8015	Light Yellow
	Soil (Encore)	48 hours/14 days	4° C	10 or 25 g Encore	10 or 25 g	8015	Label on Bag
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	8015	Light Yellow
TPH-GRO/PVOC	Soil (Encore)	48 hours/21 days	4° C	10 or 25 g Encore	See Table 1 in Method	Wisconsin PUBL-SW-140	Label on Bag

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
	Soil (MeOH Preserved)	14 days	4° C	Pre-tared 40 mL VOA vial and 10 mL ampule of methanol	10 g	Wisconsin PUBL-SW-140	Light Yellow
Petroleum	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8015	Salmon
Hydrocarbons	Water	7 days/47 days	4° C/HCl to pH <2.0	1000 mL amber glass bottle	1000 mL	Wisconsin PUBL-SW-141	Gray
(DRO)	Soil/Waste (High Level Bulk)	14 days/54 days	4° C	60 mL glass jar or	fill the jar	8015	Manila
	Soil/Waste	10 days/47 days	4° C	Tared VOC vial	See Table 1 in Method	Wisconsin PUBL-SW-141	Gray
Pesticides	Water	7 days/47 days	4° C/pH 5-9	1000 mL amber glass bottle	1000 mL	608	Yellow/White
PCBs	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	608, 8082	Salmon
Methoxychlor	Water	7 days/47 days	4° C/pH 6-8	1000 mL amber glass bottle	1000 mL	608.2	Yellow/White
Pesticides	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8081	Manila
PCBs	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8082	Manila
PCB Oils	Oil	N/A	None	40 mL VOA vial	20 mL	8082	Manila
Organo-phosphorous	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8141	Salmon
Pesticides	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8141	Manila
Phenoxy Acid Herbicides	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8151	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8151	Manila
Polynuclear aromatic	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	610, 8100	Salmon
Hydrocarbons*	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8310, 8270	Manila
Acid Extractables	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8041, 8270	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8041, 8270	Manila
Base/Neutral Extractables	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	8270	Salmon
	Soil/Waste	14 days/54 days	4° C	60 mL glass jar	fill the jar	8270	Manila
TCLP-							
Volatiles	Soil/Waste	14 days/28 days	4° C	60 mL glass jar	100 g	1311	Yellow/Black
Semi-Volatiles	Soil/Waste	14 days/21 days/61 days	4° C	125 mL glass jar	250 g	1311	Manila
Metals	Soil/Waste	180 days/360 days (Hg-28 days/56 days)	None	125 mL glass jar	250 g	1311	Manila
Pesticide/Herbicide	Soil/Waste	14 days/21 days/61 days	4° C	125 mL glass jar	250 g	1311	Manila
Dioxins/	Water	7 days/47 days	4° C	1000 mL amber glass bottle	1000 mL	Screen-625	Salmon

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Furans							
	Soil/Waste	None Required	4° C	60 mL glass jar	fill the jar	Screen-625	Manila

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
METALS							
Metals, Total (including phosphorus)	Water	6 months	HNO_3 to pH <2.0	500 mL plastic bottle	500 mL	6010/6020/200.7/200.8	Red
Metals, Dissolved (including phosphorus)	Water	6 months	HNO_3 to pH <2.0	500 mL plastic bottle	500 mL	6010/6020/200.7/200.8	Red/White Stripe
	Soil/Waste	6 months	None	250 mL plastic bottle	50 g	6010/6020	White
Mercury Cold Vapor	Water	28 days	HNO_3 to pH <2.0	500 mL plastic bottle	500 mL	245.1, 7470	Red
	Soil/Waste	28 days	4° C	250 mL plastic bottle	50 g	7471	White
Low-Level	Water	28 days	None	500 mL borosilicate glass bottle**	500 mL	1631	Label on Bag

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
INORGANICS							
Color (Apparent)	Water	48 hours	4° C	125 mL plastic bottle	100 mL	110.2	Green
Color (True)	Water	48 hours	4° C	125 mL plastic bottle	100 mL	110.2	Green
Oil & Grease (HEM and SGT)	Water	28 days	4° C/H_2SO_4 to pH <2.0	1000 mL glass bottle	1000 mL	9070/1664	Dark Blue
	Soil/Waste	28 days	None	60 mL glass jar	50 g	9071	Manila
Specific Conductance	Water	28 days	4° C	125 mL plastic bottle	100 mL	2510 B./120.1/9050	Green
Acidity	Water	14 days	4° C	125 mL plastic bottle	100 mL	2310 B.	Green
pH	Water	24 hours	4° C	125 mL plastic bottle	100 mL	150.1/9041/4500-H B.	Green
	Soil/Waste	24 hours	4° C	60 mL glass jar	50 g	9040/9041/9045	
Alkalinity	Water	14 days	4° C	125 mL plastic bottle	100 mL	310.1/2320 B.	Green
Hardness	Water	6 months	HNO_3 to pH <2.0	125 mL plastic bottle	100 mL	130.2/2340 C.	Red
Biochemical Oxygen Demand (BOD)	Water	48 hours	4° C	1000 mL plastic bottle	1000 mL	5210 B.	Green
Chemical Oxygen Demand (COD)	Water	28 days	4° C/H_2SO_4 to pH <2.0	125 mL plastic bottle	100 mL	410.4/5220 D.	Dark Blue
Chromium (Hexavalent)	Water	24 hours	4° C	500 mL plastic bottle	500 mL	7196A, 3500-Cr B.	Green
	Soil/Waste	30 days/24 hours	4° C	60 mL glass jar	50 g	7196A	Manila
Organic Carbon (TOC)	Water	28 days	4° C/H_2SO_4 to pH <2.0	3-40 mL VOA vials	40 mL	415.1/5310 D./9060	Salmon
	Soil/Waste	28 days	4° C	60 mL glass jar	10 g	MSA 29-3.5.2/415.1/9060	Manila

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Ortho-Phosphate	Water	48 hours	4° C	125 mL plastic bottle	100 mL	365.1/4500-P E.	Green
Total Phosphorus	Water	28 days	H_2SO_4 to pH <2.0	125 mL plastic bottle	100 mL	365.1/4500-P F.	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	365.1/4500-P F.	Manila
Total Kjeldahl Nitrogen (TKN)	Water	28 days	4° C/H_2SO_4 to pH <2.0	125 mL plastic bottle	100 mL	351.2	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	351.2	Manila
Ammonia	Water	28 days	4° C/H_2SO_4 to pH <2.0	125 mL plastic bottle (500 mL for wastewater)	100 mL (200 mL for wastewater)	350.1/4500-NH_3 G.	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	350.1/4500-NH_3 G.	Manila
Nitrite	Water	48 hours	4° C	125 mL plastic bottle	100 mL	300.0/9056/353.2/354.1/ 4500 NO_2-B/4500 NO_2-F	Green
	Soil/Waste	28 days/48 hours	4° C	60 mL glass jar	50 g	353.2/9056	Manila
Nitrate	Water	48 hours	4° C	125 mL plastic bottle	100 mL	300.0/9056/353.2/4500 NO_3-F	Green
	Soil/Waste	28 days/48 hours	4° C	60 mL glass jar	50 g	9056/353.2/4500 NO_3-F	Manila
Nitrite plus Nitrate (No distinction between NO_2 and NO_3)	Water	28 days	4° C/H_2SO_4 to pH <2.0	125 mL plastic bottle	100 mL	353.2/4500 NO_3-F	Dark Blue
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	353.2/4500 NO_3-F	Manila
Total Volatile Solids	Water	7 days	4° C	125 mL plastic bottle	100 mL	160.4	Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	2540-G	Manila
Turbidity	Water	48 hours	4° C	125 mL plastic bottle	100 mL	180.1/2130 B.	Green
Sulfate	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/375.4/9038	Green
	Soil/Waste	28 days	4° C	60 mL glass jar	50 g	9056/375.2/9038/4500 SO_4-F	Manila
Sulfite	Water	48 hours	4° C/3 mL 1% EDTA	125 mL plastic bottle	100 mL	377.1	Manila

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Sulfide, Total	Water	7 days	4° C/Pre-Preserved with Zinc Acetate; NaOH Added in field to pH ≥9	125 mL plastic bottle	100 mL	9034/376.1/376.2/4500 S_2-D 4500 S_2-F	Light Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	9034	Manila
Cyanide*	Water	14 days	4° C/NaOH to pH >12	1000 mL plastic bottle	1000 mL	335.2/335.4/9012/9014	Light Blue
	Soil/Waste	14 days	4° C	60 mL glass jar	50 g	9012/9014	Manila
Cyanide, Available	Water	14 days	1 Lead Carbonate bottle 1 Lead Carbnate/NaOH bottle 1 NaOH bottle	125 mL amber glass bottles	125 mL	OIA-1677	Light Blue
Coliform Fecal and Total	Water	24 hours	4° C/$Na_2S_2O_3$	Sterile plastic bottle or Whirl-Pak	100 mL	9222-D/9223-B	White
Bromide	Water	28 days	4° C	125 mL plastic bottle	100 mL	9056/ASTM D1246-88	Green
Chloride	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/325.2/4500-Cl E.	Green
	Soil	28 days	4° C	60 mL glass jar	50 g	9056/325.2/4500-Cl E.	Manila
Chlorine Residual	Water	Analyze Immediately	4° C	125 mL plastic bottle	100 mL	HACH-8167	Green
Total Solids (% Moisture)	Water	7 days	4° C	125 mL plastic bottle	100 mL	160.3/2540 B.	Green
	Soil/Waste	7 days	4° C	60 mL glass jar	50 g	3550	Manila
Total Dissolved Solids (TDS)	Water	7 days	4° C	1000 mL plastic bottle	1000 mL	160.1/2540 C.	Green
Total Suspended Solids (TSS)	Water	7 days	4° C	1000 mL plastic bottle	1000 mL	160.2/2540 D.	Green
Fluoride	Water	28 days	4° C	125 mL plastic bottle	100 mL	300.0/9056/4500-F C.	Green
	Soil	28 days	4° C	60 mL glass jar	50 g	9056	Manila

SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (from Date Sampled)	Preservation	Container	Minimum Sample Size	Method Reference	Container Tag Color
Organic Halogen (TOX)	Water	28 days	4° C/H_2SO_4 to pH <2.0	500 mL amber glass bottle	500 mL	9020	Lilac
	Soil	28 days	4° C	60 mL glass jar	50 g	9023	Manila
Phenolics	Water	28 days	4° C/H_2SO_4 to pH <2.0	500 mL amber glass bottle	100 mL	420.2/420.4/9066	Brown
	Soil	28 days	4° C	60 mL glass jar	50 g	9066	Manila
Surfactants (MBAS)	Water	48 hours	4° C	1000 mL plastic bottle	400 mL	425.1/5540 C.	Green
Flash Point	Solid/Liquid/Waste	N/A	None	Clear glass wide mouth jar. 60 mL unless otherwisespecified.	100 g	1010/1020	White
	Waste	N/A	None		100 g	1010/1020	White
Corrosivity (pH and Method 1110)	Waste	N/A	None	(Appropriate to Sample) 500 mL glass or plastic bottle	500 mL	9040/9041/1110	White
Paint Filter (Free Liquids)	Soil/Waste	N/A	None	(Appropriate to Sample) 250 mL glass jar or 125 mL plastic bottle	100 g	9095	White
Radiologicals (Alpha + Beta, Alpha, Beta, Ra 226, Ra 228	Water	6 months	HNO_3 to pH <2.0	1000 mL plastic bottles or 1000 mL glass bottle	1000 mL		White
Reactivity (Releasable CN and S)	Waste	14 days CN, 7 days S	4° C	(Appropriate to Sample 125 mL plastic bottle or 60 mL glass jar	10 g	SW- 846 Chapter 7	White

*Sample must also be preserved with Sodium Thiosulfate or Ascorbic Acid if chlorinated
**All low-level mercury bottles are stored filled with 5 mL of concentrated HCl and Millipore water
NOTE: For Organics parameters, container lid should be Teflon.
NOTE: For Inorganic parameters, container lid should be plastic or Teflon lined.
NOTE: When testing for several like parameters (ICP metals, Ion Chromatograph anions), one container per sample is sufficient. For example, a sample to be tested for the 13 priority pollutant metals needs one 500 mL container.

Appendix AG

Client: **S**
Project: **Analytical Services**

Project Manager: **Jennifer L. Rice**
Date Received: **Dec-08-08 10:00**

Department: **Metals** Analysis: _____

Lab Number / Sample Name	*Container*	*Removed by (Signature)*	*Date & Time Removed*	*Date & Time Returned*	*Consumed?*	*Extract Container*
0812144-01 Pugged CKD						
0812144-02 Pugged CKD						
0812144-03 Pugged CKD						
0812144-04 Pugged CKD						

Appendix AH

TriMatrix Laboratories, Inc.

Non-Conformance Investigation Report

Client:_____ **Project Number:**_____

Sample Number(s):_____ **Date Initiated:**_____ **Date Due:**_____

Initiated By:_____ **Document Control Number:** _____

Investigation Resulting From: ☐ Internal Observation ☑ **Client Complaint** ☐ Audit ☐ Failing PT Sample

I. Area of Non-Conformance:

☐ Sample Receiving / Storage ☐ Bottle Prep ☐ Client Services / Reporting ☐ Other_____

☑ Inorganic (**Wet Chemistry** / Metals) Laboratory ☐ Organic (Volatile / Semi-Volatile / Extraction) Laboratory

II. Description of Non-Conformance:

_____

_____

_____

_____

III. Explanation of Investigation into Non-Conformance:

_____

_____

_____

_____ Initials: _____ Date: _____

IV. Resolution:

_____

_____

_____

_____

_____ Initials: _____ Date: _____

V. Follow-Up (if required):

_____

_____

_____

_____ Initials: _____ Date: _____

VI. Reviewed By:

QA Manager:_____ **Area Manager:**_____

Date Completed:_____

Appendix AI

TriMatrix Laboratories, Inc.

Preventive Action Investigation

Initiated By: _____ **Document Control Number:** _____

Date Initiated: _____ **Date Due:** _____

Investigation Resulting From: ☐ Internal Observation ☐ Client Complaint ☐ Audit ☐ Failing PE Sample

I. Area of Preventive Action:

☐ Sample Receiving / Storage ☐ Bottle Prep ☐ Client Services / Reporting ☐ Other _____

☐ Inorganic (Wet Chemistry / Metals) Laboratory ☐ Organic (Volatile / Semi-Volatile / Extraction) Laboratory

II. Description and Proposed Solutions:

_____

_____

_____

_____

III. Action Plan and Implementation Schedule:

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____ Initials: _____ Date: _____

V. Follow-Up to Monitor Effectiveness:

_____

_____

_____

_____

_____

_____ Initials: _____ Date: _____

VI. Reviewed By:

QA Manager: _____ **Area Manager:** _____

Date Completed: _____

8.0 GLOSSARY OF TERMS

ABSORBANCE - a measure of the decrease in incident light passing through a sample into the detector. It is defined mathematically as:

$$A = \left(\frac{I\,(solvent)}{I\,(solution)} \right) - \frac{\log Io}{I}$$

ALIQUOT - a measured portion of a field sample taken for analysis.

ANALYSIS DATE/TIME - the date and time of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE - the element or ion an analysis seeks to determine; the component of interest.

ANALYTICAL SAMPLE - any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification and continuing calibration blank. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, interference check samples (ICS), CRDL standard for AA (CRA), CRDL standard for ICP (CRI), laboratory control sample (LCS), method preparation blank (MPB), laboratory fortified blank (LFB), and linear range analysis sample (LRS).

AUTOZERO - zeroing the instrument at the proper wavelength. It is equivalent to running a blank to set the absorbance to zero.

AVERAGE INTENSITY - the average of two different responses from a detector.

BACKGROUND CORRECTION - a technique to compensate for background contribution to the instrument signal in the determination.

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank, Instrument Blank, Storage Blank, and Sulfur Blank.

BATCH - a group of samples prepared at the same time in the same location using the same method.

BREAKDOWN - a measure of the decomposition of certain analytes (i.e. DDT and Endrin) into by-products.

4-BROMOFLUOROBENZENE (BFB) - the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses.

CALIBRATION - the establishment of an analytical curve based on the measured response of known standards.

CALIBRATION BLANK - a volume of laboratory reagent or other inert carrier matrix.

CALIBRATION STANDARDS - a series of known standards used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected during external calibration. The calibration factor is analogous to the Response Factor (RF) calculated during internal calibration.

CASE - a finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office. A Case consists of one or more Sample Delivery Groups.

CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

CONTINUING CALIBRATION VERIFICATION - analytical standard run at periodic intervals to verify the initial calibration of the system.

CONTRACT REQUIRED DETECTION LIMIT (CRDL) - minimum level of detection acceptable as specified by the project to report.

CONTROL LIMITS - a range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

CORRELATION COEFFICIENT - the number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more dependent they are the closer the number (r) is. Determined on the basis of the least squares regression.

DAY - unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - an official record of the sample preparation (digestion).

DISSOLVED METALS - analyte elements which have not been digested prior to analysis and which will pass through a 0.45 um filter.

DRY WEIGHT - the weight of a sample analyzed based on percent solids. The weight after drying in an oven.

DUPLICATE - a second aliquot of sample that is treated the same as the original in order to determine the precision of the collection.

EXTRACTED ION CURRENT PROFILE (EICP) - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).

EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.

FIELD BLANK - any sample submitted from the field identified as a blank.

FIELD SAMPLE - Material received to be analyzed that is contained in single or multiple containers and identified by a unique Sample Number.

GAS CHROMATOGRAPH (GC) - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are either volatized directly from the sample (VOA water and low-soil), from the sample extract (VOA medium soil), or injected as an extracted sample (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture Detector (ECD). In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

HOLD TIME - the maximum allowable elapsed time expressed in hours or days from the time the sample is collected until the time of its pre-treatment or analysis.

INDEPENDENT STANDARD – an externally prepared standard solution composed of analytes from a different source than those used in the standards for the initial calibration.

INDUCTIVELY COUPLED PLASMA (ICP) - a technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

IN-HOUSE - at the laboratories facility.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the instrument.

INJECTION - introduction of the analytical sample into the instrument excitation system for the purpose of measuring concentration of an analyte.

INSTRUMENT CALIBRATION - Series of analytical standards at different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument.

INSTRUMENT DETECTION LIMIT (IDL) - determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

INSTRUMENT CHECK SAMPLE - a solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INSTRUMENT CHECK STANDARD - a multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.

INTERFERENTS - substances which affect the analysis for the element of interest.

INTERNAL STANDARDS - compounds added to analytical and quality control samples at a known concentration prior to analysis. In the methods that require them, internal standards are used as the basis for quantitation of the target compounds.

INSTRUMENT/ANALYTICAL BLANK - a blank designed to determine the level of contamination associated with the analytical instrument.

INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

SECOND SOURCE CALIBRATION VERIFICATION (SCV) STANDARD - a standard prepared from a source other than that used to prepare the quantitation standard, and used to verify the initial calibration curve.

BLANK SPIKE - a control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

LABORATORY RECEIPT DATE - the date on which a sample is received as recorded on the chain of custody.

LINEAR RANGE, LINEAR DYNAMIC RANGE - the concentration range over which the determinative instrument's analytical curve remains linear.

MATRIX - the predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

MATRIX EFFECT - in general, the effect of the particular sample matrix on the constituents with which is contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

MATRIX SPIKE - aliquot of a matrix spiked with known quantities of target compounds and subjected to the entire analytical procedure. Matrix spikes are used to indicate the efficiency of the method on the matrix by measuring the recovery of the spiked analyte.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method relative to the matrix.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards that are carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD OF STANDARD ADDITIONS (MSA) - the addition of 3 increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept and y-intercept are determined by least-square analysis. The analyte concentration is determined by the absolute value of the x-intercept.

Ideally, the spike volume is low relative to the sample volume (approximately 10% of the volume). Standard addition may counteract matrix effects; it will not counteract special effects. Also referred to as Standard Addition.

m/z - Mass to charge ratio, synonymous with "m/e"

NARRATIVE - portion of the data package which includes laboratory, contract, case and sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

PERCENT DIFFERENCE (%D) - to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference).

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105° C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105° C, including water.

PERCENT SOLIDS - the proportion of solid in a soil sample determined by drying an aliquot of the sample at 105° C.

PERFORMANCE EVALUATION MIXTURE - a calibration solution of specific analytes used to evaluate both recovery and percent breakdown as measures of performance.

PERFORMANCE TESTING (PT) SAMPLE - a single blind sample of known composition obtained from an external provider for analysis. Used by clients and regulatory agencies to evaluate laboratory performance.

PREPARATION BLANK (reagent blank, method blank) - an analytical control that contains distilled/deionized water and reagents, which is carried through the entire analytical procedure – digested/distilled/extracted and analyzed. An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.

PRIMARY QUANTITATION ION - a specific ion used to quantitate a target analyte.

PROTOCOL - a compilation of procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control.

PURGE AND TRAP (DEVICE) - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto a gas chromatographic column.

PURGEABLES – non-water soluble volatile organic compounds.

QUALITY CONTROL SAMPLE - a solution obtained from an outside source having known concentration values to be used to verify the calibration.

REAGENT BLANK - a volume of deionized, distilled water containing the same reagent matrix as the calibration standards carried through the entire analytical scheme.

REAGENT WATER - water in which an interferent is not observed at or above the minimum detection limit of the parameters of interest.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - The relative percent difference is based on the mean of two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RTc}{RTis}$$

where,

RTc = Retention time for the target or surrogate compound in continuing calibration.

Rtis = Retention time for the internal standard in calibration standard or in a sample.

RELATIVE STANDARD DEVIATION (RSD) - the variation of a series of results based on the standard deviation and average. Typically used in the evaluation of initial calibration curves.

$$RSD = \frac{SD}{\text{Average RF}}$$

RESOLUTION - the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RESPONSE - or Instrumental Response: a measurement of the output of the detector in which the intensity of the signal is proportionate to the concentration detected.

RESPONSE FACTOR (RF) - a measure of the relative response of an analyte compared to an internal standard. The RF is determined by the following equation:

$$RF = \left(\frac{Ax}{Ais} \times \frac{Cis}{Cx} \right)$$

where:

A = area of the characteristic ion measured

C = concentration

is = internal standard

x = analyte of interest

RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is

dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

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 off 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 off to 11.45.

If the figure following those to be retained is 5, and if there are no known figures 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 off to 11.44, while 11.425 is rounded off 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.

RUN - a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements.

SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE NUMBER - a unique identification number designated for each sample. The Sample Number appears on all laboratory documents which contain information on that sample.

SEMIVOLATILE COMPOUNDS - compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

SENSITIVITY - the slope of the analytical curve, i.e., functional relationship between emission intensity and concentration.

SERIAL DILUTION – a series of dilutions to attain a less concentrated solution.

SOIL - synonymous with soil/sediment or sediment as used herein.

SONICATOR - a device that uses the energy from controlled ultrasound applications to mix, disperse, and dissolve organic materials from a given solid matrix.

SPECTRA - a plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

STORAGE BLANK - a reagent water aliquot stored with samples and analyzed on a weekly basis for VOCs. The storage blank is used to determine the potential for sample contamination occurring during storage.

STOCK SOLUTION - a standard solution prepared from neat materials diluted to derive other standards.

SURROGATES (Surrogate Standard) - for semivolatiles, volatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be present in the sample.

SUSPENDED - those particulates in suspension which are retained by a 0.45 um membrane filter.

TENTATIVELY IDENTIFIED COMPOUNDS (TIC) - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

TOTAL METALS – analytes from the sample which have been digested to complete solvency prior to analysis.

TWELVE-HOUR TIME PERIOD - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the

laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. The injection time of the last analyses in the batch must be made within 12 hours of the injection time of BFB of DFTPP.

VOLATILE COMPOUNDS – non-water soluble compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

WET WEIGHT - the mass of a sample aliquot including moisture (un-dried) that is used for analysis.

WIDE BORE CAPILLARY COLUMN - a gas chromatographic column with an internal diameter (ID) that is greater than 0.32 mm. Columns with lesser diameters are classified as capillary columns.

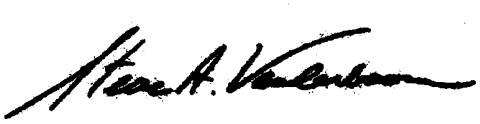
QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control
Policies and Procedures

Pace Analytical Services – Minnesota
1700 Elm Street SE, Suite 200
Minneapolis, MN 55414
612-607-1700

Pace Analytical Services – Montana
602 South 25th Street
Billings, Montana 59101
(406) 254-7226

CORPORATE APPROVAL



May 14, 2010

Steve A. Vanderboom
President, CEO
1700 Elm Street SE, Suite 200
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Date

May 14, 2010

Bruce Warden
Director of Quality, Safety, and Training
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Date

PACE ANALYTICAL SERVICES – MINNESOTA AND MONTANA
LOCAL APPROVAL

This document has been approved as the Quality Assurance Manual, effective 24MAY2010, as indicated by the following signatures:

Laboratory General Manager
612-607-6354
Date: 24May2010

Laboratory Quality Manager
612-607-6352
Date: 24May2010

Laboratory Technical Director
612-607-6381
Date: 5-24-10

Dioxin Technical Director
612-607-6387
Date: 5/24/10

Air Technical Director
612-607-6321
Date: 5/24/10

Table of Contents

1.0 INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"

Pace Analytical Services Inc. - Mission Statement

1.1 Introduction to PASI

Pace Analytical Services, Inc. (PASI) is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. PASI offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, industrial hygiene testing, explosives, high resolution mass spectroscopy (including dioxins, furans and coplanar PCB's), radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. PASI has implemented a consistent Quality System in each of its laboratories and service centers. In addition, the company utilizes an advanced data management system that is highly efficient and allows for flexible data reporting. Together, these systems ensure data reliability and superior on-time performance. This document defines the Quality System and QA/QC protocols.

Our goal is to combine our expertise in laboratory operations with customized solutions to meet the specific needs of our customers.

1.2 Statement of Purpose

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

1.3 Quality Policy Statement and Goals of the Quality System

The PASI management is committed to maintaining the highest possible standard of service for our customers by following a documented quality system. The overall objective of this quality system is to provide reliable data through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

All personnel within the PASI network are required to be familiar with all facets of the quality system and implement these policies and procedures in their daily work. This daily focus on quality is applied with initial project planning, continued through all field and laboratory activities, and is ultimately included in the final report generation.

PASI management demonstrates its commitment to quality by providing the resources, including facilities, equipment and personnel to ensure the adherence to these documented policies and procedures and to promote the continuous improvement of the quality system. All PASI personnel comply with all current applicable state, federal, and industry standards (such as the NELAC, NVLAP and ISO 17025 standards).

1.4 Pace Analytical Services Core Values

- **INTEGRITY**
- **VALUE EMPLOYEES**
- **KNOW OUR CUSTOMERS**
- **HONOR COMMITMENTS**
- **FLEXIBLE RESPONSE TO DEMAND**
- **PURSUE OPPORTUNITIES**
- **CONTINUOUSLY IMPROVE**

1.5 Code of Ethics

PASI's fundamental ethical principles are as follows:

- Each PASI employee is responsible for the propriety and consequences of his or her actions.
- Each PASI employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where PASI does business or seeks to do business.
- Each PASI employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

Strict adherence by each PASI employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of PASI.

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

1.6 Standards of Conduct

1.6.1 Data Integrity

The accuracy and integrity of the analytical results produced at PASI are the cornerstones of the company. Lack of data integrity is an assault on our most basic values and puts PASI and its employees at grave financial and legal risk. Therefore, employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations and databases. Employees are prohibited from making false entries or misrepresentations of data (e.g., dates, calculations, results or conclusions).

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

1.6.2 Confidentiality

PASI employees must not (directly or indirectly) use or disclose confidential or proprietary information except when in connection with their duties at PASI. This is effective over the course of employment and for a period of two years thereafter.

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

1.6.3 Conflict of Interest

PASI employees must avoid situations that might involve a conflict of interest or appear questionable to others. The employee must be careful in two general areas:

- Participation in activities that conflict or appear to conflict with PASI responsibilities.
- Offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced. This includes bribes, kickbacks or illegal payments.

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

1.6.4 Compliance

All employees are required to read, understand and comply with the various components of the standards listed in this document. As confirmation that they understand this responsibility, each employee is required to sign an acknowledgment form (either hardcopy or in electronic database) annually (or as revisions become finalized) that becomes part of the employee's permanent record. Employees will be held accountable for complying with the Quality Systems as summarized in the Quality Assurance Manual.

1.7 Laboratory Organization

The PASI Corporate Office centralizes company-wide accounting, business development, financial management, human resources development, information systems, marketing, quality, safety, and training activities. PASI's Director of Quality is responsible for assisting the development, implementation and monitoring of quality programs for the company. See Attachment IIB for the Corporate Organizational structure.

Each laboratory within the system operates with local management, but all share common systems and receive support from the Corporate Office.

A General Manager (GM) supervises each regional laboratory. Some operations may have an Assistant General Manager (AGM) in situations where the General Manager is responsible for

multiple laboratory facilities and is not necessarily in the facility on a regular basis. Quality Managers (QM) at each lab report directly to their General Manager (or Assistant General Manager) but receive guidance and direction from the Director of Quality.

The General Manager bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of the General Manager (and an Assistant General Manager), the Quality Manager serves as the next in command. He or she assumes the responsibilities of the GM until the GM is available to resume the duties of their position. In the absence of the GM and QM, management responsibility of the laboratory is passed to the Technical Director – provided such a position is identified – and then to the most senior department manager until the return of the GM or QM. The most senior department manager in charge may include the Client Services Manager or the Administrative Business Manager at the discretion of the General Manager.

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

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

Under the direction of the General Manager, the technical staff of the laboratory is generally organized into the following functional groups:

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

Appropriate support groups are present in each laboratory. The actual organizational structure for PASI – Minneapolis and Montana is listed in Attachment IIA. In the event of a change in General Manager, Quality Manager or Technical Director(s), the laboratory will notify its accrediting authorities and revise the organizational chart in the Quality Assurance Manual (QAM) within 30 days. For changes in Department Managers or Supervisors or other laboratory personnel, no notifications will be sent to the laboratory's accrediting agencies; changes to the organizational chart will be updated during or prior to the annual review process. Changes or additions in these key

personnel will also be noted by the additional signatures on the QAM Local Approval page. In any case, the QAM will remain in effect until the next scheduled revision.

1.8 Laboratory Job Descriptions

1.8.1 Senior General Manager

- Oversees all functions of all the operations within their designated region,
- Oversees the development of local General Managers within their designated region,
- Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation,
- Oversees the preparation of budgets and staffing plans for all operations within their designated region, and
- Ensures compliance with all applicable state, federal and industry standards.

1.8.2 General Manager (local lab)

- Oversees all functions of the operations,
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation,
- Prepares budgets and staffing plans,
- Monitors the Quality Systems of the laboratory and advises the Quality Manager accordingly, and
- Ensures compliance with all applicable state, federal and industry standards.

1.8.3 Operations Manager/Laboratory Manager

- In the absence of the GM, performs all duties as listed above for the General Manager,
- Oversees the daily production and quality activities of all departments,
- Manages all departments and works with staff to ensure department objectives are met,
- Works with all departments to ensure capacity and customer expectations are accurately understood and met,
- Works with General Manager to prepare appropriate budget and staffing plans for all departments,
- Responsible for prioritizing personnel and production activities within all departments, and
- Performs formal and informal performance reviews of departmental staff.

1.8.4. Quality Manager

- Oversees the laboratory Quality Systems while functioning independently from laboratory operations. Reports directly to the General Manager,
- Monitors Quality Assurance policies and Quality Control procedures to ensure that the laboratory achieves established standards of quality,

- Maintains records of quality control data and evaluates data quality,
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives,
- Reviews and maintains records of proficiency testing results,
- Maintains the document control system,
- Assists in development and implementation of appropriate training programs,
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements,
- Maintains certifications from federal and state programs,
- Ensures compliance with all applicable state, federal and industry standards, and
- Maintains the laboratory training records, including those in the Learning Management System (LMS).

1.8.5 Technical Director

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

1.8.6 Administrative Business Manager

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

1.8.7 Client Services Manager

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

1.8.8 Project Manager

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

1.8.9 Project Coordinator

- Responsible for preparation of project specifications and provides technical/project support,
- Coordinates project needs with other department sections and assists with proposal preparation,
- Prepares routine proposals and invoicing,
- Responsible for scanning, copying, assembling and binding final reports, and
- Other duties include filing, maintaining forms, process outgoing mail, maintaining training database and data entry.

1.8.10 Department Manager/Supervisor

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

1.8.11 Group Supervisor/Leader

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

1.8.12 Laboratory Analyst

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

1.8.13 Laboratory Technician

- Prepares standards and reagents according to published methods or in house procedures,
- Performs preparation and analytical steps for basic laboratory methods,
- Works under the direction of a Laboratory Analyst on complex methodologies,
- Assists Laboratory Analysts on preparation, analytical or data reduction steps for complex methodologies, and

- Monitors quality control data as required or directed. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

1.8.14 Sample Management Personnel

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

1.8.15 Systems Administrator or Systems Manager

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

1.8.16 Safety/Chemical Hygiene Officer

- Maintains the laboratory Chemical Hygiene Plan.
- Plans and implements safety policies and procedures.
- Maintains safety records.
- Organizes and/or performs safety training.
- Performs safety inspections and provides corrective/preventative actions.
- Assists personnel with safety issues (e.g. personal protective equipment).

1.8.17 Program Director/Hazardous Waste Coordinator

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

1.9 Training and Orientation

Each new employee receives a five part orientation: human resources, ethics and data integrity, safety, Quality Systems, and departmental.

The human resources orientation includes benefits, salary, and company policies. All records are stored with Human Resources.

The ethics and data integrity training covers the obligations of each employee to ensure the defensibility of laboratory data. Employees are provided with general policies related to ethics in the laboratory and specific examples of improper practices that are unacceptable in any PASI facility. The employee is trained to make the right decisions with regards to laboratory practices and where to go for answers in circumstances where they may be unclear as to the correct protocol.

The safety orientation includes an in-depth review of the PASI Chemical Hygiene Plan/Safety Plan, which are consistent with the requirements of OSHA's Hazard Communication Program (29 CFR 1910.1200) and other pertinent regulations.

The Quality Systems orientation provides the new employee with information through an introduction to the Quality Assurance Manual and SOPs, acceptable record keeping practices, and the individual's responsibility to data quality. Quality Systems training is reinforced with the new employee as specific topics are covered during the departmental or analytical method training. Quality Systems training will address policies and practices that ensure the quality and defensibility of the analytical data. These topics include but are not limited to traceability of measurements, method calibration, calibration verification, accuracy, precision and uncertainty of measurements, corrective actions, documentation and root cause analysis.

The new employee's Department Supervisor provides the employee with a basic understanding of the role of the laboratory within the structure of PASI and the basic elements of that individual's position.

Supervised training uses the following techniques:

- Hands-on training
- Training checklists/worksheets (e.g. from LMS new hire workbooks)
- Lectures and training sessions
- Method-specific training
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Proficiency testing programs.
- On-line courses

Group Supervisors/Leaders are responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's training file indicates what procedures an analyst or a technician is capable of performing, either independently or with supervision. The files also include documentation of continuing capability (see Section 3.4 for details on

Demonstration of Capability requirements). Training documentation files for each person are maintained by the Quality Office either in hardcopy format or within the Learning Management System (LMS).

All procedures and training records are maintained and available for review during laboratory audits. These procedures are reviewed/updated periodically by lab management. Additional information can be found in SOP S-ALL-Q-020 ***Training Procedures*** or its equivalent revision or replacement.

1.10 Laboratory Safety

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

1.11 Security and Confidentiality

Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by PASI staff. Keyless door-lock combinations (and computer access codes/logins) are changed periodically. Posted signs direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors to the facility must sign the Visitor's Logbook maintained by the receptionist. A staff member will accompany them during the duration of their stay on the premises unless the GM, QM or TD specify otherwise. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out. The last staff member to leave their department for the day should ensure that all outside access points to that area are secure.

Additional security is provided where necessary, e.g., specific secure areas for sample, data and customer report storage, as requested by customers or cases where national security is of concern. These areas are lockable within the facilities, or are in secure offsite storage. Access is limited to specific individuals or their designees. Security of sample storage areas is the responsibility of the Sample Custodian. Security of samples and data during analysis and data reduction is the responsibility of Group Supervisors. Security of customer report archives is the responsibility of the Client Services Manager. These secure areas are locked whenever these individuals or their designees are not present in the facility.

Access to designated laboratory sample storage locations is limited to authorized personnel only. Provisions for lock and key access are provided. No samples are to be removed without proper authorization. If requested by customer or contract, samples are not to be removed from secure storage areas without filling out the associated internal Chain-of-Custody records.

Standard business practices of confidentiality are applied to all documents and information regarding customer analyses. Specific protocols for handling confidential documents are described in PASI SOPs. Additional protocols for internal identification of samples and data by number only are implemented as required under contract-specific Quality Assurance Project Plans (QAPPs).

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

2.0 SAMPLE CUSTODY

2.1 Sampling Support

Each individual PASI laboratory provides shipping containers, sample containers (including applicable chemical preservatives), custody documents, and field quality control samples (e.g., trip blanks) to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VIII. Note that all analyses listed are not necessarily performed at all PASI laboratories and there may be additional laboratory analyses performed that are not included in these tables. PASI - Minneapolis and Montana may provide pick-up and delivery services to their customers when needed.

2.2 Field Services

Pace Analytical has a large Field Services Division which is based in their Minneapolis facility as well as limited field service capabilities in some of the other facilities. Field Services provides comprehensive nationwide service offerings including:

- Stack Testing
- Ambient Air
- CEM Certification Testing
- Air Quality Monitoring
- Onsite Analytical Services- FTIR and GC
- Real-time Process Diagnostic/Optimization Testing
- Wastewater, Groundwater and Drinking Water Monitoring
- Stormwater and Surface Water Monitoring
- Soil and Waste Sampling
- Mobile Laboratory Services

Field Services operates under the PASI Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services for a unit specific Quality Program. All procedures and methods used by Field Services are documented in Standard Operating Procedures and Procedure Manuals.

2.3 Project Initiation

Prior to accepting new work, the laboratory reviews performance capability. The laboratory establishes that sufficient resources (personnel, equipment capacity, analytical method capability, etc.) are available to complete the required work. The customer needs and data quality objectives are defined and appropriate environmental test methods are assured to meet customer's requirements by project managers or sales representative. Project Managers review laboratory certifications. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or daily operations meetings.

The laboratory maintains records of all such reviews, including discussions with customers. Routine analytical project documentation of quotes, notes, dates, initials and/or recordings is maintained in a project folder by project management. Conditions for new and more complex contracts are determined by the General Managers and sales representatives. Quality Management is consulted on technical requirements and operations staff provides input on volume capacities. Evidence of these reviews is maintained in the form of awarded Request for Proposals (RFPs), signed quotes or contracts, and a Customer Relationship Management (CRM) database. If a review identifies a potential mismatch between customer requirements and laboratory capabilities and/or capacities, Pace will specify its level of commitment by listing these exceptions to the requirements within the RFP, quote or contract.

Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-ALL-C-006 ***Review of Analytical Requests*** or its equivalent revision or replacement.

2.4 Chain-Of-Custody

A chain-of-custody (COC) (see Attachment VII) document provides the legal documentation of samples from time of collection to completion of analysis. Importance is stressed on completeness of COCs. PASI has implemented Standard Operating Procedures to ensure that sample custody traceability and responsibility objectives are achieved for every project.

Field personnel or client representatives complete a chain-of-custody form for all samples. Samples are received by the laboratory accompanied by these forms.

If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

The sampler is responsible for providing the following information on the chain-of-custody form:

- Customer project name
- Project location or number
- Field sample number/identification
- Date and time sampled
- Sample type (matrix)
- Preservative
- Requested analyses
- Sampler signature
- Relinquishing signature
- Date and time relinquished
- Sampler remarks (if applicable)
- Custody Seal Number (if applicable)
- Regulatory Program Designation
- The state where the samples were collected to ensure all applicable state requirements are met
- Turnaround time requested
- Purchase order number

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

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

2.5 Sample Acceptance Policy

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

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

All samples must:

- Have unique customer identification that are clearly marked with durable waterproof labels on the sample containers and that match the chain of custody.
- Have clear documentation on the chain of custody related to the location of the sampling site with the time and date of sample collection.
- Have the sampler's name and signature
- Have the requested analyses clearly marked
- Have clear documentation of any special analysis requirements (data deliverables, etc.);
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be correctly preserved unless method allows for laboratory preservation.
- Be received within holding time. Any samples with hold times that are exceeded will not be processed without prior customer permission.
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval.
- Be received within appropriate temperature ranges - not frozen but ≤6°C (See Note 1), unless program requirements or customer contractual obligations mandate otherwise (see Note 2). The cooler temperature is recorded directly on the COC and the SCUR. Samples that are delivered to the lab immediately after collection are considered acceptable if there is evidence that the chilling process has been started, for example by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data will be appropriately qualified on the final report.

  Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be

read and recorded to ±0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ±0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤6°C limit (i.e. 6.2°C rounded and recorded as 6°C).

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

Note 3: Biological Tissue Samples must be received frozen at ≤0°C.

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

- Presence of custody seals or tapes on the shipping containers
- Sample condition: Intact, broken/leaking
- Sample holding time
- Sample pH when required
- Appropriate containers

Samples for drinking water analysis that are improperly preserved, or are received past holding time, are rejected at the time of receipt, with the exception of VOA samples that are tested for pH at the time of analysis.

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

2.6 Sample Log-in

After sample inspection, all sample information on the chain-of-custody is entered into the Laboratory Information Management System (LIMS).

This permanent record documents receipt of all sample containers including:

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

All samples received are logged into the LIMS system within one working day of receipt. Sample login may be delayed due to customer clarification of analysis needed, corrective actions for sample receipt non-conformance, or other unusual circumstances. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the

laboratory will use 00:00 as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

The Laboratory Information Management System (EPIC Pro) automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of BB-XXXXX-YYY. The BB represents the laboratory identification within Pace's laboratory network. The 5 digit "X" number represents the project number followed by a 3 digit sample number. The project number is a sequential number that is assigned as a new project is created. The sample number corresponds to the number of samples submitted by the client. In addition to the unique sample ID, there is a sample container ID that consists of the sample number, the container type (ex. BP1U), and bottle 1 of Y, where Y represent the total number of containers of that particular type. Together the sample LIMs number and sample container ID number create a unique barcode encryption that can be linked to the sample analysis requested by the client. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the client's field identification; it will be a permanent reference number for all future interactions.

Current region codes are noted below. More may be added without updating this document.

10 = Minnesota	35 = Florida
92 = Asheville and Charlotte	20 = Gulf Coast
60 = Kansas	30 = Pittsburgh
50 = Indianapolis	40 = Green Bay
3038 = Pittsburgh Radiological	17 = Pace Life Sciences
25 = Seattle	

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

Samples with hold times that are near expiration date/time may be sent directly to the laboratory for analysis at the discretion of the Project Manager and/or General Manager.

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

2.7 Sample Storage

2.7.1 Storage Conditions

Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross-contamination (e.g. volatile samples are stored separate from other samples). All sample fractions, extracts, leachates and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method

2.7.2 Temperature Monitoring

Samples are taken to the appropriate storage location (ambient, refrigerator, freezer) immediately after sample receipt and check-in procedures are completed. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

The temperature of each refrigerated storage area is maintained at ≤6°C unless state or program requirements differ. The temperature of each freezer storage area is maintained at < - 10°C unless state or program requirements differ. The temperature of each storage area is monitored and recorded each workday. If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

- The temperature is rechecked after two hours to verify temperature exceedance. Corrective action is initiated if necessary.
- The Quality Manager and/or laboratory management are notified if the problem persists.
- The samples are relocated to a proper environment if the temperature cannot be maintained after corrective actions are implemented.
- The affected customers are notified.
- Documentation is provided on analytical report.

2.7.3 Hazardous Materials

Pure product or potentially heavily contaminated samples may be tagged as "hazardous" or "lab pack" and are stored separately from other samples.

2.7.4 Foreign/Quarantined Soils

Depending on the soil disposal practices of the laboratory, foreign soils and soils from USDA regulated areas are segregated. The USDA requires these samples to be incinerated or sterilized by an approved treatment procedure. Additional information regarding USDA regulations and sample handling can be found in applicable local laboratory SOPs.

Additional information on sample storage can be found in S-MN-C-001 ***Sample Management*** or its equivalent replacement, in S-MN-S-003 ***Waste Handling and Management,*** and in S-MN-Q-253***, Procedure for Handling of USDA Regulated Soils***, or equivalent replacement.

2.8 Sample Protection

PASI laboratory facilities are operated under controlled access to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted.

Samples are removed from storage areas by designated personnel and returned to the storage areas, if necessary, immediately after the required sample quantity has been taken.

Upon customer request, additional and more rigorous chain-of-custody protocols for samples and data can be implemented. For example, some projects may require complete documentation of sample custody within the secure laboratory.

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

2.9 Subcontracting Analytical Services

Every effort is made to perform chemical analyses for PASI customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory (inside or outside the PASI network) becomes necessary, a preliminary verbal communication with an appropriate laboratory is undertaken. Customers are notified in writing of the lab's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations.

Prior to subcontracting samples to a laboratory outside Pace Analytical, the potential sub-contract laboratory will be pre-qualified by verifying that the subcontractor meets the following criteria:

- All certifications required for the proposed subcontract are in effect,
- Sufficient professional liability and other required insurance coverage is in effect, and
- Is not involved in legal action by any federal, state, or local government agency for data integrity issues and has not been convicted in such investigation at any time during the past 5 years.

Additional information can be found in SOP S-All-Q-027 ***Evaluation & Qualification of Vendors*** or its equivalent replacement. The contact and preliminary arrangements are made between the PASI Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract laboratory agreement include:

- Method of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Laboratory certification requirement
- Price per analysis
- Turn-around time requirements

Chain-of-custody forms are generated for samples requiring subcontracting to other laboratories. Sample receiving personnel re-package the samples for shipment, create a transfer chain-of-custody form and record the following information:

- Pace Analytical Laboratory Number
- Matrix
- Requested analysis
- Special instructions (quick turn-around, required detection or reporting limits, unusual information known about the samples or analytical procedure).
- Signature in "Relinquished By"

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

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

Additional information can be found in S-MN-C-004 ***Subcontracting Samples*** or its equivalent revision or replacement.

2.10 Sample Retention and Disposal

Samples (and sample by-products) must be retained by the laboratory for a period of time necessary to protect the integrity of the sample or sample by-product (e.g. method holding time) and to protect the interests of the laboratory and the customer.

Unused portions of samples are retained by each laboratory based on program or customer requirements for sample retention and storage. The sample retention time is a minimum of 45 days from receipt of the samples. Samples requiring storage beyond this time due to special requests or contractual obligations will not be stored under temperature controlled conditions unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

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

Additional information can be found in S-MN-S-003 ***Waste Handling and Management*** and S-MN-C-001 ***Sample Management*** or their equivalent revisions or replacements.

3.0 ANALYTICAL CAPABILITIES

3.1 Analytical Method Sources

PASI laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. The latest valid editions of methodologies are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, A2LA, A-Class, NVLAP and State Agencies. Section 11 (References) is a representative listing of general analytical protocol references. PASI discloses in writing to its customers and regulatory agencies any instances in which modified methods are being used in the analysis of samples.

In the event of a customer-specific need, instrumentation constraint or regulatory requirement, PASI laboratories reserve the right to use valid versions of methods that may not be the most recent edition available.

3.2 Analytical Method Documentation

The primary form of documentation of analytical methods is the Standard Operating Procedure (SOP). SOPs contain pertinent information as to what steps are required by an analyst to successfully perform a procedure. The required contents for the SOPs are specified in the company-wide SOP for Preparation of SOPs (S-ALL-Q-001).

The SOPs may be supplemented by other training materials that further detail how methods are specifically performed. This training material will undergo periodic, documented review along with the other Quality System documentation.

3.3 Analytical Method Validation

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

Additional information can be found in SOP S-MN-Q-252 ***Methods Validation and Modification Studies***, or equivalent replacement.

3.4 Demonstration of Capability (DOC)

Analysts complete an initial demonstration of capability (IDOC) study prior to performing a method or when there is a change in instrument type, personnel or test method (when a defined 'work cell' is in operation, the entire work cell must meet the criteria). The mean recovery and standard deviation of each analyte, taken from 4 replicates of a quality control standard is

calculated and compared to method criteria (if available) or established lab criteria for evaluation of acceptance. Each laboratory maintains copies of all demonstrations of capability and corresponding raw data for future reference and must document the acceptance criteria prior to the analysis of the DOC. Demonstrations of capability are verified on an annual basis.

Alternative demonstration of capability procedures may be used for IDOC for methods that don't lend themselves to the "4 replicate" approach. For methods that only measure precision, the precision of four laboratory duplicate pairs will be assessed. The relative percent differences must be within the method acceptance limits. For procedures like TCLP or SPLP, the analyst will demonstrate making the buffered solution and performing the tumbling process. The trainer or supervisor will sign-off on demonstration of capability of the tumbling process. Additional demonstration of capability options will be specified in the Method Performance section of the applicable method SOP.

For Continuing Demonstrations of Capability, the laboratories may use Performance Testing (PT) samples in lieu of the 4 replicate approach listed above. For methods or procedures that do not lend themselves to the "4 replicate" approach, the demonstration of capability requirements will be specified in Section 13 – Method Performance of the applicable SOP.

Additional information can be found in SOP S-ALL-Q-020 ***Training Procedures*** or its equivalent revision or replacement.

3.5 Regulatory and Method Compliance

PASI understands that expectations of our customers commonly include the assumption that laboratory data will satisfy specific regulatory requirements. Therefore PASI attempts to ascertain, prior to beginning a project, what applicable regulatory jurisdiction, agency, or protocols apply to that project. This information is also required on the Chain-of-Custody submitted with samples.

PASI makes every effort to detect regulatory or project plan inconsistencies, based upon information from the customer, and communicate them immediately to the customer in order to aid in the decision-making process. PASI will not be liable if the customer chooses not to follow PASI recommendations.

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

4.0 QUALITY CONTROL PROCEDURES

4.1 Data Integrity System

The data integrity system at PASI provides assurances to management that a highly ethical approach is being applied to all planning, training and implementation of methods. Data integrity is crucial to the success of our company and Pace Analytical is committed to providing a culture of quality throughout the organization. To accomplish this goal, PASI has implemented a data integrity system that encompasses the following four requirements:

1. A data integrity training program: standardized training is given to each new employee and a yearly refresher is presented to all employees. Key topics within this training include:
   a. Need for honesty in analytical reporting
   b. Process for reporting data integrity issues
   c. Specific examples of unethical behavior and improper practices
   d. Documentation of non-conforming data that is still useful to the data user
   e. Consequences and punishments for unethical behavior
   f. Examples of monitoring devices used by management to review data and systems
2. Signed data integrity documentation for all employees: this includes a written quiz following the Ethics training session and written agreement to abide by the Code of Ethics and Standards of Conduct explained in the employee manual.
3. In-depth, periodic monitoring of data integrity: including peer data review and validation, internal data audits, proficiency testing studies, etc.
4. Documentation of any review or investigation into possible data integrity infractions. This documentation, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be available for review for lab assessors and must be retained for a minimum of five years.

PASI management makes every effort to ensure that personnel are free from any undue pressures that affect the quality of their work including commercial, financial, over-scheduling, and working condition pressures.

Corporate management also provides all PASI facilities a mechanism for confidential reporting of data integrity issues that ensures confidentiality and a receptive environment in which all employees are comfortable discussing items of ethical concern. The anonymous message line is monitored by the Corporate Director of Quality who will ensure that all concerns are evaluated and, where necessary, brought to the attention of executive management and investigated. **The message line voice mail box number is available in the Pace Employee Handbook.**

4.2 Method Blank

A method blank is used to evaluate contamination in the preparation/analysis system. The method blank is processed through all preparation and analytical steps with its associated samples.

A method blank is processed at a minimum frequency of 1 per preparation batch. In the case of a method that has no separate preparation step (e.g. volatiles), a method blank is processed with no more than 20 samples of a specific matrix performed by the same analyst, in the same method, using the same standards or reagents.

The method blank consists of a matrix similar to the associated samples that is known to be free of the analytes of interest. Laboratories will characterize a representative matrix as "clean" if the matrix contains contaminants at less than ½ the laboratory's reporting limit.

Each method blank is evaluated for contamination. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the reporting limit and is greater then 1/10 of the amount of that analyte found in any associated sample. Corrective actions include the re-preparation and re-analysis of all the samples (where possible) along with the full set of required quality control samples. Data qualifiers must be applied to any result reported that is associated with a contaminated method blank.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

For Ohio VAP projects, the lab must minimize the use of qualified data. In the case of method blank contamination, the lab is required to reanalyze the associated samples with an acceptable blank (no reportable contamination) if there is sufficient sample remaining. The lab must make every effort to take the appropriate corrective actions and resolve any anomalies regarding method blanks for Ohio VAP projects.

4.3 Laboratory Control Sample

The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis.

An LCS is processed at a minimum frequency of 1 per preparation batch. In the case of a method that has no separate preparation step (e.g. volatiles), an LCS will be processed with no more than 20 samples of a specific matrix performed by the same analyst, in the same method, using the same standards or reagents.

The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes.

The LCS contains **all** analytes specified by a specific method or by the customer or regulatory agency (which may include full list of target compounds, with certain exceptions. These exceptions may include analyzing only specific Aroclors when PCB analysis is requested or not spiking with all EPA Appendix compounds when a full Appendix list of compounds is requested). In the absence of specified components, the lab will spike with the following compounds:

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

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

The LCS is evaluated against the method default or laboratory-derived acceptance criteria. For those methods that require laboratory-derived limits, method default control limits may be used until the laboratory has a minimum of 20 (preferably greater than 30) data points from which to derive internal criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any associated sample containing an 'out-of-control' compound must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier.

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

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

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

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

For Ohio VAP projects, the lab must minimize the use of qualified data. In the case of LCS failures, the lab is required to reanalyze the associated samples with an acceptable LCS (all applicable recoveries within acceptable limits) if there is sufficient sample remaining. The lab must make every effort to take the appropriate corrective actions and resolve any anomalies regarding LCSs for Ohio VAP projects.

For Department of Defense projects, the lab is not allowed to have any target analytes that exceed its LCS control limits. In the case of LCS failures, the lab is required to reanalyze the associated samples with an acceptable LCS (all applicable recoveries within acceptable limits) if there is sufficient sample remaining. The lab must make every effort to take the appropriate corrective actions and resolve any anomalies regarding LCSs for Department of Defense projects. See applicable method SOPs for further corrective action.

4.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch (see LCS) unless the MS is actually used as the LCS.

A Matrix Spike/Matrix Spike Duplicate (MS/MSD) set is processed at a frequency specified in a particular method or as determined by a specific customer. This frequency will be specified in the applicable method SOP or customer QAPP. In the absence of such requirements, an MS/MSD set is routinely analyzed once per every 20 samples per general matrix (i.e. soil, water, biota, etc.) per method.

The MS and MSD consist of the sample matrix that is then spiked with known concentrations of target analytes. Lab personnel spike customer samples that are specifically designated as MS/MSD samples or, when no designated samples are present in a batch, randomly select samples to spike that have adequate sample volume or weight. Spiked samples are prepared and analyzed in the same manner as the original samples and are selected from different customers if possible.

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

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

A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

For Ohio VAP projects, the lab must minimize the use of qualified data. In the case of MS/MSD failures, the lab is required to reanalyze the associated samples only when the associated LCS also fails acceptance criteria and if there is sufficient sample remaining. When an LCS is acceptable and the MS results are outside of criteria, and no system anomaly is detected, the samples will be reported with appropriate data qualifiers indicating matrix interference. The lab must make every effort to take the appropriate corrective actions and resolve any anomalies regarding LCSs for Ohio VAP projects.

4.5 Surrogates

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

Surrogates are added to each customer sample (for organics), method blank, LCS and MS prior to extraction or analysis. The surrogates are evaluated against the method or laboratory-derived acceptance criteria. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error. An exception to this would be samples that have high surrogate values but no reportable hits for target compounds. These samples would be reported, with a qualifier, because the implied high bias would not affect the final results.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.6 Sample Duplicate

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

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

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

For Ohio VAP projects, the lab must minimize the use of qualified data. In the case of duplicate samples exceeding the RPD criteria found in applicable analytical SOPs, the lab is required to reanalyze the associated sample and duplicate as long as no sampling error was detected (if there is sufficient sample remaining). If the sample and duplicate still do not agree, a comment would be made stating there is a sample anomaly (i.e. non-homogeneous). The lab must make every effort to take the appropriate corrective actions and resolve any anomalies regarding sample duplicates for Ohio VAP projects.

4.7 Internal Standards

Internal Standards are method-specific analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. At a minimum, the laboratory will follow method specific guidelines for the treatment of internal standard recoveries as they are related to the reporting of data.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

For Ohio VAP projects, samples with internal standard failures, outside of method criteria, must be reanalyzed to confirm sample matrix effect. The lab must make every effort to take the

appropriate corrective actions and resolve any anomalies regarding internal standards for Ohio VAP projects.

4.8 Field Blanks

Field blanks are blanks prepared at the sampling site in order to monitor for contamination that may be present in the environment where samples are collected. These field quality control samples are often referenced as field blanks, rinseate blanks, or equipment blanks. The lab analyzes these field blanks as normal samples and informs the customer if there are any target compounds detected above the reporting limits.

4.9 Trip Blanks

Trip blanks are blanks that originate from the laboratory as part of the sampling event and are used to monitor for contamination of samples during transport. These blanks accompany the empty sample containers to the field and then accompany the collected samples back to the lab. These blanks are routinely analyzed for volatile methods where ambient background contamination is likely to occur.

4.10 Limit of Detection (LOD)

PASI laboratories are required to use a documented procedure to determine a limit of detection (LOD) for each analyte of concern in each matrix reported. All sample-processing steps of the preparation and analytical methods are included in this determination. For any test that does not have a valid LOD, sample results below the limit of quantitation (LOQ) cannot be reported.

The LOD is initially established for the compounds of interest for each method in a clean matrix with no target analytes present and no interferences at a concentration that would impact the results. The LOD is then determined every time there is a change in the test method that affects how the test is performed or when there has been a change in the instrument that affects the sensitivity. If required by customer, method or accreditation body, the LOD will be re-established annually for all applicable methods.

Unless otherwise noted, the method used by PASI laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. Where required by regulatory program or customer, the above referenced procedure will be followed.

Where specifically stated in the published method, LODs (or MDLs) will be performed at the listed frequency.

The validity of the LOD must be shown by detection (a value above zero) of the analytes in a QC sample in each quality system matrix. The QC sample must contain the analyte at no more than 3X the LOD for a single analyte test and 4X the LOD for multiple analyte tests. This verification must be performed on each instrument used for sample analysis and reporting of data. The validity of the LOD must be verified as part of the LOD determination process. This verification must be done prior to the use of the LOD for sample analysis.

An LOD study is not required for any analyte for which spiking solutions or quality control samples are not available (e.g. temperature).

The LOD, if required, shall be verified annually for each quality system matrix, technology and analyte. In lieu of performing full LOD (MDL) studies annually, the lab can verify the LOD (MDL) on an annual basis, providing this verification is fully documented and does not contradict other customer or program requirements that the lab must follow. The requirements of this verification are:

- The spike concentration of the verification must be no more than 3X times the LOD for single analyte tests and 4X the LOD for multiple analyte tests.
- The lab must verify the LOD on each instrument used for the reporting of sample data.
- The lab must be able to identify all target analytes in the verification standard (distinguishable from noise).

For Ohio VAP projects, a valid MDL must be in place prior to sample analysis. MDLs must be spiked at or below the reporting limit. The MDL will not be accepted if it was spike higher than the reporting limit.

Additional information can be found in SOP S-ALL-Q-004 ***Method Detection Limit Studies*** or its equivalent revision or replacement.

4.11 Limit of Quantitation (LOQ)

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

There must be a sufficient buffer between the LOD and the limit of quantitation (LOQ). The LOQ must be higher than the LOD.

To verify the LOQ, the laboratory will prepare a sample in the same matrix used for the LCS. The sample will be spiked with target analytes at the concentration(s) equivalent to or less than the RL(s). This sample must undergo the routine sample preparation procedure including any routine sample cleanup steps. The sample is then analyzed and the recovery of each target analyte determined. The recovery for each target analyte must meet the laboratories current control limits.

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

Additional information can be found in SOP S-ALL-Q-004 ***Method Detection Limit Studies*** or its equivalent revision or replacement.

4.12 Estimate of Uncertainty

PASI laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, PASI laboratories base this estimation on the recovery data obtained from the Laboratory Control Spikes. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-MN-Q-255 ***Estimation of Measurement Uncertainty*** or its equivalent revision or replacement.

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

4.13 Proficiency Testing (PT) Studies

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

The lab initiates an investigation whenever PT results are deemed 'unacceptable' by the PT provider. All findings and corrective actions taken are reported to the Quality Manager. A corrective action plan (including re-analysis of similar samples) is initiated and this report is sent to the appropriate state accreditation agencies for their review.

PT samples are treated as typical customer samples, utilizing the same staff, methods, equipment, facilities, and frequency of analysis. PT samples are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

Comparison of analytical results with anyone participating in the same PT study is prohibited prior to the close of the study.

Additional information can be found in SOP S-MN-Q-258 ***PE/PT Program*** or its equivalent revision or replacement.

4.14 Rounding and Significant Figures

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

Rounding

PASI-Minnesota and Montana follows the odd / even guidelines for rounding numbers:

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

Significant Digits

Unless specified by federal, state or local requirements or on specific request by a customer, PASI-Minneapolis and Montana reports all analytical results to 3 significant digits, regardless of the magnitude of the value reported.

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

- Values > 10 – Reported to 3 significant digits
- Values ≤ 10 – Reported to 2 significant digits

5.0 DOCUMENT MANAGEMENT AND CHANGE CONTROL

5.1 Document Management

Additional information can be found in SOP S-ALL-Q-002 ***Document Management*** or its equivalent revision or replacement.

Pace Analytical Services, Inc. has an established procedure for managing documents that are part of the quality system. The list of managed documents includes, but is not limited to, Standard Operating Procedures, Quality Assurance Manuals, quality policy statements, training documents, work-processing documents, charts, posters, memoranda, notices, forms, software, and any other procedures, tables, plans, etc. that have a direct bearing on the quality system.

A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. This establishes that there are no invalid or obsolete documents in use in the facility. All documents are reviewed periodically and revised if necessary. Obsolete documents are systematically discarded or archived for audit or knowledge preservation purposes.

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

SOPs, specifically, are available to all lab staff via the Learning Management System (LMS) which is a secure repository that is accessed through an internet portal. As a local alternative to the hard copy system of controlled documents, secured electronic copies of controlled documents may be maintained on the lab's local server. These document files must be read-only for all personnel except the Quality Department and system administrator. Other requirements for this system are as follows:

- Electronic documents must be readily accessible to all facility employees.
- All hardcopy SOPs must be obtained from the Quality Department.

5.1.1 Quality Assurance Manual (QAM)

The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for PASI. The base QAM template is distributed by the Corporate Quality Department to each of the regional Quality Managers. The regional management personnel modify the necessary and permissible sections of the base template and submit those modifications to the Corporate Director of Quality for review. Once approved and signed by both the CEO and the Director of Quality, the General Manager, Quality Manager and Technical Director(s) sign the Quality Assurance Manual. Each regional Quality Manager is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis by all of the PASI Quality Managers and revised accordingly by the Director of Quality.

5.1.2 Standard Operating Procedures (SOPs)

SOPs fall into two categories: company-wide documents (starting with the prefix S-ALL-) and facility-specific documents (starting with the individual facility prefix).

The purpose of the company-wide SOPs is to establish policies and procedure that are common and applicable to all PASI facilities. Company-wide SOPs are document-controlled by the corporate quality office and signed copies are distributed to all of the regional Quality Managers. The regional management personnel sign the company-wide SOPs. The regional Quality Manager is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies.

Regional PASI facilities are responsible for developing facility-specific SOPs applicable to their respective facility. The regional facility develops these facility-specific SOPs based on the corporate-wide SOP template. This template is written to incorporate a set of minimum method requirements and PASI best practice requirements. The regional facilities may add to or modify the corporate-wide SOP template provided there are no contradictions to the minimum method or best practice requirements. Facility-specific SOPs are controlled by the regional Quality Manager according to the corporate document management policies.

SOPs are reviewed every two years at a minimum (a more frequent review may be required by state or federal agencies or customers). A review of the document does not necessarily constitute a re-issue of a new revision. Documentation of this review and any applicable revisions are made in the last section of each SOP. This provides a historical record of all revisions.

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

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

For Ohio VAP certification, it is required by the Ohio Administrative Code that the lab must seek Ohio VAP review and approval of all SOPs and Quality Manual subsequent modifications prior to implementation.

For DoD approval, SOPs are reviewed annually.

5.1.3 Other Documentation

Additional documents such as Forms and Spreadsheets are controlled through the document management system.

5.2 Document Change Control

Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After revisions are approved, a revision number is assigned and the previous version of the document is officially retired. Copies may be kept for audit or knowledge preservation purposes.

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

5.3 Management of Change

The process for documenting necessary changes within the laboratory network are not typically handled using the corrective or preventive action system as outlined in section 9.0. Management of Change is a proactive approach to dealing with change to minimize the potential negative impact of systematic change in the laboratory and to ensure that each change has a positive desired outcome. This process will primarily be used for the implementation of large scale projects and information system changes as a means to apply consistent systems or procedures within the laboratory network. The request for change is submitted by the initiator and subsequently assigned to an individual or team for development and planning. The final completion of the process culminates in final approval and verification that the procedure was effectively implemented. Additional information can be found in SOP S-MN-Q-257 ***Method of Change*** or its equivalent revision or replacement.

6.0 EQUIPMENT AND MEASUREMENT TRACEABILITY

Each PASI facility is equipped with sufficient instrumentation and support equipment to perform the relevant analytical testing or field procedures performed by each facility. Support equipment includes chemical standards, thermometers, balances, disposable and mechanical pipettes, etc. This section details some of the procedures necessary to maintain traceability and perform proper calibration of instrumentation and support equipment. See Attachment III for a list of equipment currently used at the Minnesota and Montana PASI facilities.

6.1 Standards and Traceability

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

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

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

All prepared standard or reagent containers include the PASI identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials. This ensures traceability back to the standard preparation logbook.

If a second source standard is required to verify an existing calibration or spiking standard, this standard is purchased from a different supplier. If no second source is available, a second standard from a different lot may be purchased from the same supplier if the lot can be demonstrated as prepared independently from other lots.

Additional information concerning standards and reagent traceability can be found in the SOP S-ALL-Q-025 ***Standard and Reagent Preparation and Traceability*** or its equivalent revision or replacement.

6.2 General Analytical Instrument Calibration Procedures

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

Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers (e.g. J flag) or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers (e.g. E flag) or explained in the narrative. Any specific method requirement for number and type of calibration standards supersedes the general requirement. Instrument and method specific calibration criteria are explained within the specific analytical standard operating procedures for each facility.

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

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

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

6.2.1 General Organic Calibration Procedures

Calibration standards are prepared at a minimum of five concentrations for organic analyses. Results from all calibration standards must be included in constructing the calibration curve with the following exceptions:

- The lowest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The reporting limit must be adjusted to the lowest concentration included in the calibration curve.
- The highest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done an individual analyte basis. The upper limit of quantitation must be adjusted to the highest concentration included in the calibration curve.
- Multiple points from either the high end or the low end of the calibration curve may be excluded as long as the remaining points are contiguous in nature and the minimum number of levels remain as established by method or standard operating procedure.

The reporting limit or quantitation range, which is appropriate, must be adjusted accordingly.

- Results from a concentration level between the lowest and highest calibration levels can be excluded from the calibration curve for an acceptable cause with approval from the responsible department supervisor if the results for all analytes are excluded and the point is replaced by re-analysis. Re-analysis must occur within the same 12 hour tune time period for GC/MS methodologies and within 8 hours of the initial analysis for non-GC/MS methodologies. All samples analyzed prior to the re-analyzed calibration curve point must be re-analyzed after the calibration curve is completed.

Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Curves that do not meet the appropriate criteria require corrective action that may include re-running the initial calibration curve. All initial calibrations are verified with a standard obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

The calibration curve is periodically verified by the analysis of a mid-level continuing calibration verification (CCV) standard during the course of sample analysis. Calibration verification is performed at the beginning and end of each analytical batch (except if an internal standard is used only one verification at the beginning of the batch is needed), whenever it is expected that the analytical system may be out of calibration, if the time period for calibration has expired, or for analytical systems that contain a calibration verification requirement. This verification standard must meet acceptance criteria in order for sample analysis to proceed.

In the event that the CCV does not meet the acceptance criteria, a second CCV may be injected as part of the diagnostic evaluation and corrective action investigation. If the second CCV is acceptable, the analytical sequence is continued. If both CCVs fail, the analytical sequence is terminated and corrective action is initiated. Sample analysis cannot begin until after documented corrective action has been completed and two consecutive CCVs have been analyzed (NELAC). (If required by specific state, program, or customer specification, the instrument is re-calibrated after two consecutive CCV failures.) All samples analyzed since the last compliant CCV are re-analyzed for methodologies utilizing external calibration.

When instruments are operating unattended, the autosamplers may be programmed to inject consecutive CCVs as a preventative measure against CCV failure with no corrective action. In this case, both CCVs must be evaluated to determine potential impact to the results. A summary of the decision tree and necessary documentation are listed below:

- If both CCVs meet the acceptance criteria, the analytical sequence is allowed to continue without corrective action. (The 12 hour clock begins with the injection of the second CCV.)
- If the first CCV does not meet the acceptance criteria and the second CCV is acceptable, the analytical sequence is continued and the results are reported.
- If the first CCV meets the acceptance criteria and the second CCV is out of control, the samples preceded by the out of control CCV must be re-analyzed in a compliant analytical sequence.

- If both CCVs are out of control, all samples since the last acceptable CCV must be re-analyzed in a compliant analytical sequence.

Some analytical methods require that samples be bracketed by passing CCVs analyzed both before and after the samples. This is specific to each method but, as a general rule, all external calibration methods require bracketing CCVs. Most internal standard calibrations do not require bracketing CCVs.

Some analytical methods require verification based on a time interval; some methods require a frequency based on an injection interval. The type and frequency of the calibration verifications is dependent on both the analytical method and possibly on the quality program associated with the samples. The type and frequency of calibration verification will be documented in the method specific SOP employed by each laboratory.

For Ohio VAP projects, the lab must minimize the use of qualifed data. In the case of calibration verification standard failures, the lab is required to reanalyze the CCV and the associated samples so as not to report qualified data (sample data may only be reported if the failure produces a high bias and the samples are non-detect). Where possible, the second attempt should be made using the original aliquot of the standard unless there is reason to suspect that the standard is the cause of failure. The lab must make every effort to take the appropriate corrective actions and resolve any anomalies regarding calibration verification standard failures for Ohio VAP projects.

6.2.2 General Inorganic Calibration Procedures

The instrument is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. A calibration blank is also included. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. The number of calibration standards used depends on the specific method criteria or customer project requirements, although normally a minimum of three standards is used.

The ICP and ICP/MS can be standardized with a zero point and a single point calibration if:

- Prior to analysis, the zero point and the single point calibration are analyzed and a linear range is established,
- Zero point and single point calibration standards are analyzed with each batch
- A standard corresponding to the LOQ is analyzed with the batch and meets the established acceptance criteria
- The linearity is verified at the frequency established by the method or manufacturer.

All initial calibrations are verified with a standard obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

During the course of analysis, the calibration curve is periodically verified by the analysis of calibration verification standards. A calibration verification standard is analyzed within

each analytical batch at method/program specific intervals to verify that the initial calibration is still valid. The CCV is also analyzed at the end of the analytical batch.

A calibration blank is also run with each calibration verification standard to verify the cleanliness of the system. All reported results must be bracketed by acceptable CCVs. Instrument and method specific calibration acceptance criteria are explained within the specific analytical standard operating procedures for each facility.

Interference check standards are also analyzed per method requirements and must meet acceptance criteria for metals analyses.

6.3 Support Equipment Calibration Procedures

All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until repaired. The laboratory maintains records to demonstrate the correction factors applied to working thermometers.

Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths are checked in the expected use range with NIST traceable references in order to ensure the equipment meets laboratory specifications.

6.3.1 Analytical Balances

Each analytical balance is checked and (if necessary) calibrated annually by a qualified service technician. The calibration of each balance is checked each day of use with weights traceable to NIST. Calibration weights are ASTM Class 1 (or other class weights that have been calibrated against a NIST standard weight) and are re-certified annually against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the Quality department.

6.3.2 Thermometers

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

Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures. Each thermometer is individually numbered and assigned a correction factor based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

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

6.3.3 pH/Electrometers

The meter is calibrated before use each day, using fresh buffer solutions. See method specific SOPs for additional information..

6.3.4 Spectrophotometers

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

6.3.5 Mechanical Volumetric Dispensing Devices

Mechanical volumetric dispensing devices including bottle top dispensers, pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis. The accuracy of glass microliter syringes is verified and documented prior to use.

Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-MN-Q-264 ***Support Equipment*** or its equivalent revision or replacement.

6.4 Instrument/ Equipment Maintenance

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

The Laboratory Operations Manager and department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems and coordinate instrument repair and maintenance. The analysts have a primary responsibility to perform routine maintenance.

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

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

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

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service

- Current location in the laboratory
- Condition when received (new, used, etc.)
- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

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

When maintenance is performed to repair an instrument problem, depending on the initial problem, demonstration of return to control may be satisfied by the successful analysis of a reagent blank or continuing calibration standard. The entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily.

7.0 CONTROL OF DATA

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

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

7.1 Analytical Results Processing

When analytical, field, or product testing data is generated, it is either recorded in a bound laboratory logbook (e.g. Run log or Instrument log) or copies of computer-generated printouts are appropriately labeled and filed. These logbooks and other laboratory records are kept in accordance with each facility's Standard Operating Procedure for documentation storage and archival. If the lab chooses to minimize or eliminate its paper usage, these records can be kept as electronic records. In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues.

The primary analyst is responsible for initial data reduction and review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting discrepancies in logbooks and as footnotes or narratives, and uploading analytical results into the LIMS.

The primary analyst then compiles the initial data package for verification. This compilation must include sufficient documentation for data review. It may include standard calibrations, chromatograms, manual integration documentation, electronic printouts, chain-of-custody forms, and logbook copies.

Some agencies or customers require different levels of data reporting. For these special levels, the primary analyst may need to compile additional project information, such as initial calibration data or extensive spectral data, before the data package proceeds to the verification step.

7.2 Data Verification

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

Analysts performing the analysis and subsequent data reduction have primary responsibility for quality of the data produced. The primary analyst initiates the data verification process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. Data review checklists, either hardcopy or electronic, are used to document the data review process. The primary analyst is responsible for the initial input of the data into the LIMS.

The completed data package is then sent to a designated qualified reviewer (this cannot be the primary analyst). The following criteria have been established to qualify someone as a data reviewer. To perform secondary data reviewer, the reviewer must:

1. Have a current Demonstration of Capability (DOC) study on file and have an SOP acknowledgement form on file for the method/procedure being reviewed; or, See Note
2. Have a DOC on file for a similar method/technology (i.e. GC/MS) and have an SOP acknowledgment form on file for the method/procedure being reviewed; or, See Note
3. Supervise or manage a Department and have an SOP acknowledgment form on file for the method/procedure being reviewed; or,
4. Have significant background in the department/methods being reviewed through education or experience and have an SOP acknowledgment form on file for the method/procedure being reviewed.

Note: Secondary reviewer status must be approved personally by the Quality Manager or General Manager in the event that this person has no prior experience on the specific method or general technology (i.e. GC/MS).

This reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer also validates the data entered into the LIMS.

Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data (or designating the review of data electronically). The Operations or Project Manager examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution.

7.3 Data Reporting

All data segments pertaining to a particular PASI project number are delivered to the Client Services Department (Project Manager) for assembly into the final report. All points mentioned during technical and QC reviews are included in a case narrative if there is potential for data to be impacted.

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

1. A title which designates the report as “Final Report”, “Laboratory Results”, “Certificate of Results”, etc.
2. Name and address of laboratory (or subcontracted laboratories, if used).
3. Phone number and name of laboratory contact where questions can be referred.
4. A unique number for the report (project number). The pages of the report shall be numbered and a total number of pages shall be indicated (usually in the cover letter).
5. Name and address of customer and name of project (if applicable).

6. Unique identification of samples analyzed (including customer sample numbers).
7. Identification of any sample that did not meet acceptable sampling requirements (from NELAC or other governing agency), such as improper sample containers, holding times missed, sample temperature, etc.
8. Date and time of collection of samples, date of sample receipt by the laboratory, dates of sample preparation and analysis, and times of sample preparation and analysis when the holding time for either is 72 hours or less.
9. Identification of the test methods used.
10. Identification of sampling procedures if sampling was conducted by the laboratory.
11. Deviations from, additions to, or exclusions from the test methods. These can include failed quality control parameters, deviations caused by the matrix of the sample, etc., and can be shown as a case narrative or as defined footnotes to the analytical data.
12. Identification of whether calculations were performed on a dry or wet-weight basis.
13. Reporting limits used.
14. Final results or measurements, supported by appropriate chromatograms, charts, tables, spectra, etc.
15. A signature and title of person accepting responsibility for the content of the report (can be an equivalent electronic identification) and date report was issued.
16. A statement clarifying that the results of the report relate only to the samples tested or to the samples as they were received by the laboratory.
17. If necessary, a statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory.
18. Identification of all test results provided by a subcontracted laboratory or other outside source.
19. Identification of results obtained outside of quantitation levels.

Additional items may be required per Client QAPPs or different state regulations, i.e. Affidavit for Ohio VAP reports.

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

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

The following positions are the only approved signatories for PASI final reports:

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

See SOP S-MN-L-132 ***Data Reduction, Validation and Reporting in the Environmental Laboratory***, or equivalent replacement for additional information.

7.4 Data Security

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

7.5 Data Archiving

All records compiled by PASI are maintained legible and retrievable and stored secured in a suitable environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. These records may include, but are not limited to, customer data reports, calibration and maintenance of equipment, raw data from instrumentation, quality control documents, observations, calculations and logbooks. These records are retained in order to provide for possible historical reconstruction including sampling, receipt, preparation, analysis and personnel involved. NELAP-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the Quality Manager or a designated Data Archivist.

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

In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained by the acquiring entity for a minimum of five years. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

7.6 Data Disposal

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

8.0 QUALITY SYSTEM AUDITS AND REVIEWS

8.1 Internal Audits

8.1.1 Responsibilities

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

8.1.2 Scope and Frequency of Internal Audits

The complete internal audit process consists of the following four sections:

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

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

Examples of system-wide elements that can be audited include:

- Quality Systems documents, such as Standard Operating Procedures, training documents, Quality Assurance Manual and all applicable addenda
- Personnel and training files.
- General laboratory safety protocols.
- Chemical handling practices, such as labeling of reagents, solutions, standards, and associated documentation.
- Documentation concerning equipment and instrumentation, calibration/maintenance records, operating manuals.
- Sample receipt and management practices.
- Analytical documentation, including any discrepancies and corrective actions.
- General procedures for data security, review, documentation, reporting and archiving.
- Data integrity issues such as proper manual integrations.

When the operations of a specific department are evaluated, a number of additional functions are reviewed including:

- Detection limit studies
- Internal chain-of-custody documentation
- Documentation of standard preparations
- Quality Control limits and Control charts

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

A representative number of data audits are completed annually. The report format of any discrepancy is similar to that of other internal audits.

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

8.1.3 Internal Audit Reports and Corrective Action Plans

Additional information can be found in SOP S-ALL-Q-011 ***Audits and Inspections*** or its equivalent revision or replacement.

A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the Quality Manager writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

When audit findings cast doubt on the effectiveness of the operations or on the correctness of validity of the laboratory's environmental test results, the laboratory will take timely corrective action and notify the customer in writing within 3 business days, if investigations show that the laboratory results may have been affected.

Once completed, the internal audit report is issued jointly to the Laboratory General Manager and the manager(s)/supervisor(s) of the audited operation at a minimum. The responsible manager(s)/supervisor(s) responds within 14 days with a proposed plan to correct all of the deficiencies cited in the audit report. The Quality Manager may grant additional time for responses to large or complex deficiencies (not to exceed 30 days). Each response must include timetables for completion of all proposed corrective actions.

The Quality Manager reviews the audit responses. If the response is accepted, the Quality Manager uses the action plan and timetable as a guideline for verifying completion of the

corrective action(s). If the Quality Manager determines that the audit response does not adequately address the correction of cited deficiencies, the response will be returned for modification.

To complete the audit process, the Quality Manager performs a re-examination of the areas where deficiencies were found to verify that all proposed corrective actions have been implemented. An audit deficiency is considered closed once implementation of the necessary corrective action has been audited and verified. This is usually within 60-90 days after implementation. If corrective action cannot be verified, the associated deficiency remains open until that action is completed.

8.2 External Audits

PASI laboratories are audited regularly by regulatory agencies to maintain laboratory certifications, and by customers to maintain appropriate specific protocols.

Audit teams external to the company review the laboratory to assess the existence of systems and degree of technical expertise. The Quality Manager and other QA staff host the audit team and assist in facilitation of the audit process. Generally, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. In some cases, items of concern are discussed during a debriefing convened at the end of the on-site review process.

The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the Quality Manager. The Laboratory General Manager provides the necessary resources for staff to develop and implement the corrective action plans. The Quality Manager collates this information and provides a written report to the audit team. The report contains the corrective action plan and expected completion dates for each element of the plan. The Quality Manager follows-up with the laboratory staff to ensure corrective actions are implemented.

8.3 Quarterly Quality Reports

The Quality Manager is responsible for preparing a quarterly report to management summarizing the effectiveness of the laboratory Quality Systems. This status report will include:

- Overview of quality activities for the quarter
- Certification status
- Proficiency Testing study results
- SOP revision activities
- Company-wide 3P Document implementation (internal program)
- External audit findings
- Internal audit (method/system) findings
- Manual integration audit findings (Mintminer)
- Raw Data and Final Report review findings
- MDL activities
- Corrective action activities
- Training activity status

- Other significant Quality System items

The Corporate Director of Quality utilizes the information from each laboratory to make decisions impacting the Quality Systems of the company as a whole. Each General Manager utilizes the quarterly report information to make decisions impacting Quality Systems and operational systems at a local level.

Additional information can be found in SOP S-ALL-Q-014 ***Quality System Review*** or its equivalent revision or replacement.

8.4 Annual Managerial Review

A managerial review of Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements.

The managerial review must include the following topics of discussion:

- Policy and procedure suitability
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventative actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources and staffing.

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

8.5 Customer Service Reviews

As part of the annual managerial review listed previously, the sales staff is responsible for reporting on customer feedback, including complaints. The acquisition of this information is completed by performing surveys.

The sales staff continually receives customer feedback, both positive and negative, and reports this feedback to the lab management in order for them to evaluate and improve their management system, testing activities and customer service.

In addition, the labs must be willing to cooperate with customers or their representatives to clarify customer requests and to monitor the lab's performance in relation to the work being performed for the customers.

9.0 CORRECTIVE ACTION

Additional information can be found in SOP S-MN-Q-262 ***Corrective Action/Preventive Action Process*** or its equivalent revision or replacement.

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

9.1 Corrective Action Documentation

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

- Internal Laboratory Non-Conformance Trends
- PE/PT Sample Results
- Internal and External Audits
- Data Review
- Client Complaints
- Client Inquiries
- Holding Time violations

Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g. matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

The person who discovers the deficiency or non-conformance initiates the corrective action documentation on the Non-Conformance Corrective/ Preventative Action report and/or LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

In the event that the laboratory is unable to determine the cause, laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance problem, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance problem. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within Lab Track or on the Corrective/Preventative Action Report.

After all the documentation is completed, the routing of the Corrective/Preventative Action Report and /or Lab Track will continue from the person initiating the corrective action, to their immediate supervisor or the Project Manager and finally to the Quality Manager, who is responsible for final review and signoff of all formal corrective/preventative actions.

9.2 Corrective Action Completion

9.2.1 Internal Laboratory Non-Conformance Trends

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

- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Lab accident
- Spike Failure
- Instrument Failure
- Final Reporting error

9.2.2 PE/PT Sample Results

Any PT result returned to the Quality Manager as “not acceptable” requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The Quality Manager reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the Quality Manager and reported to the applicable regulatory authorities.

9.2.3 Internal and External Audits

The Quality Manager is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for reporting back to the auditing body, the root cause of the issue, and the corrective action taken to resolve the findings. The Quality Manager is also responsible for providing any back-up documentation used to prove that a corrective action has been completed.

9.2.4 Data Review

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

9.2.5 Client Complaints

Project Managers are responsible for issuing corrective action forms for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor. After the corrective actions have been listed, the Project Manager reviews the corrective action to determine if the customer needs or concerns are being addressed.

9.2.6 Client Inquiries

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

9.2.7 Holding Time Violations

In the event that a holding time requirement has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the Quality Manager must be made aware of these holding time violations.

The Project Manager must contact the customer for appropriate decisions to be made with the resolution documented and included in the customer project file. The Quality Manager includes a list of all missed holding times in their Quarterly Report to the corporate office.

9.2.8 Sample Acceptance Policy deviations

Any deviation form the Sample Acceptance Policy listed in this Manual must be documented on the Chain-of-Custody or other applicable form by the sample receiving personnel by the Project Manager. Analysts or supervisors that discover such deviations must contact the sample receiving personnel or appropriate Project Manager so they can initiate the proper documentation and customer contact. If a more formalized corrective action must be documented, the Quality Manager is made aware of the situation.

The customer is notified of these deviations as soon as possible so they can make decisions on whether to continue with the sample analysis or re-sample. Copies of this documentation are included in the project file.

9.2.9 Quality Control outside of acceptance criteria

The analyst that is generating or validating Analytical data is responsible for checking the results against established acceptance criteria (quality control limits). The analyst must immediately address any deficiencies discovered. Method blank, LCS or matrix spike failures are evaluated against method, program, and customer requirements and appropriate footnotes are entered into the LIMS system. Some deficiencies may be caused by matrix interferences. Where possible, matrix interferences are confirmed by re-analysis.

Quality control deficiencies must be made known to the customer on the final report for their review of the data for usability. If appropriate, the supervisor is alerted to the QC failure and if necessary a formal corrective action can be initiated. This may involve the input of the Quality Manager or the General Manager.

The department supervisor and/or Operations Manager are responsible for evaluating the source of the deficiency and for returning the analytical system to control. This may involve instrument maintenance, analytical standard or reagent evaluation, or an internal audit of the analytical procedure.

See applicable analytical SOPs for further guidance on QC acceptance criteria.

9.3. Preventive Action Documentation

Pace laboratories can take advantage of several available information sources in order to identify needed improvements in all of their systems (technical, managerial, quality, etc.). These sources may include:

- Management Continuous Improvement Plan (CIP) metrics which are used by all production departments within Pace. When groups compare performance across the company, ways to improve systems are discovered. These improvements can be made within a department or lab-wide.
- Annual managerial reviews- part of this NELAC-required and NVLAP-required review is to look at all processes and procedures used by the lab over the past year and to determine ways to improve these processes in the future.
- Quality systems reviews- any frequent checks of quality systems (monthly logbook reviews, etc.) can uncover issues that can be corrected or adjusted before they become a larger issue.

When improvement opportunities are identified or if preventive action is required, the lab can develop, implement, and monitor preventive action plans.

10.0 GLOSSARY

3P Program	The Pace Analytical continuous improvement program that focuses on Process, Productivity and Performance. Best Practices are identified that can be used by all PASI labs.
Accuracy	The agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Aliquot	A portion of a sample taken for analysis.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analyte	The specific chemical species or parameter an analysis seeks to determine.
Analytical Uncertainty	A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Audit	A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
Bias	The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Blind Sample	A sample for submitted for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test analyst or laboratory proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Calibration	To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard must bracket the range of planned or expected sample measurements.
Calibration Curve	The graphic representation of known values, such as concentrations for a series of calibration standards and their instrument response.
Calibration Verification	The process of verifying a calibration by analysis of standards and comparing the results with the known amount.
Chain-of-Custody (COC)	A record that documents the possession of samples from the time of collection to receipt in the laboratory. This record generally includes the number and type of containers, mode of collection, collector, time of collection, preservation, and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Confirmation	Verification of the identity of a component through the use of an alternate scientific approach from the original method. These may include, but are not limited to: • second-column confirmation • alternate wavelength • derivatization derivative • mass spectral interpretation • additional cleanup procedures
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification (CCV)	Also referred to as a CVS in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.

Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Corrective Action	The action taken to eliminate the causes of a non-conformity, defect, or other undesirable situation in order to prevent recurrence.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more usable form.
Demonstration of Capability	A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Document Control (Management)	Procedures to ensure that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled (managed) to ensure use of the correct version at the location where the prescribed activity is performed.
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate or Replicate Analysis	The identically performed measurement on two or more sub-samples of the same sample within a short interval of time
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g. PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.

Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Finding	An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.

High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	The maximum time that samples may be held prior to preparation and/or analysis as defined by the method.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP-AES that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Verification (ICV)	A standard (usually from a second source or otherwise required vendor) analyzed after the initial calibration curve to verify that the curve is valid.
Internal Standards	A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Laboratory Control Sample (LCS)	(however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
LabTrack	Database used by Pace Analytical to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A training database used by Pace Analytical to train their employees. This system is a self-paced system which is capable of tracking all employee training requirements and documentation.

Legal Chain-of-Custody	Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit of Detection (LOD)	A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. An LOD is analyte and matrix specific and may be lab-dependent.
Limit of Quantitation (LOQ)	The minimum levels, concentrations or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each lab's learn centers are maintained by a local administrator.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.
Matrix Duplicate	A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery of each analyte.
Method	A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis) systematically presented in the order in which they are to be executed.
Method Blank	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures: and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	One way to establish a Limit of Detection (LOD); 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.
MintMiner	Program used by Pace Analytical to review large amounts of chromatographic data to monitor for errors or data integrity issues.

National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the lab's ability to reproduce those conditions.
Precision	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	Any conditions under which a sample must be kept in order to maintain the chemical and/or biological integrity of the sample.
Proficiency Testing	A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Proficiency Testing Sample	A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Protocol	A detailed written procedure for field and/or laboratory operation that must be strictly followed.
Quality Assurance (QA)	An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures required by a specific project.

Quality Control (QC)	The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality System	A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.
Quality System Matrix	These matrix definitions are to be used for purposes of batch and quality control requirements: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin • **Chemical Waste**: A product or by-product or an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other saltwater source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, printouts of chromatograms, instrument outputs, and handwritten records.

Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Reference Standard	Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e. statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL.
Reporting Limit Verification Standard (or otherwise named)	A standard analyzed at the reporting limit for an analysis to verify the lab's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Sample Condition Upon Receipt Form (SCURF)	Form used by Pace Analytical sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain-of-Custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selective Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector.
Sensitivity	The capability of a method or instrument to discriminate between measurement responses representing different levels (concentrations) of a variable of interest.
Standard	A substance or material with properties known with sufficient accuracy to permit its use to evaluate the same property in a sample.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Operating Procedure (SOP)	A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks

Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Surrogate	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty Measurement	The parameter associated with the result of a measurement that characterized the dispersion of the values that could be reasonably attributed to the measurand (i.e. the concentration of an analyte).
Verification	Confirmation by examination and objective evidence that specified requirements have been met.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

11.0 REFERENCES

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- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.
- "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).
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987
- Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C
- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.
- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.
- Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.
- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, 1988
- National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards. Most recent

- ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.

12.0 REVISIONS

The PASI Corporate Quality and Safety Manager files both a paper copy and electronic version of a Microsoft Word document with tracked changes detailing all revisions made to the previous version of the Quality Assurance Manual. This document is available upon request. All revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual Revision 12.0	General: replaced the word 'client' with 'customer', where applicable. Section 1.6.4: added language for clarity Added new section 1.8.1; responsibilities of Senior General Managers. Section 1.8.3: added reference to LMS. Added new section 1.8.17: responsibilities of Waste Coordinators. Section 1.9, last paragraph: changed 'annually' to 'periodically'. Next to last paragraph- added reference to LMS. Added new section 2.2 entitled Field Services. Section 2.3: added reference to the new Review of Analytical Requests SOP. Section 2.7.2: changed freezer temp requirement to match SOP. Section 4.10: revised and added language regarding LOD studies, initial verification and annual verification, where applicable. Section 4.11: changed PRL to RL. Section 4.13: added editable line regarding PT study information. Section 4.14: added sentence regarding rounding rules listed applying only to LIMS. Section 5.1, last bullet point: changed language to reflect that SOPs must be locked from printing if controlled electronically. Section 6.3.1: adjusted language about classes of weights potentially used. Section 6.3.3: removed customer-specific requirement to re-calibrate every four hours but added space for this to be added back in where applicable. Added reference to Attachment III in the introductory paragraph to section 6. Sections 7.1-7.3: added language for those labs that are minimizing or eliminating the need for paper copies. Section 7.2: clarified language in numbered items so that it does not appear that all 4 criteria must be applicable at one time. Section 7.3: added list of approved signatories for final reports. Section 8.1.2, last paragraph: revised language regarding data integrity issues and added a timeframe to notify customers of affected data. Added section 8.5 "Customer Service Reviews"- ISO requirement Added new section 9.3 regarding Preventive Action. Attachment IIb: updated corporate org chart Attachment VIII: revised to match the current Analytical Guides.	13Nov2008
Quality Assurance Manual revision 13.0	Increased font size of entire document. Section 1.7, fifth paragraph: changed length of time Technical Director can be gone before contacting primary authority (from 65 down to 35 days per TNI standard). Section 1.8.2: Reworded definition for Assistant GM to say "all departments". Fixed numbering issue with sub-sections for section 1.8 and used bullet points instead of numbers. Section 1.8.19: revised position title to capture requirement of some labs. Section 1.9: added language to second bullet point regarding LMS. Section 1.9: added bullet point for on-line courses. Section 2.5: added third note per request from GB (in red text). Section 2.6: Added chart of 2-digit codes (lab designations) per audit finding from GB lab (matches corporate SOPs). Section 2.7.4: added reference for Waste Handling and Management SOT. Section 3.1: added more method agency references. Section 3.4: added reference to Training SOP at end of section. Section 4.1: fixed numbering issue. Removed anonymous phone number	30Apr2010

Document Number	Reason for Change	Date
	and added reference to the Employee Handbook. Section 4.2: added paragraph of Ohio VAP required language (red text). Section 4.3, fifth paragraph: reworded second sentence for clarity. Section 4.3: added paragraphs of Ohio VAP and DoD required language (red text). Section 4.4, first paragraph: added qualifier to end of paragraph that MS limits are used to assess the batch if the MS is used in place of the LCS. Section 4.4: added paragraph of Ohio VAP required language (red text). Section 4.6: added paragraph of Ohio VAP required language (red text). Section 4.7: added paragraph of Ohio VAP required language (red text). Section 4.10: added paragraph of Ohio VAP required language (red text). Section 4.11: added paragraph of DoD required language (red text). Section 5.1, fifth paragraph: changed wording from LAN/WAN to local server (as opposed to hardcopies) and added language about LMS access. Section 5.1.2: added paragraphs of Ohio VAP and DoD required language (red text). Added new section 5.3- Management of Change. Section 6.2.1: added paragraph of Ohio VAP required language (red text). Section 6.3.2: changed NIST thermometer calibration frequency to every 3 years to match current practice. Section 7.3: added comment about Ohio VAP reporting (red text). Section 8.1.2, last sentence: reworded to match current practice. Section 8.1.3, last paragraph: reworded sentences regarding verification of corrective actions. Section 8.3: revised list of Quarterly Quality report items to match the revised SOP. Section 8.4: added last two bullet points and added second line of last paragraph to match ISO language. Section 9.1: changed bullet point items to match CAR SOP. Section 9.2.1: revised language to match SOP. Section 9.2.2: moved language from old 9.2.8 to 9.2.2. Section 9.2.4: added language to data review section. Glossary: Added definitions for analytical uncertainty, audit, bias, field of accreditation, finding, legal COC, matrix duplicate, method, PT sample, sampling, verification (per TNI standard). Glossary: Added definitions for reporting limit verification standard and initial calibration verification per request. Glossary: revised the following definitions to match new TNI language: DOC, LCS, LOD, MS, MSD, preservation, QA, QC, QC sample, raw data, reference standard, SOPs, and traceability. Also revised language within the definition for Quality System Matrix (previously just called Matrix). Glossary: deleted definition for 'detection limit'. Glossary: added definitions from company Acronym form from IT. Glossary: added definitions for LabTrack and MintMiner. Attachment VIII: added more tests to the chart per QM input including a line item for concentrated waste matrix for VOA 8260. General: changed all references to "Director of Quality, Safety, and Training" to "Director of Quality". General: revised document references to SOTs for Waste Handling and Management and Sample Management. Removed corporate org chart from Attachment IIB and will provide this as a separate document to the QMs. In this way, revisions to the corporate org chart will not necessitate a new QAM revision.	

ATTACHMENT I

Quality Control Calculations

PERCENT RECOVERY (%REC)

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

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

PERCENT DIFFERENCE (%D)

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

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

PERCENT DRIFT

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

RELATIVE PERCENT DIFFERENCE (RPD)

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

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

CORRELATION COEFFICIENT (R)

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

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

ATTACHMENT I (CONTINUED)

Quality Control Calculations (continued)

STANDARD DEVIATION (S)

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

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

AVERAGE ($\overline{X}$)

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

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

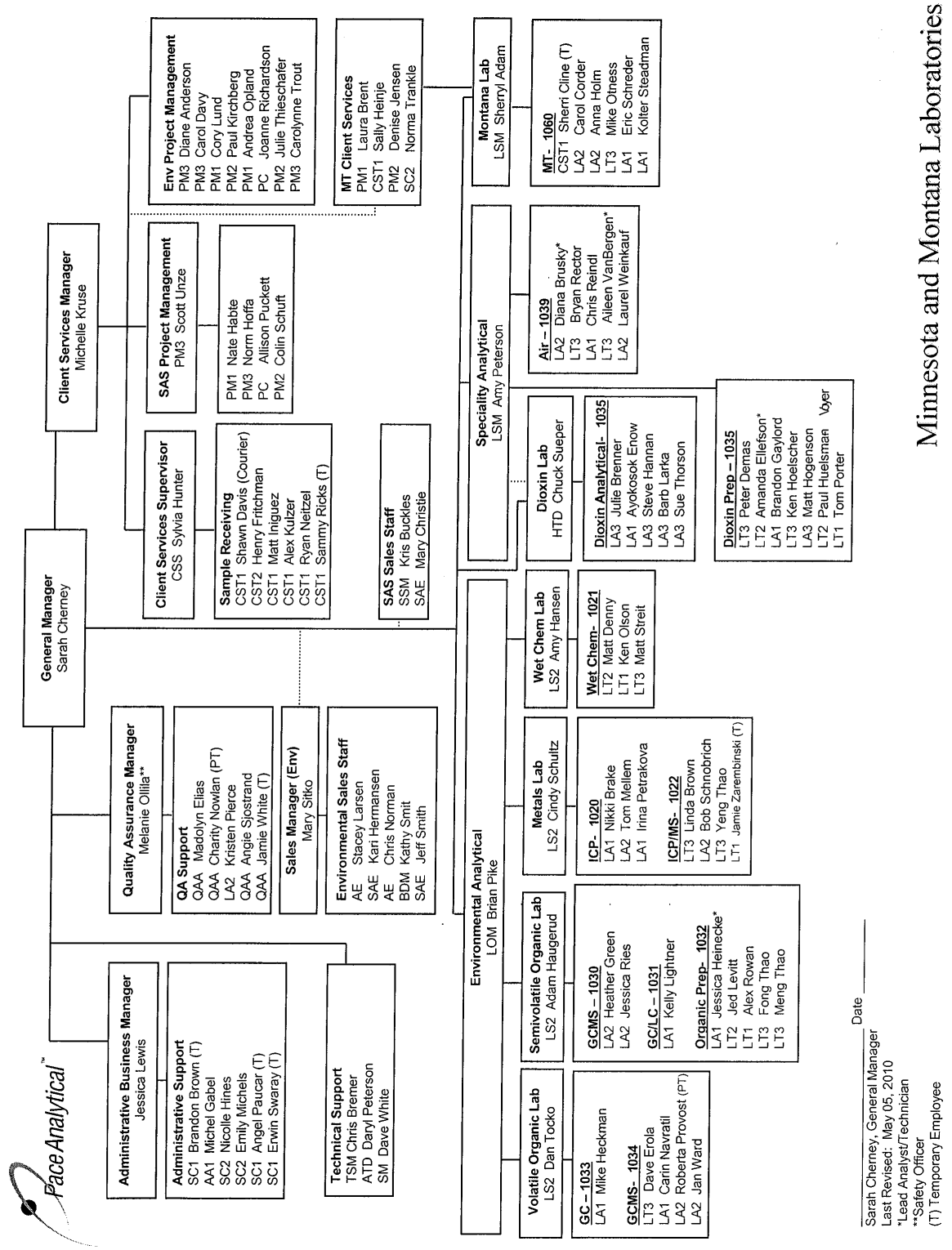
RELATIVE STANDARD DEVIATION (RSD)

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

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

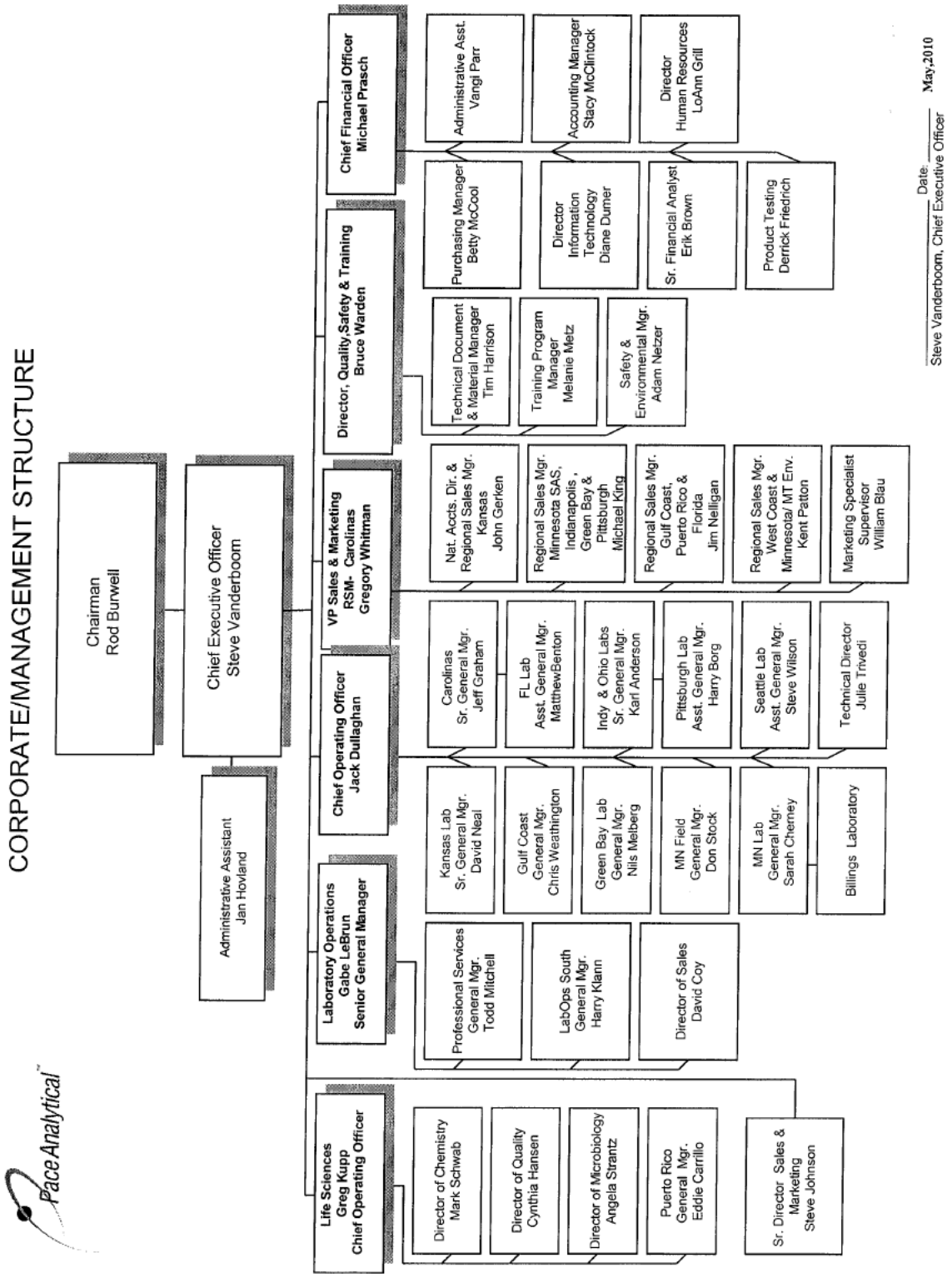
ATTACHMENT IIA

PASI – MINNESOTA AND MONTANA



ATTACHMENT IIB

PASI – CORPORATE ORGANIZATIONAL CHART



ATTACHMENT III
EQUIPMENT LIST

INSTRUMENT	MANUFACTURER	MODEL NUMBER	DETECTOR(S)	ANALYSIS
Acetone Vaporizer	Energy Technology	NA	NA	
Agilent Tray	Agilent	N 10149	MS	CPAH, PCP
Autoanalyzer Autosampler	Astoria Pacific	311	NA	N+N, NH3, TKN
Autoanalyzer Detector	Astoria Pacific	305A	Wavelength	N+N, NH3, TKN
Autoanalyzer Heater Unit	Astoria Pacific	303A	NA	N+N, NH3, TKN
Autoanalyzer Photometer	Astoria Pacific	350	NA	N+N, NH3, TKN
Autoanalyzer Power Supply	Astoria Pacific	304A	NA	N+N, NH3, TKN
AutoClave	Harvey	NA	NA	General - Wet Chem
AutoSampler	Agilent	7683	GC	CALDRO, WIDRO
AutoSampler	Varian Archon	NA	NA	8021/8015/GRO
AutoSampler	Environmental Sample Tech, Inc.	NA	NA	8021/8015/GRO
AutoSampler	Varian Archon		NA	8021/8015/GRO
AutoSampler	Environmental Sample Tech, Inc.	NA	NA	UST, BTEX
AutoSampler	O-I-Analytical	4552	NA	8260/624/TCLP/UST
AutoSampler	O-I-Analytical	4552	NA	8260 Med. Lvl Soil
AutoSampler	Varian Archon	NA	NA	8260/624/TCLP/UST
AutoSampler	Varian Archon	NA	NA	524/8260/624
AutoSampler	Environmental Sample Tech, Inc.	NA	NA	SIM/8260/624/Low & Med Lvl Soil/TCLP/UST
Autosampler	Metrohm	778 Sample Processor	NA	Alkalinity
Autosampler	Hewlett-Packard	7673	NA	EPH
Autosampler	Hewlett-Packard	7673	NA	EPH
Autosampler	Varion	Archon	NA	VPH
AutoSampler /Tower	HP	7673A	GC	AK, NWTPH
Autosampler power supply	Perstorp	509	NA	N+N, NH3, TKN
Autosampler pump	Perstorp	502	NA	N+N, NH3, TKN
AutoSampler Tower	HP	7673	GC	CALDRO, WIDRO
AutoSampler Tower	Agilent	7683	MS	Sulfolane, 8270, 625
AutoSampler Tower	Agilent	18593B	MS	8270, 625
AutoSampler Tray	HP	18596B	GC	CALDRO, WIDRO
AutoSampler Tray	Agilent	G2614A	GC	PCB, TO4
AutoSampler Tray	Agilent	7683	MS	Sulfolane, 8270, 625
AutoSampler Tray	Agilent	18596C	MS	8270, 625
Autotitrator	Metrohm	888 Titrando Titrator		Alkalinity
Balance	Ohaus	1500 D	NA	General - Metals
Balance	Fischer	7227DA	NA	General - Montana
Balance	AND	FX-3200	NA	General - VOA
Balance	AND	FX 3200	NA	General - Metals
Balance	AND	FX-2000	NA	General - O-prep
Balance	Sartorius	AC 210 S	NA	General - Wet Chem
Balance	Denver Inst	MXX-212	NA	General - VOA
Balance	Denver Inst	MXX-5001	NA	General - DRMS Prep
Balance	Sartorius	AC 210 S	NA	General - Wet Chem
Balance	Sartorius	BP 110 S	NA	General - Metals
Balance	Mettler-Toledo	AB135-S	NA	General - Wet Chem
Balance	Ohaus	Scout Pro	NA	General - Wet Chem
Balance	Denver Inst	APX-3202	NA	General - DRMS Prep
Balance	Fischer	A200DS	NA	General - Montana
Balance	Mettler	AE100	NA	General - Montana
Balance	Ohaus	Adventuer	NA	General - Montana
Balance	Mettler	AE 200	NA	General - SVOA
Canister Autosampler	Entech Instruments, Inc.	7020 CA	NA	TO-15
Canister Autosampler	Entech Instruments, Inc.	7021 CA	NA	TO-15
Canister Autosampler	Entech Instruments, Inc.	7016 CA	NA	TO-15
Canister Autosampler	Entech Instruments, Inc.	7017 CA	NA	TO-15
Canister Autosampler	Entech Instruments, Inc.	7018 CA	NA	TO-15
Canister Autosampler	Entech Instruments, Inc.	7019 CA	NA	TO-15
Centrifuge	Fischer	Centific	NA	General - Montana
Centrifuge	IEC	Centra GP8	NA	General - O-prep
Centrifuge	Damon/IEC Division	NA	NA	General - O-prep
Centrifuge	International Clinical Centrifuge	CL28899M	NA	General - O-prep

ATTACHMENT III – CONT'D

Closed Cup - Penske	Precision Scientific	NA	NA	Flashpoint
COD Reactor	Bioscience, Inc.	NA	NA	COD
Colony Counter	Gallenkamp	Colony Counter	NA	HPC
Colony Counter	Darkfield Quebec	Colony Counter	NA	HPC
Concentrator	Entech Instruments, Inc.	7100A	GC/MS	TO-15
Concentrator	Entech Instruments, Inc.	7100A	GC/MS	TO-15
Concentrator	Entech Instruments, Inc.	7100A	GC	TO3 BTEX
Concentrator	Entech Concentrator	7100A	GC/MS	TO-15
Concentrator	Tekmar Dohrmann	3100	NA	8021/8015/GRO
Concentrator	Tekmar Dohrmann	3100	NA	8021/8015/GRO
Concentrator	Tekmar	3000	NA	8021/8015/GRO
Concentrator	Tekmar	3000	MS	UST, BTEX
Concentrator	Tekmar	3100	MS	8260/624/TCLP/UST
Concentrator	Tekmar	3100	MS	8260 Med. Lvl Soil
Concentrator	Tekmar Dohrmann	3100	MS	8260/624/TCLP/UST
Concentrator	Tekmar	3000	MS	524/8260/624
Concentrator	Tekmar	3000	GC	SIM/8260/624/Low & Med Lvl Soil/TCLP/UST
Concentrator	Zymark	TurboVap II	NA	EPH
Concentrator	Zymark	TurboVap II	NA	EPH
Concentrator	Zymark	Turbo Vap II	NA	
Conductivity meter	Oaktom	Con 110 Series	NA	Specific Conductivity
Digestion Block	Thomas Cain Inc.	Deena 60	NA	
Digestion Block	Environmental Express	NA	NA	
Digestion Block	MIDI-STIL	NA	NA	
Diss. Oxy Meter	YSI	5000	NA	BOD
Evaporator	Organomation	N-Evap 112	NA	Fractions
Explandable Ion Analyzer	Orion	EA 940	Ions	Chlorides
Extractor	Horizon Technology	Spe-dex 4790	NA	Oil & Grease
Extractor	Horizon Technology	Spe-dex 4791	NA	Oil & Grease
Extractor	Horizon Technology	Spe-dex 4792	NA	Oil & Grease
Extractor	Horizon Technology	Spe-dex 4793	NA	Oil & Grease
Furnace	Sybron Thermolyne	1300	NA	General - Montana
Furnace	Leco	S-144DR	NA	General - Montana
GC	Agilent Technologies	6890N	GC/MS	TO-15
GC	HP	5890	TCD	3C
GC	Agilent Technologies		GC/MS	TO-15
GC	Agilent Technologies	G1530A	GC/FID/TCD	RSK 175
GC	Agilent Technologies	6890	GC/MS	TO-15
GC	Agilent	6890N	GC	PCB, TO4
GC	HP	6890	MS	CPAH, PCP
GC Oven	HP	5890	GC	AK, NWTPH
GC Oven	HP	5890 SII	GC	CALDRO, WIDRO
GC Oven	Agilent	6890N	GC	CALDRO, WIDRO
GC System	HP	5890	PID/FID	8021/8015/GRO
GC system	HP	5890 Series II	PID/FID	8021/8015/GRO
GC system	HP	G1530A	PID/FID	8021/8015/GRO
GC System	HP	6890	GC	UST, BTEX
GC System	Agilent	6890	GC	8260/624/TCLP/UST
GC System	Agilent	6890	GC	8260 Med. Lvl Soil
GC System	Agilent Technologies	6850	MS	SIM/8260/624/Low & Med Lvl Soil/TCLP/UST
GC System	HP	6890	GC	8260/624/TCLP/UST
GC System	Agilent	6890A	GC	524/8260/624
GC/MS	Agilent	6890	GC/MS	1613/8290/Mthd 23,29/DW/PCB
GC/MS	Micromass	Autospec Ultima	GC/MS	1613/8290/Mthd 23,29
GC/MS	HP	6890A	GC/MS	1613/8290/Mthd 23,29/1614
GC/MS	Micromass	Autospec Ultima	GC/MS	1613/8290/Mthd 23,29
GC/MS	CTC Analytics	6890N	GC/MS	1613/8290/Mthd 23,29/TO9/DW
GC/MS	Micromass	Autospec Premier	GC/MS	1613/8290/Mthd 23,29/PCB
GC/MS	Thermo Scientific	Trace GC Ultra	GC/MS	1613/8290/Mthd 23,29/DW
GC/MS	Thermo Scientific	Trace GC Ultra	GC/MS	1613/8290/Mthd 23,29/DW
GC/MS	DFS	High Resolution Magnetic Sector MS	GC/MS	1613/8290/Mthd 23,29/DW

ATTACHMENT III – CONT'D

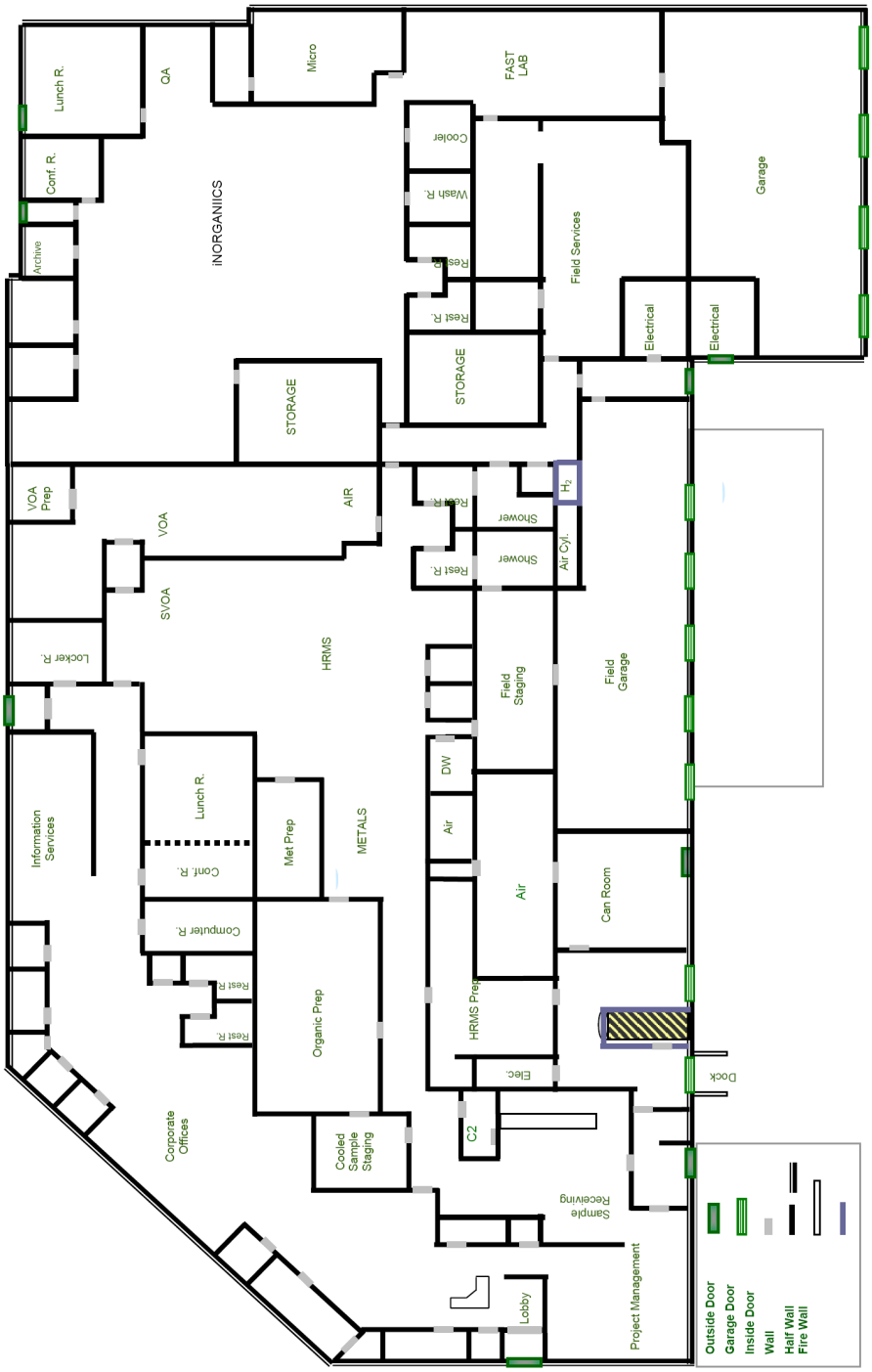
GC/MS	Thermo Scientific	Trace GC Ultra	GC/MS	1613/8290/Mthd 23,29/DW
GC/MS	Thermo Scientific	Trace GC Ultra	GC/MS	1613/8290/Mthd 23,29/DW
GC/MS	DFS	High Resolution Magnetic Sector MS	GC/MS	1613/8290/Mthd 23,29/DW
GC/Oven	Agilent	6890N	MS	Sulfolane, 8270, 625
GC/Oven	Agilent	6890A	MS	8270, 625
GCMS	ALS Ready	6890A	GC	TO3 BTEX
GCMS	Agilent	7890A	GC/MS	TO13, CPAH
GCMS	High Volume Injector		GC/MS	TO13, CPAH
Headspace Sampler	Agilent Technologies	G1888	GC/FID/TCD	RSK 175
Hot Block	Environmental Express	NA	NA	6010/Mercury/6020/200.8/Mthd 29
Hot Block	Environmental Express	NA	NA	6010/Mercury/6020/200.8/Mthd 29
Hot Block	Environmental Express	NA	NA	6010/Mercury/6020/200.8/Mthd 29
Hot Block	SCP Science	Digi Block	NA	6010/Mercury/6020/200.8/Mthd 29
Hot Plate	Thermolyne	Cimarec 3	NA	General - Wet Chem
Hot Plate	Presto		NA	General - Wet Chem
Hot Plate	Presto	Tilt'n Drain Big Griddle	NA	General - Wet Chem
Hot Plate	Corning	NA	NA	General - Wet Chem
IC Autosampler	Dionex	AS40-1	NA	Anions
ICP	Perkin Elmer Instruments	Optima 4300 DV	SCCD	Metals
ICP	Perkin Elmer Instruments	Optima 4300 DV	SCCD	Metals
ICPMS	Perkin Elmer Sciex	Elan 9000	MS	Metals
ICPMS	Thermo Scientific	Xseries 2	MS	Metals
Incubator	Fisher Scientific	Isotemp Incubator	NA	BOD
Incubator	Fisher Scientific	307	NA	BOD
Incubator	Fisher Scientific	307C	NA	BOD
Injector Tower	Agilent	7683	MS	CPAH, PCP
Ion Analyzer	Orion	EA 940	NA	pH
Ion Chromatograph	Dionex	ICS1000	NA	Anions
KoneLab	Thermo Fisher Scientific	Konelab 20	NA	Colormetric
Mass Spec	HP	5973	MS	CPAH, PCP
Micro 100 Turbidimeter	Scientific Inc.	Micro 100 Turbidimeter	NA	Turbidity
Microscope	Olympus	BH-2	NA	Asbestos
Microscope	Olympus	BH-2	NA	Asbestos
Microscope	Olympus	G10X	NA	Asbestos
Microscope	Olympus	BH-2	NA	Asbestos
Microwave	GE	JES1142WD04	NA	HPC Auger
Microwave	CEM	MarsXpress	NA	
Microwave Solvent Extraction	Milestone	Ethos E	NA	Mthd 29
MS	Agilent Technologies	5973 Network	GC/MS	TO-15
MS	Agilent Technologies		GC/MS	TO-15
MS	Agilent Technologies	5973 inert	GC/MS	TO-15
MS Detector	Agilent	5973N	MS	Sulfolane, 8270, 625
MS Detector	Agilent	5973N	MS	8270, 625
MS Detector	HP	5973	MS	UST, BTEX
MS Detector	Agilent	5973	MS	8260/624/TCLP/UST
MS Detector	Agilent	5973	MS	8260 Med. Lvl Soil
MS Detector	HP MS	5973	MS	8260/624/TCLP/UST
MS Detector	Agilent	5973	MS	524/8260/624
MS Detector	Agilent Technologies	5975C	MS	SIM/8260/624/Low & Med Lvl Soil/TCLP/UST
Muffle Furnace	Fischer Scientific	Isotemp Muffle Furnace	NA	General - O-prep
Muffle Furnace	Sybron	Thermolyne	NA	General - Montana
N-EVAP	Organomation	112	NA	General - O-prep
N-EVAP	Organomation	112	NA	General - O-prep
N-EVAP	Organomation	112	NA	General - HRMS Prep
N-EVAP	Organomation	112	NA	General - HRMS Prep
N-EVAP	Organomation	112	NA	General - HRMS Prep
Oven	Despatch	LDB Series	NA	General - AIR
Oven	Scientific Prod.	DK63	NA	General - HRMS Prep
Oven	Thermo Scientific	NA	NA	General - VOA
Oven	Precision Scientific	130 DM	NA	General - Wet Chem
Oven	VWR Scientific	1370F	NA	General - Wet Chem

ATTACHMENT III – CONT'D

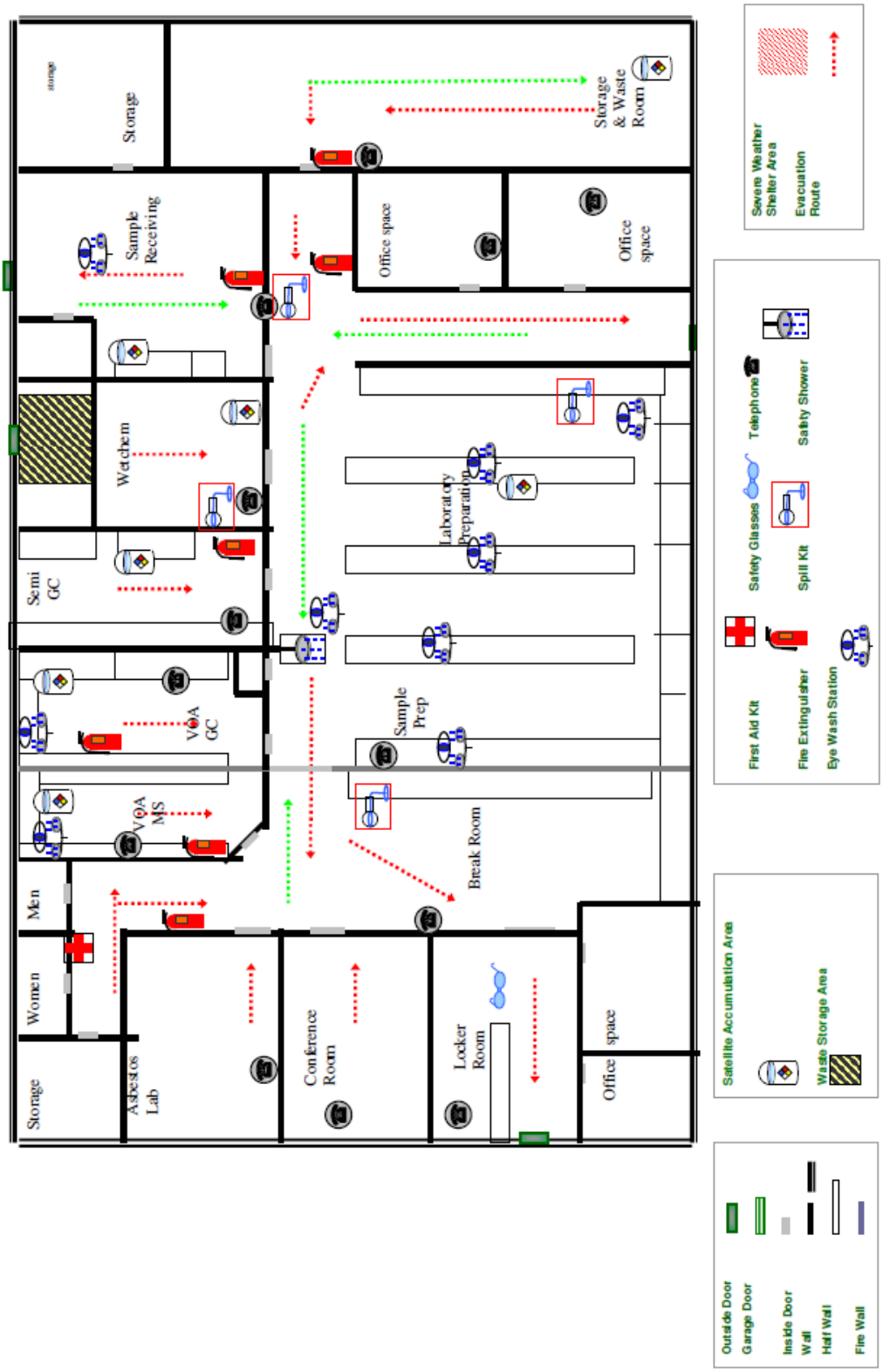
Oven	Fisher Scientific	Isotemp Oven	NA	General - Wet Chem
Oven	Fisher Scientific	NA	NA	% Moisture
Oven	Baxter DS-64	DS-64	NA	% Moisture
Oven	Fischer	Isotemp 255D	NA	General - Montana
Oven	Fischer	Isotemp 630F	NA	General - Montana
Oven	Despatch	288A	NA	General - Montana
pH Meter	IQ Scientific Instruments	NA	NA	pH
pH Meter	Orion	NA	NA	pH
pH meter	Fischer	AR50	NA	pH
Sample Concentrator	Tekmar	3100	NA	VPH
Smart Chem	West Co Scientific Instruments	Smart Chem 200	NA	Colormetric
Sonicator	Fisher Scientific	FS20D	NA	
Sonicator	Masonix	XL 2020	NA	3550
Sonicator	Masonix	XL 2015	NA	3550
Sonicator	Masonix	Sonicator 3000	NA	3550
Sonicator	Masonix	Sonicator 3000	NA	3550
Sonicator	Fisher Scientific	FS220	NA	8260/8021/8015/GRO
Sonicator	Fischer	FS60	NA	General - Montana
Sonicator	Heat Systems	Sonicator XL	NA	General - Montana
Sonicator	Branson	Sonfier 450	NA	General - Montana
Soxtherm	Gerhardt	NA	NA	8082
Soxtherm	Gerhardt	NA	NA	8082
Soxtherm	Gerhardt	NA	NA	8082
Soxtherm	Gerhardt	NA	NA	8082
Spectrometer	Hach	DR 2700	NA	COD
Spectrophotometer	Thermo	Aquamate	NA	Cr VI, N02, Tphos, Ophos
Stereoscope	Fischer	NA	NA	Asbestos
Stir Plate	Barnstead/Thermolyne	Super-Nuova	NA	General - Metals
Stir Plate	Corning Stirrer	NA	NA	General - Wet Chem
Stir Plate	Fisher Scientific	NA	NA	General - Wet Chem
Stir Plate	Thermolyne	Cimarec 2	NA	General - Wet Chem
Stir Plate	Fisher Scientific	NA	NA	General - Wet Chem
SVOA GC	Hewlett-Packard	5890A	FID/PID	EPH
SVOA GC	Hewlett-Packard	5890	FID/PID	EPH
Thermoreactor	Neutec Group Inc.	ECO 25	NA	COD
Tower	HP	N279	GC	PCB, TO4
Tower	Agilent	7683	GC	CALDRO, WIDRO
Tumbler	Associated Design & Mfg. Co.	3740-24BRE	NA	
Turbidimeter	Scienific Inc	Micro 100 Turbidimeter	NA	Turbidity
Turbidimeter	HF Scientific	Micro 1000	NA	Turbidity
UltraSonicator	Branson	8510	NA	General - O-prep
Vacuum Pump	Edwards	E2M2	NA	
VOA GC	Aglient	6890	FID/PID	VPH
VOA glassware oven	Sargent Welch		NA	General - Montana
Vortex Mixer	American Scientific Prod.	S8223-1	NA	General - Wet Chem
VPH GC	Hewlett-Packard	5890	PID/FID	VPH
Water Bath	Fisher Scientific	Isotemp 210	NA	General - Wet Chem
Waterbath	Northwest Fixtures		NA	General - Montana
	Agilent		GC	DRO
	Agilent	6890N	MS	SIM, PCP
	Agilent	6890	MS	8280

ATTACHMENT IV

PASI – MINNESOTA



ATTACHMENT IV CONT.
PASI-MONTANA DIVISION



ATTACHMENT V
SOP LIST

SOP Title	Number
Determination of Methane, Ethane, and Ethene in Air Modified TO-3	S-MN-A-002
Analysis of Air Samples for Volatile Organic Compounds by Gas Chromatography/PID-FID method TO-3	S-MN-A-003
Cleaning, Certification, Leak Checking and Preparation for Shipment of SUMMA Passivated Canisters	S-MN-A-004
Determination of Fixed Gases in Air by Modified 3C	S-MN-A-005
Methane, Ethane, Ethene, and Propane in Water by GCFID mod. 3810 and RSK 175	S-MN-A-007
Analysis of Whole Air Sample for Volatile Organic Compound by GC/MS EPA TO15/TO14	S-MN-A-013
Sample Management	S-MN-C-001
Bottle Preparatation	S-MN-C-003
Internal Chain of Custody	S-MN-C-005
Subcontracting Samples	S-MN-C-004
Bottle Order Database	S-MN-C-006
The Determination of Specific Aromatic Compounds and Gasoline Range Organic in Water and Soils	S-MN-O-427
Purgeable Total Petroleum Hydrocarbons in Water (8015 Mod / CA LUFT)	S-MN-O-525
Purgeable Total Petroleum Hydrocarbons in Water (NWTPH)	S-MN-O-555
Determination of Gasoline Range Organices by Method AK101	S-MN-O-556
Sample Preparation and Analysis of Polychlorinated Biphenyls (PCBs) in Ambient Air using High Volume Polyurethane Foam	S-MN-A-010
Analysis of Polychlorinated Biphenyls in Oil, Soil, Water, Wipe and Air Matrixes	S-MN-O-432
Determination of Diesel Range Organics in Water and Soil (Wisconsin modified DRO)	S-MN-O-466
Determination of Diesel Range Organics In Water & Soil SW8015 (Modified)	S-MN-O-489
Ethylene glycol, Propylene Glycol, Triethylene Glycol by Modified 8015	S-MN-O-533
The Determination of Extractable Petroleum Hydrocarbons by Method NwTPH-Dx	S-MN-O-553
The Determination of Diesel Range Organics and Residual Range Organics by AK102-AK103	S-MN-O-554
Preparation and Analysis of Samples for the Determination of Dioxins and Furans by USEPA Method 8290	S-MN-H-001
Preparation and Analysis of Samples for the Determination of Dioxins and Furans using USEPA Method 1613B	S-MN-H-002
Preparation and Analysis of Samples for the Determination of 2,3,7,8-TCDD using USEPA Method 1613B, Drinking Water	S-MN-H-003
Percent Lipids Determination	S-MN-H-004
Preparation and Analysis of Samples for the Determination of PCDDs, PCDFs, and PCBs by modified USEPA Method 23, TO9, or NY State Guidelines	S-MN-H-005
Preparation and Analysis of Samples for the Determination of Dioxins and Furans by USEPA Method 8280	S-MN-H-007
Method 1668, PCB Congenger (WHO List)	S-MN-H-009
Preparation and Analysis of Samples for the Determination of Chlorinated Biphenyl Congeners by USEPA Method 1668A	S-MN-H-014
Preparation and Analysis of Samples for the Determination of Polybrominated Diphenyl Ether Congeners	S-MN-H-016
Preparation and Analysis of Samples for the Determination of Dioxins and Furans by USEPA Method 8290A	S-MN-H-019

ATTACHMENT V – CONT'D

Preparation and Analysis of Samples for the Determination of Dioxin and Furans by USEPA Method DLM2.0	S-MN-H-021
Preparation and Analysis of Samples for the Determination of Chlorinated Biphenyl Congeners	S-MN-H-022
Operation and Maintenance of the Perkin Elmer ELAN 9000 ICP-MS	S-MN-I-525
TCLP/SPLP	S-MN-I-312
Inductively Coupled Plasma Atomic Emission Spectroscopy (RCRA)	S-MN-I-313
Hardness by Calculation	S-MN-I-338
Mercury im Liquid and Solid/Semis-Solid Waste	S-MN-I-359
Digest Procedure for Aqueous Samples to be Analyzed by Induct Coupled Plasma (SW-846)	S-MN-I-458
Metals Preparation for Solid samples, Wipes and Filters	S-MN-I-460
Analysis of Air Samples by EPA Method 29	S-MN-I-487
Metals Analysis by ICP/MS - Method 6020 and 200.8	S-MN-I-492
Mercury in End Caps and Glass Samples	S-MN-I-517
Preparation of Aqueous Samples for ICPMS Analysis by Method 3030C	S-MN-I-523
Operation of the DEENA automated Prep System	S-MN-I-531
Mercury in Solid Waste by CLP	S-MN-I-557
Operation and Maintenance of the Perkin Elmer Optima 4300 ICP	S-MN-I-567
Operation and Maintenance of the Milestone Ethos E Microwave Digester	S-MN-I-575
Extractable Base/Neutral and Acid Organic Compounds in Liquid, Solid, and TCLP Matrices by Gas Chromatography/Mass Spectrometry Capillary Column Technique	S-MN-O-436
8270-L Extractable Base/Neutral and Acid Organic Compounds in Water and Liquid Matrices by GC/MS Capillary Column Technique w/Selective Ion Monitoring	S-MN-O-507
Extractable Base/Neutral and Acid Organic Compounds in Liquid by EPA Method 625	S-MN-O-532
Determination of Parent and Alkylated PAH Compounds in Solid and Liquid Matrices by GC/MS SIM	S-MN-O-561
Sulfolane Extraction and Analysis in Liquid Matrices by GC/MS Capillary Column Technique w/Selective Ion Monitoring	S-MN-O-563
Analysis of Air samples by GC/MS - Method TO-13	S-MN-O-534
Analysis of Volatile Organic Compounds by GC/MS Method 8260	S-MN-O-521
Analysis of Volatile Organic Compounds by GC/MS Method 624	S-MN-O-529
Analysis of Volatile Organic Compounds in Water Method 524.2	S-MN-O-546
Analysis of 1,4 Dioxane by Selective Ion Monitoring (SIM) GC/MS SW846 Method 8260B Modified	S-MN-O-558
Method For Sonicator Tuning	S-MN-O-414
Cleaning Glassware in the Organic Laboratory	S-MN-O-465
Determination of Acid Cleanup of PCB Extracts	S-MN-O-494
Sonication Extraction Technique (SW3550) for Base/Neutral and Acid Compounds	S-MN-O-495
Continuous Liquid-Liquid Extraction (SW3520) for Base/Neutral and Acid Compounds	S-MN-O-496
Spike Verification in the Organic Prep Lab	S-MN-O-497
Preparation of Anhydrous Sodium Sulfate for Extraction Purposes	S-MN-O-500
Nitrogen Evaporation Technique	S-MN-O-503
Sample Concentration Technique	S-MN-O-504
Continuous Liquid-Liquid Extraction (SW3520) for Polyaromatic Hydrocarbons by 8270-SIM	S-MN-O-506
Solvent Exchange into Hexane	S-MN-O-509
Copper Cleanup Procedure for Polychlorinated Biphenyls	S-MN-O-527
Continuous Liq/Liq extraction for Method 8270C (Dual pH) by SW 3520C	S-MN-O-539

ATTACHMENT V – CONT'D

Soxhlet Extraction for PAH Analysis by GC/MS:SIM	S-MN-O-540
DrieRite Regeneration Procedure	S-MN-O-557
Percent Solids (Moisture)	S-MN-I-367
Separatory Funnel Extraction	S-MN-O-566
Data Archiving	S-MN-L-106
Reagent Water Quality	S-MN-L-110
Generation of EDD	S-MN-L-112
Preventative, routine, and non-routine maintenance	MN-L-114
Receipt and Storage of Laboratory Supplies	S-MN-L-117
Common Laboratory Calculations and Statistical Evalation of Data	S-MN-L-125
Data Reduction, Validation, and Reporting in the Env Lab	S-MN-L-132
Syringe Technique	S-MN-L-139
Procedure for Handling Aqueous Organic Extractable Samples Containing Sediment	S-MN-L-142
Purchasing Laboratory Supplies	S-MN-L-143
Quality Manual	Quality Manual
Training Procedures Addendum	S-All-MN-Q-020
Volatiles Sample Compositing Procedure	S-MN-O-541
Manual Integration	S-MN-Q-214
Precision and Accuracy Measurement, Evaluation, and Trend Assessment	S-MN-Q-205
Control of Hazardous Energy Program - Lockout/Tagout	S-MN-Q-249
Method Validation and Modification Studies	S-MN-Q-252
Procedure for Handling of USDA regulated soils	S-MN-Q-253
Estimation of Measurement Uncertainty	S-MN-Q-255
Method of Change	S-MN-Q-257
PT Program	S-MN-Q-258
Use of A2LA Terms and Symbols	S-MN-Q-260
Conflict of Interest Plan	S-MN-Q-261
Corrective Action and Preventative Action	S-MN-Q-262
Support Equipment	S-MN-Q-264
Quality System Review	S-MN-Q-265
Use and Operation of Lab Track System	S-MN-Q-266
MintMinerÓ Data File Review	S-MN-Q-267
Method Detection Limit Studies	S-MN-Q-269
Chemical Hygiene Plan/Safety Manual	S-MN-S-001
Waste Training Management Requirements	S-MN-S-002
Waste Handling	S-MN-S-003
Water Extraction of Soil	S-MN-I-334
Biochemical Oxygen Demand (BOD)	S-MN-I-348
COD-Titrimetric, Low Level	S-MN-I-351
Phenols	S-MN-I-354
Oil & Grease - 1664	S-MN-I-357
Hexavalent Chromiumin in Water, Wastewater, and Soil	S-MN-I-358
Alkalinity, Titrimetric	S-MN-I-365
Total Cyanide in Water	S-MN-I-366
Fluoride in Water and Wastewater	S-MN-I-470
Chemical Oxygen Demand (COD) in Water, Wastwaters and Industrial Wastes	S-MN-I-472
Determination of Total and Ortho Phosphorus in Aqueous Samples by SmartChem	S-MN-I-473
Specific Conductivity	S-MN-I-474

ATTACHMENT V – CONT'D

Ortho Phosphorus	S-MN-I-477
Particulate Matter (PM10) (Method 5) in the Atmosphere	S-MN-I-484
Settleable Solids	S-MN-I-486
Standard Test Method for Screening Apparent Specific Gravity and Bulk Density Waste	S-MN-I-493
Determination of Total Recoverable Phenolics by Flow Injection Colorimetry	S-MN-I-494
Turbidity in Water	S-MN-I-501
Chlorine, Total Residual in Water	S-MN-I-502
Use and Maintenance of the Konelab	S-MN-I-507
Determination of Nitrate/Nitrite in surface/wastewaters by Flow Injection Analysis by SmartChem	S-MN-I-508
Determination of Chloride by Konelab	S-MN-I-509
Determination of Sulfate by Konelab	S-MN-I-510
Determination of Ammonia by Konelab Analysis, Colorimetry	S-MN-I-511
Determination of Nitrite by Konelab(Spectrophotometric Method)	S-MN-I-514
Amenable Cyanide and Weak Dissociable Cyanide in Water	S-MN-I-515
Paint Filter Liquids Test	S-MN-I-516
Gravimetric Determination of Oil and Grease by SPE	S-MN-I-520
Dissolved Oxygen	S-MN-I-524
Measurement of pH in Water, Soil, and Waste	S-MN-I-526
Determination of TSP and PM 10	S-MN-I-527
Measurement of Solids in Water and Wastewater	S-MN-I-528
Total CN in Water - Macro Distillation	S-MN-I-529
Weak Acid Disociable Cyanide in Water - Macro Distillation	S-MN-I-530
Total Coliform Bacteria	S-MN-MB-001
Fecal Coliform by MF	S-MN-MB-002
Heterotrophic Plate Count	S-MN-MB-003
Total Coliform Bacteria by MF	S-MN-MB-005
Sample Container Sterility Verification	S-MN-MB-006
The Determination of Ammonia by SmartChem	S-MN-I-559
Determination of NO3/NO2 by SmartChem	S-MN-I-560
COD by Hach 2700	S-MN-I-563
Determination of Inorganic Anions by Ion Chromatography	S-MN-I-532
Quality Control Recordkeeping For Bulk Asbestos Analysis	S-MN-I-533
Bulk Analysis Using Polarized Light Microscopy	S-MN-I-534
Microscope Adjustment - Phase Contrast	S-MN-I-535
Microscope Alignment	S-MN-I-536
Fiber Counts By NIOSH 7400 Using Excel Spreadsheet	S-MN-I-537
The Determination of Nitrate-Nitrite by Flow Analyzer	S-MN-I-539
TKN By Colorimetry	S-MN-I-541
Determination of Ammonia Nitrogen by Automated Phenate	S-MN-I-542
Phosphorus, Ortho and Total	S-MN-I-543
Sulfides	S-MN-I-544
Asbestos Data Review	S-MN-I-545
Total Sufur by LECO	S-MN-I-547
Colormetric Hexavalent Chromium	S-MN-I-548
Particle Size Analysis	S-MN-I-549
Sobek	S-MN-I-550
Fluoride Distillation	S-MN-I-551

ATTACHMENT V – CONT'D

Coarse Fragment	S-MN-I-552
Water Soluble Sulfate and Chloride	S-MN-I-554
Specific Conductivity SW2510B	S-MN-I-555
pH Paste	S-MN-I-558
Cation - Anion Balance	S-MN-I-562
Wet Sieve	S-MN-I-568
The Determination of Volatile Petroleum Hydrocarbons by Method MA-VPH	S-MN-O-559
The Determination of Extractable Petroleum Hydrocarbons by Method MA-EPH	S-MN-O-560
Petroleum Hydrocarbons as Diesel in Water and Soil	S-MN-O-562
Organic Matter	S-MN-O-564
Purgeable Total Petroleum Hydrocarbons in Water and Soil	S-MN-O-565

ATTACHMENT VI
CERTIFICATION LIST

State	Agency	Program	Cert #
A2LA		Dioxin; DOD	2926.01
Alabama	Dept of Environmental Mgmt	Dioxin-DW	40770
Alaska	Dept. of Environmental Conservation	Contaminated Sites (6010B, 6020, 8260B, PCBs, PAHs)	UST-078
Alaska	Dept. of Environmental Conservation	Dioxin-DW	MN00064
Arizona	Dept of Health Services	Dioxin-DW, WW, HW	AZ0014
Arkansas	Dept of Environmental Quality	Dioxins	88-0680
California	Dept of Health Services	Dioxin-DW, WW, HW Envir-DW, WW, HW	01155CA
Colorado	Dept. of Public Health & Environment	Dioxin-DW	Pace Analytical
Colorado	Dept. of Public Health & Environment	Asbestos Registration	17119*
Connecticut	Dept of Public Health	Dioxins	PH-0256
Delaware	Health & Social Services	Dioxin-DW	
EPA Region 5	Water Division	Dioxin-DW	WD-15J
EPA Region 8	Water Division	Dioxin-DW, DW	MN00064 MT00012*
Florida (NELAP)	Dept of Health Services	Diox-DW, WW, HW, Air Envir-DW, WW, HW, Air	E87605
Georgia	Environmental Protection Division	Dioxin-WW, HW via NELAP	E87605
Georgia	Dept of Natural Resources	Dioxin-DW	959
Guam	Guam EPA	Dioxin-DW	Pace Analytical
Idaho	Dept. of Health & Welfare	Dioxin-DW	Pace Analytical MT00012*
Hawaii	Dept of Health	Dioxin-DW	SLD
Hennepin County Waste Generator License		NA	
Illinois	Illinois EPA	Dioxin-DW, HW, WW via NELAP	200011
Indiana	Dept of Health	Dioxin-DW via EPA Region 5	C-MN-01
Iowa	Dept.of Natural Resources	Envir.-DW, WW, UST	368
Kansas	Dept of Health and Environment	Dioxin-DW Envir-DW, WW, HW	E-10167
Kentucky	Dept of Environmental Protection	Dioxin-DW	90062
Louisiana DEQ	Department of Environmental Quality	Dioxin-WW, HW, Air	3086
Louisiana DHH	Department of Health and Hospitals	Dioxin-DW	LA090015
Maine	Dept of Human Services	Dioxin-DW via EPA Region 5	2007029
Maryland	Dept. of Heath and Mental Hygiene	Dioxin-DW	322
Michigan	Dept. of Public Health	Dioxin-DW	9909
Minnesota	Dept of Health	Envir-DW, WW, HW	027-053-137
Minnesota	Department of Commerce	Petrofund	1240
Mississippi	Dept. of Health and Environmental Control	Dioxin-DW	Pace
Montana	Dept of Health	Dioxin-DW	92 40*
Nebraska	Dept. of Health & Human Services.	Dioxin-DW	Pace

The scope and application certificates are maintained in the filing cabinets in the QA Department.

ATTACHMENT VI – CONT'D

State	Agency	Program	Cert #
Nevada	Health Division	Dioxin-DW, WW	MN_00064_2000_72
New Jersey	Dept of Environmental Protection	Dioxin-DW, WW, HW Envir-WW, HW, Air	MN002
New Mexico	NM Environment Dept.	Dioxin-DW, Env-DW	Pace
New York	Dept of Health	Dioxin-DW, WW, Air Envir-Air	11647
North Carolina	Dept of Environment, Health and Natural Resources	Envir-WW, HW	530
North Carolina	State Public Health Laboratory	Dioxin-DW	27700
North Dakota	Dept of Health and Consolidated Labs	Envir-DW, WW, HW	R-036
		RCRA tests via FI cert	R-036A
NVLAP		Asbestos	101292-0*
Ohio	Ohio EPA	Dioxin-DW via EPA Region 5	4150
Ohio Vap	VAP	Dioxin and Air	CL101
Oklahoma	Dept of Environmental Quality	Dioxin-DW	D9921
Oklahoma	Dept of Environmental Quality	Envir-HW	9507
Oregon	ELAP	Dioxin-DW, WW, HW, Air Enviro: Air	MN200001-005
Pennsylvania	Dept of Environmental Protection	Dioxin-DW, WW, HW Envir: DW, WW, HW	68-00563
Puerto Rico	Department of Health	Dioxin, DW metals	Pace
Saipan (CNMI)	Div. Of Environmental Quality	Dioxin-DW	MP0003
South Carolina	Dept. of Health and Environmental Control	Dioxin-DW, WW, HW	74003001
Texas	Department of Health	Dioxin-DW, WW, HW	T104704192-08A-TX
Tennessee	Department of Health	Dioxin-DW Envir-DW	2818
Utah	Department of Health	Dioxin-DW, WW, HW	ID# PAM
Virginia	Dept of General Services	Dioxin-DW	251
Washington	Dept of Ecology	Dioxin-DW, WW, HW Envir-DW, WW, HW	C486
Wisconsin	Dept of Natural Resources	Dioxin-DW, WW, HW Envir-DW, WW, HW	999407970
Wyoming	Via EPA Region 8	Dioxin DW	
West Virginia	Dept of Health and Human Resources	Dioxin-DW, Air	9952C

*Certification held by the Montana Division.

The scope and application certificates are maintained in the filing cabinets in the QA Department.

ATTACHMENT VII
CHAIN OF CUSTODY (EXAMPLE)

CHAIN-OF-CUSTODY / Analytical Request Document

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

Pace Analytical™ www.pacelabs.com

Page: of

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

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

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

REGULATORY AGENCY
☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER
☐ UST ☐ RCRA ☐ OTHER
Site Location STATE:

Section D Required Client Information

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

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

ITEM #	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test ↓ Y/N	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				
9																				
10																				
11																				
12																				

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS	Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER:

SIGNATURE of SAMPLER: DATE Signed (MM/DD/YY):

ATTACHMENT VIII
METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
2, 3, 7, 8-TCDD	1613B	Soil	8oz Glass	None	90/40 Days
2, 3, 7, 8-TCDD	1613B	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	90/40 Days
2, 3, 7, 8-TCDD	8290	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Acidity	SM2310B	Water	Plastic/Glass	≤6°C	14 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	≤6°C	14 Days
Alpha Emitting Radium Isotopes	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Anions by IC, including Br, Cl, F, NO_2, NO_3, SO_4	300.0/300.1/ SM4110B	Water	Plastic/Glass	≤6°C	Br, Cl, F, SO_4 (28 Days) NO_2, NO_3 (48 Hours)
Anions by IC, including Br, Cl, F, NO_2, NO_3, SO_4	300.0/9056	Soil	Plastic/Glass	≤6°C	Br, Cl, F, SO_4 (28 Days) NO_2, NO_3 (48 Hours)
Aromatic and Halogenated Volatiles	8021	Soil	5035 vial kit	See 5035 note*	14 days
Aromatic and Halogenated Volatiles	601/602/8021	Water	40mL vials	pH<2 HCl; ≤6°C; $Na_2S_2O_3$ if Cl present	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Soil	8oz Glass	≤6°C	14 Days
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤6°C; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Soil	8oz Glass	≤6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.1/525.2	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/30 Days
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤6°C	48 hours
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	14 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag	None	48 Hours
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Chloride	SM4500Cl/9250/	Water	Plastic/Glass	None	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
	9251/9252				
Chlorinated Herbicides	8151	Soil	8oz Glass Jar	≤6°C	7/40 Days
Chlorinated Herbicides	8151	Water	1L Amber Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Chlorinated Herbicides	515.1	Water	1L Amber Glass	≤6°C; $Na_2S_2O_3$ if Cl present	14/28 Days
Chorine, Residual	SM4500Cl	Water	Plastic/Glass	None	15 minutes
COD	SM5220C/ 410.3/410.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Color	SM2120B,C,E	Water	Plastic/Glass	≤6°C	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	6 Months
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN/9010/ 9012/335.4	Water		pH>12 NaOH; ≤6°C; ascorbic acid if Cl present	14 Days, 24 Hours if Sulfide present
Diesel Range Organics-TPH DRO	8015	Soil	8oz Glass Jar	≤6°C	14/40 Days
Diesel Range Organics-TPH DRO	8015	Water	1L Glass	≤6°C	7/40 Days
Diesel Range Organics (WI)	WI MOD DRO	Soil	8oz Glass Jar	≤6°C	10/47 Days
Diesel Range Organics (WI)	WI MOD DRO	Water	1L Glass	≤6°C	14/40 Days
Dioxins & Furans	TO-9	Air	PUF	None	30/45 Days
EDB & DBCP	504.1/8011	Water	40mL vials	≤6°C; $Na_2S_2O_3$ if Cl present	14 Days
Explosives	8330/8332	Water	1L Glass	≤6°C	7/40 Days
Explosives	8330/8332	Soil	8oz Glass Jar	≤6°C	14/40 Days
Fecal Coliform	SM9222D	Water	100mL Plastic	≤6°C	6 Hours
Fecal Coliform	SM9222D	Soil	100mL Plastic	≤6°C	6 Hours
Ferrous Iron	SN3500Fe-D	Water	Glass	None	Immediate
Flashpoint/Ignitability	1010/1030	Water	Plastic/Glass	None	28 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gas Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Soil	5035 vial kit	See 5035 note*	14 days
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤6°C	14/7 Days
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	6 Months

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Heterotrophic Plate Count (MPC)	EPA 9215B	Water	100mL Plastic	≤6°C	24 Hours
Hexavalent Chromium	7196/218.6/ SM3500Cr	Water	Plastic/Glass	≤6°C	24 Hours
Hydrogen Halide & Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Low Level Mercury	1631	Water	Glass	BrCl	90 days (if preserved and oxidized)
Mercury	7471	Soil	8oz Glass Jar	≤6°C	28 days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Metals	7300/7303	Air	Filters	None	6 Months
Metals (and other ICP elements)	6010	Soil	8oz Glass Jar	None	6 months
Metals (and other ICP elements)	6010/6020/200.7/ 200.8	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Methane, Ethane, Ethene	EPA Mod 8015	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag	None	48 Hours
Methanol, Ethanol	EPA 8015	Water	40mL vials	≤6°C	14 Days
Methanol, Ethanol	EPA 8015	Soil	2oz Glass	≤6°C	14 Days
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Nitrogen, Kjeldahl	SM4500-Norg; 351.1/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/ 352.1	Water	Plastic/Glass	≤6°C	48 Hours
Nitrogen, Nitrate & Nitrite	SM4500-NO3/ 353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Nitrogen, Nitrite	SM4500-NO2/ 353.2	Water	Plastic/Glass	≤6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/ 351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag	None	48 Hours
Odor	SM2150B	Water	Glass	≤6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/ 9070	Water	Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Organochlorine Pesticides & PCBs	TO-4	Air	PUF	None	7/40 Days
Organochlorine Pesticides & PCBs	8081/8082/608	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Organochlorine Pesticides & PCBs	8081/8082	Soil	8oz Glass Jar	≤6°C	14/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Organophosphorous Pesticides	8141	Soil	8oz Glass Jar	≤6°C	14/40 Days
Organophosphorous Pesticides	8141	Water	1L Amber Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particulates	PM-10	Air	Filters	None	6 Months
Permanent Gases	EPA 3C	Air	Summa Canister	None	14 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag	None	48 Hours
pH	SM4500H+B/9040/ 9041/150.2	Water	Plastic/Glass	None	15 minutes
Phenol, Total	420.1/420.4/9065/ 9066	Water	Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; ≤6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Phosphorus, Total	EPA 365.4	Soil	Plastic/Glass	≤6°C	28 Days
Polynuclear Aromatic Hydrocarbons	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons	8270 SIM	Soil	8oz Glass Jar	≤6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons	8270 SIM	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226 Radon Emanation Technique	903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤6°C	7 Days
Solids, Total (FOC)	ASTM D2974	Soil	Plastic/Glass	≤6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤6°C	7 Days
Solids, Total Suspended	SM2540D	Water	Plastic/Glass	≤6°C	7 Days
Solids, Total Volatile	SM2540E	Water	Plastic/Glass	≤6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤6°C	28 Days
Stationary Source Dioxins & Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	6 Months, 28 Days for

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
					Hg
Stationary Source Metals	EPA 29	Air	Filters	None	6 Months, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	6 Months
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	6 Months
Sulfate	SM4500SO4/9036/ 9038/375.2/ASTMD516	Water	Plastic/Glass	≤6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants	SM5540C	Water	Plastic/Glass	≤6°C	48 Hours
Total Organic Carbon (TOC)	SM5310B,C,D/ 9060	Water	Glass	pH<2 H_2SO_4 or HCl; ≤6°C	28 Days
Total Organic Halogen (TOX)	SM5320/9020/ 9021	Water	Glass; no headspace	≤6°C	14 Days
Tritium	906.0	Water	Glass	pH<2 HNO_3	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤6°C	48 Hours
Uranium Radiochemical Method	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
Volatiles	TO-14	Air	Summa Canister	None	30 Days
Volatiles	TO-14	Air	Tedlar Bag	None	48 Hours
Volatiles	TO-15	Air	Summa Canister	None	30 Days
Volatiles	8260	Soil	5035 vial kit	See 5035 note*	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤6°C; $Na_2S_2O_3$ if Cl present	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤6°C; $Na_2S_2O_3$ if Cl present	14 Days (7 unpreserved)
Volatiles	524.1/524.2	Water	40mL vials	pH<2 HCl; ≤6°C; $Na_2S_2O_3$ if Cl present	14 Days
Alaska DRO	AK102	Soil	8oz Glass	≤6°C	14/40 Days
Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤6°C	14/40 Days
Alaska RRO	AK103	Soil	8oz Glass	≤6°C	14/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Alaska GRO	AK101	Soil	5035 vial kit	See 5035 note*	14 Days
Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤6°C	14 Days

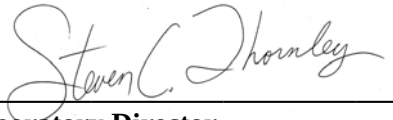
5035 Note: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4°C, **and** one vial methanol preserved at ≤6°C **and** one container of unpreserved sample stored at ≤6°C.

Quality Assurance Program Plan for the Analysis of Soil-Gas Samples Collected with BEACON's Passive Soil-Gas System

Prepared by

Beacon Environmental Services, Inc.
323 Williams Street
Suite D
Bel Air, MD 21014

President **May 25, 2010**
Date



Laboratory Director **May 25, 2010**
Date

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1.0 INTRODUCTION

The objective of Beacon Environmental Services, Inc. (BEACON) is to provide analytical data that are valid and defensible and which are provided in a timely manner for purposes of screening a site for volatile and semivolatile organic compounds (VOCs and SVOCs). This Quality Assurance Program Plan (QAPP) has been prepared in direct response to these goals. This plan describes the quality assurance program to be implemented and the quality control procedures to be followed for the analysis of passive soil-gas samples by gas chromatography/mass spectrometry (EPA Methods 8260C, 8270, TO-17 and TO-1). EPA Methods 8260C and 8270 are modified for the injection of the sample by thermal desorption.

2.0 ORGANIZATION AND RESPONSIBILITIES

2.1 Structure

Beacon Environmental Services, Inc., (BEACON) is a privately owned company that provides services for the collection and analysis of passive soil gas samples collected in accordance with BEACON's Quality Assurance Program Plan for Passive Soil-Gas Sampling. BEACON's office is located at 323 Williams Street, Bel Air, MD 21014, which is approximately seven miles north of I-95 between Baltimore, MD and Philadelphia, PA.

2.2 Roles and Responsibilities

All laboratory personnel are involved with the Quality Assurance (QA) Program. The extent of their involvement depends on their assignment in the laboratory; however, all laboratory personnel are trained for their role in supporting the analyses of samples.

2.2.1 President

The President of BEACON has overall responsibility for the operation of the laboratory. The specific responsibilities include:

- providing support and resources for the QA Program
- maintaining laboratory staffing
- coordinating training of personnel in all aspects of the laboratory
- approving equipment acquisition
- developing the laboratory budget
- maintaining and implementing the marketing program
- implementing the operational aspects of the QA Program
- ensuring the laboratory data quality as the Quality Assurance Officer
- reviewing data requirements for each project with Laboratory Director
- ensuring corrective actions specified by the Laboratory Director are implemented

2.2.2 Laboratory Director

The Laboratory Director reports to the President of BEACON. The Laboratory Director is primarily responsible for on-schedule completion of assigned laboratory work and for supervising all laboratory activities, including implementation of the Quality Assurance/Quality Control (QA/QC) program. The Laboratory Director enlists and encourages the cooperation of all the staff in the program. Specific responsibilities of the Laboratory Director include:

- ensuring that all analyses are performed according to the methods and protocols specified by the client
- reviewing all analytical data by (i) checking documentation for completeness and proper sample identification, (ii) checking raw data for calculation, interpretation, or clerical errors, (iii) assuring that produced quality control data are acceptable
- ensuring laboratory data quality

- coordinating analytical work to ensure that all tasks are completed within established time frames
- overseeing preventative maintenance activities
- establishing analytical priorities and reviewing data requirements for each project
- reviewing initiated corrective actions and recommending additional measures, if necessary
- ensuring corrective actions are implemented
- reviewing quality control data to determine if test data are acceptable
- supervising the updating of accuracy, precision, and method detection limits
- performing periodical system audits to assure compliance with all quality assurance requirements
- evaluating and implementing changes in methodology and quality control measures
- identifying quality control problems and taking measures to correct or eliminate the problem source
- validating all data and assuring that data sets are accurate before reporting

2.2.3 Analysts

Analysts report to the Laboratory Director. They are responsible for on-schedule performance and documentation of all analyses assigned. Moreover, their responsibilities include:

- performing required analyses according to test methods specified
- assuring that all analytical equipment has been properly calibrated before beginning the analyses
- assuring that all identifying information (including sample control numbers, project numbers, and client information) have been accurately transcribed into records or computer data bases
- assuring that all calculations are correct
- assuring that appropriate confirmatory tests or procedures have been completed
- identifying, documenting, and beginning corrective actions on any quality control problem that relates to the analytical method
- maintaining equipment in working conditions and documenting all preventive maintenance and repairs

2.2.4 Operations Manager

The Operations Manager reports to the President of BEACON and is responsible for the administrative aspects of the laboratory. Specific responsibilities include:

- coordinating the preparation of passive soil-gas samplers and the shipment of kits
- receiving samples as primary sample custodian
- initiating paperwork for sample analyses on appropriate laboratory documents (including establishing project files and sample receipt records) as required for analysis
- obtaining, filing, and distributing pertinent project information to laboratory staff
- reporting laboratory results to the client
- understanding and following aspects of QA program related to job function
- managing BEACON central file system, which includes project statements of work or proposals, quality assurance plans, chain-of-custody records, and final data reports
- initiating and tracking archives for all laboratory documents

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

This section presents the QA objectives for the chemical data in terms of precision, accuracy, completeness, representativeness, and comparability.

3.1 Precision

Precision is the mutual agreement among individual measurements of the same property and is a measure of the random error component of the data collection process. The overall precision of the data is the sum of that resulting from the sampling and analysis. The sampling precision is assessed by collecting field sample duplicates, when appropriate, and accompanying every sample batch with a trip blank. The analytical precision is determined by preparing and analyzing spiked replicate samples. Precision can be expressed in several different ways, each of which has its uses; for multiple measurements these include the standard deviation, the relative standard deviation, and the range.

3.2 Accuracy

Accuracy is the degree of agreement of a measured value with the true or expected value of the measured quantity. It is a measure of the bias or systematic error of the entire data collection process. Sources of these errors include the sampling process, field and laboratory contamination, sample handling, sample matrix, sample preparation methods, and calibration and analysis procedures. Sampling accuracy is assessed by evaluating the results of sample preparation blanks, ambient-air control samples, field sample location duplicates, and trip blanks. Analytical accuracy is assessed through the use of calibration verification samples, method blanks, laboratory control samples, and sample preparation blanks.

3.3 Representativeness

Data representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representation is a quantitative parameter that is most concerned with the proper design of the sampling program. The sampling program has been designed so that the samples collected are as representative as possible of the medium being sampled and that a sufficient number of samples will be collected. Representativeness is addressed by the description of the sampling techniques and the rationale used to select the sampling locations.

3.4 Completeness

Completeness is defined as the percentage of measurements made that are judged to be valid data. To achieve this objective, every effort is made to avoid sample loss through accidents or inadvertence. Accidents during sample transport or lab activities that cause the loss of the original sample will result in irreparable loss of data. The assignment of a set of laboratory numbers to a batch of samples that have undergone chain-of-custody inspection makes it more difficult for the analyst to overlook samples when setting up a batch of samples for analysis. The laboratory numbers also make it easy during the data compilation stage to pick out the samples which have not been analyzed and to order their analysis before the data are reported and before holding times have been exceeded. The completeness of each batch of

samples can be calculated by dividing the total number of analyses completed by the number that should have been performed on that batch times 100.

3.5 Comparability

Data comparability is a measure of the confidence with which one data set can be compared to another. It cannot be described in quantitative terms, but must be considered in designing the sampling plans, analytical methodology, quality control, and data reporting. The use of standard EPA-verified sampling techniques and validated, EPA-approved analytical methods assures that the parameters being measured are comparable with data generated from other sources with comparable equipment. Reporting of data in units used by other organizations also assures comparability.

3.6 Project QA Objectives

Unless otherwise specified, the accuracy objective is 70-130%, for analytical precision ±25%, and completeness 99%. The accuracy and precision are based on the analysis of the standard spiking of a blank adsorbent sampler. The accuracy is the percent recovery of the target analytes, and the precision is the standard deviation of successive percent recoveries. The results of samples for which the recovery of the standards does not fall within these limits will be qualified as being outside the control limits. Beacon voluntarily participates in performance testing using blind samples to evaluate our method.

4.0 SAMPLE CUSTODY PROCEDURES

The laboratory chain-of-custody procedures will document sample possession from the time of shipment to the time of receipt and final analysis, in accordance with BEACON procedures and federal guidelines. The National Enforcement Investigations Center (NEIC) of U. S. EPA defines custody of evidence in the following ways (i) it is in your actual possession, (ii) it is in your view, after being in your physical possession, (iii) it was in your possession and then you locked or sealed it up to prevent tampering, and (iv) it is in a secure area.

4.1 Chain-Of-Custody Form

A chain-of-custody form serves as permanent documentation of sample validity in which is recorded all pertinent aspects of sample collection and handling prior to delivery to the laboratory. A chain-of-custody form is generated when project adsorbent samplers are shipped to the client. A completed chain-of-custody form contains the following information: (i) client name and project site, (ii) sample identification name/number, (iii) attendant chain-of-custody names, signatures, dates, and times (if applicable), including name of courier, (iv) analyses requested, (v) date and time of receipt, and (vi) special instructions. The laboratory has the final responsibility to ensure that all necessary documentation is properly recorded. Deviations from established protocols are recorded on the chain-of-custody form. The laboratory will make note of samples collected for analysis without a correctly prepared and relinquished chain-of-custody form and contact the client prior to initiating analysis.

4.2 Sample Receipt

The Operations Manager and those designated are authorized sample custodians. Each authorized employee shall be prepared to testify in a court of law as to the nature and extent of access to, or possession of, any sample in the custody of BEACON. The sample custodian performs the following tasks after receiving samples: (i) inspects sample shipping containers for presence/absence and condition of custody seals, samples, and field kit equipment, (ii) records condition of both shipping containers and sample containers on the chain-of-custody form, (iii) verifies and records agreement or non-agreement of information on sample documents on the chain-of-custody form, (iv) verifies the number of sample containers received is equal to the number of samples listed on the chain-of-custody form, (v) logs all samples into sample receipt logbook that records sample identifications, requested analytical method, project identification number, any discrepancies in packaging or labeling, date, and name of sample custodian (vi) signs chain-of-custody shipped with samples, (vii) communicates any problems or discrepancies to the Laboratory Director, (viii) places samples in a project specified bag and places the bag in the sample refrigerator, and (ix) places chain-of-custody and field deployment forms in client file.

4.3 Sample Security and Accessibility

The laboratory is maintained in a safe and secure manner at all times. The facility is locked when not occupied and is monitored for fire and unauthorized access. BEACON personnel escort all visitors at all times while inside the facility.

When not actually employed in analysis, the samples are stored in a refrigerator. In the event of unsupervised intrusion, such as by police, firefighting personnel, or by burglary, such incident will be documented on the chain-of-custody form. Clients will be notified by phone and in writing.

4.4 Sample Retention and Disposal

There are no established holding times for soil-gas samples collected on adsorbent cartridges; however, because the medium is an adsorbent, it can be held for more than 28 days without any demonstrated loss, as verified through holding times studies. As standard practice to meet client turn-around time requirements, samples are analyzed typically within five (5) days of sample receipt. BEACON's sampler design allows for a secondary adsorbent cartridge to be included in each sampler. That secondary cartridge may be held for confirmatory or duplicate analysis according to contractual obligations. Following analysis of the passive soil-gas samples, adsorbent cartridges are reconditioned for use in a future project or disposed of if they no longer meet quality control standards.

4.5 Document Control

BEACON's goal of the document control program is to assure that all documents for the project are accounted for when the project is completed. Accountable documents include chain-of-custody records, sample receipt logbooks, instrument logbooks, field notes, and other documents that may relate to the collection of samples and sample analyses.

4.5.1 Recordkeeping

All data entries are made in indelible, water-resistant ink. The date of the entry and the observer is clear on each entry. The observer uses his or her full name or initials. All information is recorded in a notebook or on other records at the time the observations are made. Recording information on loose pieces of paper is not allowed. When a mistake is made, the wrong entry is crossed out with a single line, initialed and dated by the person making the entry, and the correct information is recorded. Obliterating or writing over an incorrect entry is not allowed, nor is the use of correction tape or fluid on any laboratory records. Each page in logbooks is sequentially numbered and shows the laboratory name.

4.5.2 Laboratory Records

Following are some of the records that are used to document activities in the laboratory. These are in addition to the documents discussed elsewhere is this QA manual.

Project File

The project file is a folder that is established when the project is initiated. The project file is labeled with the project number, project name and location, and client name. The chain-of-custody forms, field notes, correspondence, instrument printouts, and a copy of the final report are placed in the project file.

Instrument Run Log

An instrument run log that records daily observations and operations is maintained for each major analytical instrument. The instrument log contains the following: (i) identification of all analyses made in sequence (including those analyses or runs that are not acceptable or project related), (ii) the autosampler sequence, if applicable, (iii) date, (iv) the project number, (v) analyst, (vi) maintenance notes, and (vii) a description of any corrective action taken.

Refrigerator/Freezer Logs

The maintenance and documentation of the operating temperatures of refrigerators and freezers are recorded in logs for each piece of equipment. Proper storage of samples and standards in refrigerators and freezers is critical to maintenance of their integrity. Each page of the logs is numbered sequentially and provides descriptive information on the piece of equipment. The following information is recorded on a daily basis: (i) date, (ii) analyst, (iii) temperature, and (iv) any corrective action required.

Conditioning Oven Log

The maintenance and documentation of the operating temperature and performance of the conditioning oven are recorded in the conditioning oven log. The temperature, gas flow, and gas leak checks are recorded in the log when adsorbent samplers are conditioned to assure that the adsorbent samplers are properly conditioned. Each page of the conditioning oven log is numbered sequentially and provides descriptive information on the oven. The following information is recorded in the log when adsorbent samplers are conditioned: (i) date and time when conditioning starts and ends, (ii) the project number, (iii) adsorbent material, (iv) oven set temperature, (v) gas flow rate, (vi) temperature check, (vii) gas leak and flow check, (viii) analyst, and (ix) any corrective or maintenance action required.

5.0 CALIBRATION PROCEDURES AND FREQUENCY

5.1 Calibration Program

A formal calibration program controls instruments and equipment used by BEACON. The program verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. All instruments and equipment that measure a quantity, or whose performance is expected at a stated level, are subject to calibration. Chemical calibration or standardization, which refers to those operations in which instrument response is related to analyte mass, is discussed in this section.

5.2 Chemical Calibration

Chemical calibrations consist of initial and continuing calibrations, which are discussed in subsequent sections. The calibration criteria are based on those given in EPA Method 8260C and as described in the Solid Waste Manual (SW-846), as well as U. S. EPA Method TO-1 and TO-17 as described in the Compendium for Methods for the Determination of Toxic Organic Compounds in Ambient Air (EPA/600/4/89/017). Chemical calibrations are documented and stored in computer files as well as on hard-copy printouts.

5.2.1 Initial Calibration

The initial multi-point calibration consists of the establishment of a calibration or standard curve, which associates instrument response and analyte mass or concentration. The curve is constructed by measuring the responses of a series of spiked adsorbent cartridges. Calibration factors (CFs) are calculated using the internal calibration technique. The percent relative standard deviation of the CFs must be below 30% for the curve to be assumed linear and valid. The percent relative standard deviation (RSD) is calculated by dividing the standard deviation for the CF by the mean of the CFs and multiplying by 100. The low-point calibration mass is below the reporting limit to ensure accuracy and eliminate reporting false negatives.

5.2.2 Continuing Calibration

The initial calibration curve and tuning criteria is verified at the beginning of each analytical sequence for all GC/MS methods. An analytical sequence, including the initial calibration, continuing calibration, method blank, and sample analyses, will not exceed 24 hours. The criterion for the acceptance of the continuing calibration is based on the percent difference between the calculated mass injected and the value obtained by the initial calibration comparison. For GC/MS analysis, certain System Performance Check Compound (SPCC) and Calibration Check Compound (CCC) criteria must be met prior to sample analysis. The target compound value obtained during the continuing calibration must be within ±30% of the initial calibration for the calibration to be verified valid. If the continuing calibration varies more than ±30% then corrective action must be taken to restore the system and a new calibration verification run or a new calibration curve must be prepared before any more samples are run.

5.2.3 GC/MS Tuning and Mass Calibration

When analyzing samples with GC/MS instrumentation, it is necessary to demonstrate that the GC/MS meets the standard mass spectral abundance criteria prior to data collection. The method specific criteria must be demonstrated prior to any standards, blanks, or samples being analyzed and for each 24-hour analytical sequence.

5.2.4 Analytical Standards

Analytical standards are purchased from ISO 9001 registered, and NIST-NVLAP accredited suppliers. All identifying paperwork accompanying standards purchased are kept in a standards log that include (i) name of standard, (ii) supplier of standard, (iii) date received, (iv) date of expiration, and (v) concentration of standard. All standards are stored at or below 4° Celsius in standard vial sealers. Information is listed on each vial identifying the standard, the concentration of the standard, and the expiration date. All dilutions of standards performed at BEACON to prepare specific concentrations are documented in the Standard Preparation Log, which records: (i) date of preparation, (ii) analyst, (iii) lot number, concentration, and supplier, (iv) volume and type of solvent, (v) concentration of final standard, and (vi) any necessary comments. The prepared standards are labeled with the date and concentration and stored at or below 4° Celsius in sealed amber glass vials.

6.0 ANALYTICAL PROCEDURES

6.1 Analytical Method

The analytical methods followed are based on U. S. EPA Methods 8260C, 8270 as described in the Solid Waste Manual (SW-846), as well as U. S. EPA Method TO-17, and TO-1 as described in the Compendium for Methods for the Determination of Toxic Organic Compounds in Ambient Air (EPA/625/R-96/010b). Methods 8260C and 8270 have been modified for thermal desorption of adsorbent cartridges to screen sites for the targeted compounds. A summary of each of the analytical methods with quality assurance procedures and acceptance criteria specific to each method is provided under separate cover.

Sample matrices are adsorbed vapors. All analytical methods used by BEACON have the same procedure for sample introduction, which is thermal desorption. The thermal desorber heats sample cartridges in a helium atmosphere to temperatures ranging from 200° to 350° C, the actual temperature dependent on the adsorbent used. To check for contamination in the system, a system or method blank is run at the beginning of an analytical batch, as well as after any high-level detection that results in potential carry over. If necessary, blanks are run until the system is clean and, if needed, the system is conditioned until the baseline of the respective detector is stable. A blank is run following the conditioning to check for cleanliness of the system. A blank is run after the daily continuing calibration.

If the sample response of any target compound falls outside the calibration range, the concentration of the compound will be estimated and reported as such. However, the design of the passive soil-gas sampler with two adsorbent cartridges enables the analyst to perform a second analysis from each location where the analytical acceptance criteria does not initially pass.

6.2 Instrumentation

The analytical methods are performed with gas chromatographs connected to mass spectrometers (GC/MS). All GC/MS analytical systems are equipped with thermal desorption sample introductory systems for analysis. The analytical instruments employed for each method are described in the method specific procedures, which are provided under separate cover.

6.3 Detection Limits

Method detection limits (MDL) are determined using the U.S. EPA procedure published in 40 CFR Part 136, Appendix B. The 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." This procedure requires that "all sample processing steps of the analytical method be included in the determination of the method detection limit." MDLs therefore are influenced by the sample matrix and sample preparation process as well as the analytical instrumentation.

A minimum of seven replicates spiked at one to five times the expected MDL are analyzed. The MDL is calculated by multiplying the standard deviation for n replicates by the one-tailed Student's t-value for n-1 degrees of freedom and a 99% confidence level. On average the MDL for target individual analytes is less than five nanograms per adsorbent sampler. Because the definition of an MDL is concerned only with detection of an analyte and not its accurate quantitation, MDLs are not used in reporting data. Instead, data are reported using reporting limits, which are levels above that which reliable quantitative results can be obtained. The reporting limit used for the method analytes is 25 nanograms per adsorbent sampler. MDL studies are performed periodically to ensure that the values are at least five times less than the reporting limits.

When the measured mass of an analyte is below its reporting limit but the instruments response meets the identification criteria for the method, the measured mass is reported with a "J" (estimated) flag. If the analyte is not detected or the identification criteria are not met, the reporting limit is shown (*e.g.*, <25).

7.0 DATA REDUCTION, VALIDATION, AND REPORTING

The procedures for data handling followed in the laboratory are an important part of the laboratory quality assurance program. BEACON treats all records and project data as client confidential. Client information will not be shown to anyone outside BEACON without the client's approval.

7.1 Data Collection

For all analyses, the raw data are collected by the associated computer system and software and sorted in data files. The raw data are handled such that manual transcriptions are avoided to as great an extent as possible. The standards chosen for the methods contain the analytes to be targeted on the adsorbent cartridges. After the analyst has fully quantified the data, it is electronically transcribed to a PC-based database for further processing.

7.2 Data Reduction

Data reduction includes all processes that change either the reporting of values or numbers of data items. The data reduction processes used in the laboratory include establishment of calibration curves, calculation of sample concentrations from instrument responses, and computation of quality control parameters. The masses of analytes present on the adsorbent cartridges are determined, using the calibration function (CF) from the continuing calibration verification (Section 5.2.2), with the analytical system specific software program.

7.3 Data Validation

Data validation is a systematic process of reviewing data against a set of criteria to identify outliers or errors and to delete suspect values or to flag them for the user. Laboratory data review starts with the analyst and the laboratory quality control procedures discussed in Section 9.0. The analyst reviews the quality control data as the data are generated against the method specific criteria and takes the specified corrective action when the data are out of control. After analyses and data workup are complete, the analyst checks the data for errors in transcriptions and calculations.

The Laboratory Director is responsible for final validation of the data. Validation starts with verifying that the required quality control procedures were effectively in place and followed by the analyst, and that the generated documentation shows that samples, data, and analytical results were acquired and processed in a controlled and traceable manner.

The following aspects affecting data quality are checked in manual and computerized fashion before data entry and issuance of the final report: (i) the initial and continuing calibration verifications must meet method specific criteria and (ii) the method blanks must be devoid of all targeted compounds at quantities greater than the reporting limits. Any sample sets or data that are judged unacceptable are identified and the appropriate corrective action measures initiated. Data qualifiers are assigned to all applicable data.

Data validation also encompasses a review of (i) proper chain of custody sample login and sample handling procedures, (ii) holding times, (iii) efficiency of the preparation of adsorbent samplers prior to shipment (evaluated by analysis of a preparation blank), (iv) the method used to analyze samples, (v) internal standard areas and surrogate standard recoveries, (vi) blank system checks, (vii) calculations to verify and ensure computations performed correctly, (viii) transcription of raw and final data, and (ix) detection and reporting limits.

7.4 Reporting

Tabular laboratory report forms are generated from the data stored in the computer database and are placed in the project file. All sample identifications, calculations, and final report contents shall be manually reviewed prior to issuance of the final report. Project specific quality control objectives determine the reporting format, which can include the following: (i) narrative, (ii) tabular results of sample analysis, (iii) tabular results of method blank analysis, (iv) tabular results of continuing calibration verifications, (v) chromatograms of samples, method blanks, and calibration verifications, and (vi) raw quantitation data. CLP-equivalent data packages are available, upon request, including Forms 1, 2, 4, 5, 6, 7, and 8.

7.5 Data Storage

The project files containing all client data are placed in the central files numerically according to the project number. All project files are maintained securely within BEACON's corporate office, which has a monitored alarm system and is locked at all times when BEACON personnel are not present. Unless superseded by program, project, or client specific requirements, the disposal date of the archived files is five years from the archive date. Unless otherwise specified by the client, electronic data are maintained by BEACON for a period of two years. This guidance is consistent with the Good Laboratory Practices in 21 CFR Part 58 and Good Manufacturing Practices in 21 CFR Part 820. The Laboratory Director is responsible for ensuring that all electronic data are stored to prevent deterioration and that records are maintained identifying the storage drive, archive date, and discard date.

8.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

This section describes the quality control procedures that will be followed during sample analysis, including analysis of quality control samples. The quality control requirements with frequencies and acceptance criteria are as follows: (i) a multi-point calibration is performed initially, and as required, and is accepted if the percent relative standard deviation (RSD) for the CFs over that range is less than 30%, (ii) calibration verification is performed according to method specific criteria (which are provided under separate cover), and (iii) method blank checks are performed at the beginning of sample batch analysis, plus after analysis of a high level sample, and are repeated until the system is confirmed clean. The results of an analysis that fails a QC criterion will be reported and flagged to indicate the problem.

8.1 Calibration

The criteria and frequencies for initial and continuing calibrations, as well as system tunes, are discussed in Section 5.0.

8.2 Blanks

A method blank is performed by analyzing a conditioned adsorbent cartridge by thermal desorption. This checks for system cleanliness. One or more trip blanks, which are cartridges prepared, transported, and analyzed with field samples but intentionally not exposed, are also analyzed with each project.

8.3 Internal Standards (IS)

Internal standards are organic compounds that are similar to the analytes of interest in chemical composition, extraction, and chromatography, but are not normally found in environmental samples and are used for internal calibration. For analyses following EPA Method 8260C, quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve. Internal standards are spiked onto all standards, blanks, and samples. If retention time of an IS changes by more than 30 seconds or the response of an IS changes by a factor of two (-50% to +100%) from that in the daily continuing calibration check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When used as a diagnostic tool to monitor retention times and responses (area counts) in all samples, spikes, blanks, and standards, internal standards effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance. Sample results are flagged when the IS retention time and/or response do not fall within the method specific control limits. When IS changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration, method variables includings flow and temperatures, and system cleanliness are checked and retuning and recalibration and detector cleaning are performed if needed. For analyses following TO-17, the IS must fall within ±40% to pass acceptance criteria.

8.4 Surrogates

Surrogates are organic compounds that are similar to the analytes of interest in chemical composition, extraction, and chromatography, but are not normally found in environmental samples. For analysis of samples by GC/MS methods, surrogate compounds are spiked onto all standards, blanks, and samples in

order to monitor the analysis of the samples. The mass of each surrogate is quantitatively determined in each analytical run and percent recoveries are calculated. Surrogate recoveries are monitored for analysis performance. For example, in the case of high petroleum contamination on samples the bromofluorobenzene recoveries normally exceed recoveries requiring that sample to be closely analyzed for false negatives and false positives as well as ion masking.

8.4 Duplicates

Duplicates are a pair of subsamples of a field sample that are taken through the entire preparation and analysis process to estimate the precision of the method. Because only one analysis of each adsorbent cartridge is possible, sample duplicates cannot be analyzed. However, two adsorbent cartridges are contained within each sampler; therefore, two samples can be analyzed from each location as a sample location duplicate.

8.5 General Laboratory Controls

In addition to instrument calibration and the analysis of quality control samples, the following controls will be implemented: (i) reagents and solvents will be certified (ii) reagent storage environment and duration will meet EPA guidelines, (iii) regular laboratory screening will be performed to assure a clean air environment, (iv) volumetric measurements will be made with certified glassware and recommended syringes, (v) data reduction computations will be independently checked, and (vi) only fully trained personnel will perform laboratory analyses.

9.0 PERFORMANCE AND SYSTEM AUDITS

The Laboratory Director maintains and summarizes the performance of the systems at BEACON. Continual calibration and MDL studies are ongoing. Each laboratory procedure affecting data quality and validity will be reviewed periodically to assure performance. Performance evaluation (PE) samples are not available for the analysis of soil-gas samples passively collected on an adsorbent media.

10.0 PREVENTATIVE MAINTENANCE PROCEDURES

Periodic preventative maintenance is required for all sensitive equipment. Instrument manuals will be kept on file for reference if equipment needs repair. The troubleshooting section of factory manuals may be used in assisting personnel in performing maintenance tasks. The individual responsible for the instrument documents maintenance in the laboratory analytical log.

Replacement parts are kept on hand to minimize down time. Table 10.1 lists specific maintenance procedures followed to ensure the consistent performance of the instruments and equipment.

Table 10.1 Preventive Maintenance Procedures

Equipment	Action	Frequency
Gas Chromatograph	Flows and pressures checked	Prior to and during analysis
	Unions checked for leaks	Prior to and during analysis
	Gas line filters replaced	Color indicating
	Detectors cleaned	As needed
	Relays replaced	As needed
Markes UltrA-Unity Thermal Desorber	Software checks for leaks	Prior to each analysis
	Replace worn O-ring	After leaks are determined by software
	Set the trap flow	As the analytical method requires
	Check for trap contamination	Each analysis
	Replace trap	When needed
	Replace purge gas	As needed
Gas Chromatograph/Mass Spectrometer	Tune MSD	As needed
	Check the calibration vial	Every six months
	Check the foreline pump oil	Weekly or when discolored
	Replace the foreline pump oil	Every six months or as needed
	Clean the ion source	When performance deteriorates
	Check for leaks	Prior to analysis
Conditioning Oven	Flows and pressures checked	Prior to and during conditioning
	Unions checked for leaks	Prior to and during conditioning
Refrigerators	Temperatures checked and logged	Each work day
	Defrosted and cleaned	Quarterly
Analytical Balances	Balanced checked	Prior to use
	Zero checked	Prior to use
	Deflection checked	Prior to use
	100 mg calibration mass checked	Prior to use
	If any checks fail, balance maintenance performed	

11.0 STATISTICAL ASSESSMENT OF DATA QUALITY

The statistical tests necessary to verify proper analytical function are performed as soon as practical after the measurements on which they are based are available. The results of the tests are compared with the control limits to determine if the data can be used. If the limits are exceeded, the Laboratory Director is notified and a decision is made concerning the appropriate action to be taken.

11.1 Calculation of Precision

Precision is the mutual agreement among individual measurements of the same property, usually under similar conditions. Precision can be expressed in several different ways, such as standard deviation, relative standard deviation, relative percent difference, and range.

Standard Deviation

The standard deviation measures the dispersion of replicate values about their mean.

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{n-1}}$$

where:

- s = standard deviation
- x_i = replicate values
- $\overline{x}$ = mean of replicate values
- n = number of replicate runs

Relative Standard Deviation

$$RSD = \frac{s}{\overline{x}}$$

The relative standard deviation (RSD) is used for replicate measurements and is the ratio of the standard deviation of the measurements to their mean.

where:

- s = standard deviation of replicate run values
- $\overline{x}$ = mean of the replicate run values

Relative Percent Difference

The relative percent difference (RPD) is used for duplicate measurements and is calculated by dividing the difference between the values by their mean and multiplying by 100.

$$RPD = \frac{|x_1 - x_2|}{\frac{(x_1 + x_2)}{2}} \times 100$$

where x_1 and x_2 are the duplicate/replicate values

Percent Difference

The percent difference (%D) is a measure of the difference between a reference value and a measured one.

$$\%D = \frac{|r - x|}{r} \times 100$$

where:
- r = reference value
- x = measured value

11.2 Calculation Of Accuracy

Accuracy is the degree of agreement of a measured value with the true or expected value or the measured quantity. The accuracy of control sample measurements is generally expressed as a percent recovery. For samples without a background level of the analyte, such as reference materials, laboratory control samples, and performance evaluation samples, the percent recovery (%R) is calculated from:

$$\%R = \frac{X}{T} \times 100$$

where:
- X = the found concentration
- T = the true or assumed concentration.

The percent recovery for measurements in which a known amount of analyte (a spike) is added to an environmental sample (matrix spike) is calculated from:

$$\%R = \frac{X - B}{T} \times 100$$

where:

X = the found concentration
B = the background concentration
T = the true or assumed concentration.

12.0 NONCOMFORMANCE AND CORRECTIVE ACTION PROCEDURES

Action taken to improve improper performance of instruments and analytical systems must be scrupulously documented. In addition, care must be taken that appropriate personnel are alerted to conditions that may affect data quality.

12.1 Analytical Systems

Any corrective action taken to adjust or maintain the instrument will be recorded in the laboratory analytical log as appropriate. The following actions require performing an initial calibration: (i) detector cleaning or replacement, (ii) chromatographic column replacement, (iii) changing any pressure regulator or temperature regulator setting, and (iv) failure of continuing calibration verifications to meet criteria. The Laboratory Director and any other analyst will be informed when initial calibration is required. Any unusual difficulty encountered in calibration will be brought to the attention of each analyst, the Laboratory Director, and the President.

12.2 Samples, Sample Receipt, and Chain-of-Custody Documents

Any discrepancies noted in sample container labeling or sample chain-of-custody documents are reported to the client immediately, and noted on both the chain-of-custody document and sample receipt log.

12.3 General Laboratory Equipment

The monitoring of individual laboratory units and the recording of observations in the log reserved for the individual unit will often indicate actual or impending malfunction. Observations of possible malfunction will be immediately reported to the Laboratory Director or his designated substitute. Equipment found to be nonfunctional shall be conspicuously labeled as such. Repair and/or replacement action taken will be documented in the log/file designated for that unit.

12.4 Laboratory Reports and Documentation

In the event that an error or errors are found in previously transcribed data, the Laboratory Director will be notified immediately. In the case of previously transmitted reports, any errors found will be immediately communicated to the client. All corrected reports will be conspicuously labeled as "AMENDED" and include the signature of the authorizing party and date.

12.5 Performance Sample Evaluations

The results of any performance sample evaluations are made immediately available to each participating analyst and supervisor. Any serious deviation from expected or true values constitutes cause for immediate corrective action.

13.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Fundamental to the success of this QAPP is the active participation of management in the project. Because of the small size of BEACON, the management is constantly aware of project activities and actively participates in development, review, and operation of the project. No formal reports are anticipated.

Quality Systems Manual

For

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March 15, 2010

This document has been reviewed and approved by the following:

Karl Olm
Operations Manager — 03/16/10 Date

Michael Linskens
Quality Manager — 03/16/10 Date

Nicholas Nigro
President — 03/16/10 Date

ACRONYMS

A list of acronyms used in this document and their definitions are:

AgChem	–	Agricultural chemical
AIHA	–	American Industrial Hygiene Association
ANSI	–	American National Standards Institute
ASTM	–	American Society for Testing and Materials
Blk	–	Blank
°C	–	Degrees Celsius
CAF	-	Corrective action form
cal	–	Calibration
CAS	–	Chemical Abstract Service
CCV	–	Continuing calibration verification
COC	–	Chain of custody
DOC	–	Demonstration of capability
DoD	–	Department of Defense
ECCS	–	Environmental Chemistry Consulting Services, Inc.
EPA	–	Environmental Protection Agency
g/L	–	Grams per liter
GC/MS	–	Gas chromatography/mass spectrometry
HPLC	–	High performance liquid chromatography
ICP-MS	–	Inductively coupled plasma-mass spectrometry
ICal	–	Initial calibration
ICV	–	Initial calibration verification
DOC	–	Demonstration of capability
ISE	–	Ion selective electrode
lb/in^2	–	Pound per square inch
LCS	–	Laboratory control sample
LCS-CL	–	LCS control limit
LIMS	–	Laboratory information management system
LOD	–	Limit of detection
LOQ	–	Limit of quantitation
MDL	–	Method detection limit
mg/kg	–	Milligrams per kilogram
mg/L	–	Milligrams per liter
MS	–	Matrix spike
MSD	–	Matrix spike duplicate
µg/L	–	Micrograms per liter
µg/kg	–	Micrograms per kilogram
NELAC	–	National Environmental Laboratory Accreditation Conference
NELAP	–	National Environmental Laboratory Accreditation Program
NIST	–	National Institute of Standards and Technology
OM	–	Operations manager

PCB	–	Polychlorinated biphenyl
PT	–	Proficiency test(ing)
PQL	–	Practical quantitation limit
QA	–	Quality assurance
QAPP	–	Quality assurance project plan
QC	–	Quality control
QM	–	Quality manager
QS	–	Quality system
QSM	–	Quality system manual
RL	–	Reporting limit
RPD	–	Relative percent difference
RSD	–	Relative standard deviation
SAP	–	Sampling and analysis plan
SDWA	–	Safe Drinking Water Act
SI units	–	SI base units, NIST Special Publication 330 (SP 330), The International System of Units (SI).
SOP	–	Standard operating procedure
TNI	–	The NELAC Institute
VOC	–	Volatile organic compound

1.0 INTRODUCTION

The Environmental Chemistry Consulting Services, Inc. (ECCS) quality system manual (QSM) codifies our quality system (QS), with the goal of enduring that ECCS provides the highest quality laboratory services to its clients. This introduction includes the QSM applicability, a brief overview of the ECCS organization, and our mission statement.

1.1 Applicability

This QSM is applicable to the operation and management of the QS for ECCS. The QSM applies seamlessly across on-site and fixed facility laboratory services operations. On-site laboratory services encompass those provided in temporary constructed facilities for longer term projects, and those provided in mobile laboratories for typically shorter duration projects. The QSM defines the policies, procedures, and documentation that assure analytical services continually meet a defined standard of quality that is designed to provide clients with data of known and documented quality and, where applicable, demonstrate regulatory compliance. This manual can and should be supplemented by project-specific requirements.

Policy
This QSM provides for the operation and management of the QS for ECCS' on-site and fixed facility laboratory services operations.

1.2 Company

In its simplest form, ECCS provides three partially integrated services to the environmental consulting and engineering community: (1) on-site and fixed facility laboratory services, (2) Safe Drinking Water Act (SDWA) compliance monitoring, and (3) chemistry consulting services. Over the years, a unique business model has been developed that effectively shares staff between its service areas. The on-site and fixed facility laboratory operations share resources and systems to effectively operate the overall business as a single profit center. All company personnel are shared between on-site and fixed facility laboratory operations. Normal business support operations such as sales and marketing, client services, human resources, health and safety, quality assurance (QA), purchasing, reporting, filing, and archiving serve both on-site and fixed facility laboratory operations under a single management system. This sharing concept allows our on-site and fixed facility laboratory services to be seamless from an operations and a QS standpoint. This QSM applies to the on-site and fixed facility laboratory services operations only and not SDWA compliance monitoring or chemistry consulting services.

The business model requires a special company culture for success. Employees (chemists) must be experienced self starters, skilled multi-taskers, and most importantly, consummate client services representatives. As such, the owners have strived to create a culture with core values that have empowered and trusted employees to treat the company as their own. The core values listed below have nourished the growth and success of the company to date and will continue to lead us to future growth and success:

- Empower and trust employees to treat the company as their own
- Be there to provide 110% support to fellow team members
- Deliver quality analytical services that are unique and meet or exceed the needs of our clients and the regulatory community
- Embrace the concept of educating our clients and regulators to create marketplace
- Realize the client's specific needs are paramount and above ours
- Believe that our reputation is everything
- Maintain growth and profitability to allow employees to have opportunity and be rewarded for their efforts, performance, and individual or team results
- Assure competitive (or better) salaries and benefits
- Strive for excellence and continual improvement.

1.3 Mission

Our on-site and fixed facility laboratory services mission is to provide faster, better, and cheaper solutions to our clients' project-specific analytical needs. We use our knowledge and experience to translate Environmental Protection Agency (EPA) test methods into project specific analytical solutions that can be effectively applied in an on-site laboratory environment, that are more appropriate to the project specific analytical needs, and that are cost-effective.

2.0 SCOPE OF DOCUMENT

This document provides for the policies and procedures required to successfully analyze for a wide range of environmental contaminants using standard, non-standard, and internally developed methods in ECCS' on-site and fixed facility laboratory operations.

2.1 Scope of Analytical Testing

Appendix A provides a list of the current ECCS test methods. Any newly developed test method not listed in Appendix A must follow the requirements of this QSM.

2.2 Client Expectations

This document provides for the policies and procedures required to meet or exceed the expectations of our clients and the regulatory community.

2.3 Table of Contents and Appendices

The organization of the document generally follows the outline of "2003 NELAC Standard, Chapter 5.0, Quality Systems."

2.4 Acronyms

Acronyms are used throughout the document. A listing of acronyms and their associated definition are provided after the table of contents.

3.0 ORGANIZATION

ECCS is a legally identifiable organization. Through application of the policies and procedures outlined in this manual, ECCS assures that it is impartial and that personnel are free from undue commercial, financial, or other pressures that might influence their technical judgment. ECCS is responsible for carrying out on-site and fixed facility laboratory services activities that meet the needs of the client, the requirements of various regulatory agencies and Codes, and other accrediting authorities.

3.1 Overview

ECCS is a small commercial laboratory business that operates on-site and fixed facility laboratory services operations from facilities located at 2525 Advance Road in Madison, Wisconsin. The fixed laboratory facility provides analytical services, primarily pesticide residue testing services, for the AgChem marketplace. ECCS on-site laboratory services are provided from its fleet of 10 mobile laboratories, and from custom on-site facilities designed to meet our client-specific analytical testing needs.

3.2 ECCS Formal Management Structure

ECCS formal management structure includes the titles president, operations manager (OM), and quality manager (QM). A standing operations team also exists that includes the formal managers listed above plus other key staff including client services, technical, and office/human resource (HR) personnel.

3.3 Lead Chemist

The Lead Chemist role is the operational centerpiece to a successful, high quality on-site laboratory project. Senior chemists are authorized by management to accept the role of Lead Chemist to be responsible for each on-site testing project that occurs. The diagram in Appendix B depicts the relationship of various operational sectors and the Lead Chemist during the execution of an on-site laboratory services project.

3.4 Organizational Chart

An organizational chart provided in Appendix C is current as of March 1, 2010. The assignment of responsibilities, authorities, and interrelationships of the personnel who manage, perform, or verify work affecting the quality of environmental tests is documented in Section 16 of this manual.

Policy
Management has overall responsibility for the technical operations and authority needed to generate the required level of quality for its laboratory operations.

Policy

Management's commitment to quality and to the QS is stated in the Quality Policy (See Section 4.0), which is upheld through the application of related policies and procedures.

Policy

Management ensures technical competence of personnel operating equipment, performing tests, evaluating results, or signing reports, and limits authority to perform laboratory functions to those appropriately trained and/or supervised.

3.5 Maintenance of the Quality System

Management bears specific responsibility for the QS. This includes defining roles and responsibilities to personnel, approving documents, providing required training, providing a procedure for confidential reporting of data integrity issues, and periodic review of data, policies and procedures, and documentation practices.

3.6 Audit Findings and Corrective Actions

Management ensures that audit findings and corrective actions are completed within required time frames.

3.7 Designated Alternates

Designated alternates are appointed by management during the absence of key management staff if the absence is more than 15 days.

3.8 Position Descriptions

Management has defined the minimum level of education, qualifications, experience, and skills necessary for all positions in the laboratory with position descriptions (See Section 16).

3.9 Training

Training is kept up to date as described in Section 18.

4.0 QUALITY SYSTEM

Quality Policy Statement

The objective of the QS and the commitment of management are to consistently provide our clients with data of known and documented quality that first and foremost meets the specific needs of their projects. Our policy is to use good professional practices, to continually improve quality, to provide the highest quality of service to our clients, and to comply with the various regulatory and accrediting authority Standards when they apply. This policy is implemented and enforced through the unequivocal commitment of management to the principles, practices, and ethics policies and procedures outlined in this manual.

4.1 Quality Systems

The company's QS are documented in this QSM and associated documents. Together they describe the policies, procedures, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of the organization for ensuring quality in its work processes, products, and services. The QSM is maintained current and up-to-date by the QM.

4.2 Quality Manager

The QM serves as the focal point for implementation of the QS described in this QSM. The QM has the authority and/or responsibility to:

- be the focal point for the QS and have oversight of quality control data;
- implement, maintain, and improve the QS;
- keep the QSM current;
- ensure that all staff understand their contribution to the QS;
- evaluate data objectively and perform assessments without outside influence;
- conduct internal audits on all QS at least annually;
- be knowledgeable about various regulatory requirements including those of accrediting authorities;
- notify management and staff of audit findings and corrective actions; and
- evaluate the effectiveness of training.

Policy

Management ensures that the laboratory's policies and objectives for quality are documented by reference or by inclusion in the QSM, and that the QSM is communicated to, understood by, and implemented by all personnel concerned.

Policy

Where the QSM documents laboratory requirements, a separate SOP or policy is not required.

Procedure

All employees sign a form, kept with their training records, that states that they have read and understand the current version of the QSM, including the Quality Policy Statement. FORM-GEN-009-4 of SOP "GEN-009, Documentation of Training" is used to acknowledge that employees have read and understand quality related documents. Changes to the QS will sometimes be sent to employees via e-mail. This is especially true for employees that are assigned to on-site laboratory projects. In these instances, a return e-mail is used as documentation in lieu of FORM-GEN-009-4.

5.0 DOCUMENT MANAGEMENT

This section describes procedures for document management, which includes controlling, distributing, reviewing, and accepting modifications. The purpose of document management is to preclude the use of invalid and/or obsolete documents.

Policy
All documents that affect the quality of laboratory data are managed appropriate to the scope and depth required.

Policy
The QSM and controlled standard operating procedures (SOP) are reviewed, and updated if necessary, on at least an annual basis.

5.1 Types of Documents

The laboratory manages four types of documents; controlled, approved, withdrawn and obsolete.

- A controlled document is one that is issued, tracked, and uniquely identified as part of the current QS. Controlled documents may be internal documents or external documents.
- An approved document is one that has been reviewed, and either signed and dated, acknowledged in writing, or secure by electronic means by the issuing authority.
- A withdrawn document i**s** one that has become obsolete or has been superseded by more recent versions. A withdrawn document is stamped "withdrawn."
- Obsolete documents are out of date and removed from general distribution, or otherwise prevented from unintended use. An obsolete document is stamped "withdrawn."

Policy

Controlled documents are identified, revised, distributed, implemented, and archived according to SOP "GEN-001, Document Control / Preparation of Non-Method SOPs." This SOP codifies the header requirements for all ECCS SOPs and the formatting of non-analytical method SOPs. The formatting of analytical method SOPs is addressed in SOP "GEN-002, ECCS Analytical Method Standard Operating Procedure (SOP) Creation and Revision Guidance."

Policy

Controlled copies of documents are available at all locations where operations are essential to the effective functions of the laboratory.

Policy

Changes to SOPs are incorporated into a new revision and reissued as soon as practical. Revision of SOPs is addressed in SOP "GEN-001, Document Control / Preparation of Non-

Method SOPs."

Policy

Handwritten changes to controlled documents are not allowed, except by the QM. Hand written notes for suggested changes to the next version can be made to the original document in possession of the QM. Any change must be initialed and dated.

Procedure

A master list of controlled documents that includes distribution, location, and revision dates is maintained by the QM. The controlled document list is updated each time documents are distributed or when a handwritten change is made by the QM.

Procedure

Archival of documents is addressed in SOP "GEN-001, Document Control / Preparation of Non-Method SOPs."

5.2 Electronic Document Changes

Policy
Electronic changes to documents are tracked where practicable.

Procedure
The laboratory information management system (LIMS) provides a complete audit trail to any electronic change made.

Procedure
Changes to the QSM, SOPs and associated quality documents are electronically tracked and archived where practicable.

Procedure
Archival of documents is addressed in SOP "GEN-001, Document Control / Preparation of Non-Method SOPs."

5.3 Standard Operating Procedures

SOPs are used to ensure consistency of application of common procedures, are written procedures that describe in detail how to accurately reproduce laboratory processes, and are of two types: 1) test method SOPs, which have specifically required details, and 2) general use SOPs which document the more general organizational procedures.

Policy
Copies of all SOPs are accessible to all personnel.

Procedure
Each SOP indicates the effective date, the revision number, and the signature(s) of at least the President, OM, and QM. The QM, or his designee, shall maintain a record of all ECCS SOPs.

5.3.1 Test Method SOPs

Policy
The laboratory has SOPs for all test methods within its scope, and for procedures that are part of the QS that accurately reflect how the analytical process is performed. All master copies of SOPs are maintained in the QM's office. Where equipment manuals or published methods accurately reflect laboratory procedures in detail, a separate SOP is not required.

Policy
ECCS' core business often requires modifications to standard operating procedures (SOP) to meet project and client specific objective. Any deviation from a test method is documented, including both a description of the change made and a technical justification.

Procedure
The deviation is recorded on a corrective action form. The deviation from a test method is reported to the client in the sample narrative.

Procedure
SOP "GEN-002, ECCS Analytical Method Standard Operating Procedure (SOP) Creation and Revision Guidance" provides the procedure for creation and content of analytical method SOPs.

5.3.2 General Use SOPs

Policy
The laboratory maintains general use SOPs for all essential activities to support analytical testing in the laboratory.

Policy
Deviations from general use SOPs requires documentation, however, the documentation does not have to be on a corrective action form, unless the use of a corrective action form is required by an applicable SOP.

Procedure
Deviations from general use SOPs are documented on the chain of custody, in LIMS, or in field logbooks.

6.0 REVIEW OF REQUESTS, TENDERS, AND CONTRACTS

Policy
The review of all new work assures that oversight is provided so that requirements are clearly defined, and the organization has adequate resources, capability, and test methods applicable to the customer's needs. This process assures that all work will be given adequate attention to avoid pitfalls that may compromise data quality.

Procedure for the Review of Work Requests – Fixed Facility
The OM determines if the laboratory has the necessary accreditations, resources, including schedule, equipment, deliverables, and personnel to meet the work request.

The project assistant (PA) or the project manager (PM) informs the client if the review of the work request indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily.

The client is informed of any deviation from the work request including the test method or sample handling processes. All differences between the request and the final contract are resolved and recorded before any work begins. It is necessary that the work request be acceptable to both the laboratory and the client.

The review process is repeated when there are amendments to the original contract by the client. Project personnel are given copies of the amendments if the amendment is applicable to their specific task. Copies of all amendments are maintained in the project file.

Procedure for the Review of Work Requests – On-Site Laboratories
The procedure for review of on-site laboratory work follows the procedure listed above in Section 6. Further definition is provided by SOP "GEN-013, On-site Testing."

7.0 SUBCONTRACT LABORATORY

A subcontract laboratory is defined as a laboratory external to this organization that performs analyses for the organization.

Policy
When subcontracting analytical services, the laboratory assures work requiring accreditation is placed with an appropriately accredited laboratory or one that meets applicable statutory and regulatory requirements for performing the tests.

Procedure
A list of subcontractors with a copy of the accreditation certificate is maintained as evidence of compliance.

7.1 Client Notifications

When possible, the PM notifies the client of the intent to subcontract either verbally, in an e-mail, or in writing. This communication is documented. When this communication is in the form of an e-mail, it is printed and maintained in the project file.

Policy
The laboratory performing the subcontracted work is identified in the final report. ECCS assumes responsibility to the client for the subcontractor's work, except in the case where a client or a regulating authority specifies which subcontractor is to be used.

8.0 PURCHASING SERVICES AND SUPPLIES

ECCS ensures the services and supplies required to operate its business are effective.

8.1 Services

Services refer to balance calibration, Class 1 weight set calibration, service contracts for instrumentation, supply vendors, etc.

Policy
The laboratory ensures that purchased supplies and services that affect the quality of environmental tests are of the required or specified quality by using approved suppliers and products.

Policy
The laboratory has procedures for purchasing, receiving, and storage of supplies that affect the quality of environmental tests.

Procedure
The OM or designee reviews and approves requests for services and supplies and signs the authorization prior to ordering. Project managers and/or lead chemists also have that authority for on-site laboratory projects.

8.2 Suppliers

Evaluation of suppliers is accomplished by ensuring the vendor ships the product or material ordered against the purchase requisition form.

Procedure
A signature line is included on the purchase requisition to indicate that the items requested have been reviewed and meet quality specifications (like specific grades mentioned in SOPs).

8.3 Routine Supplies

Routine supplies such as solvents, bottles, etc, do not require a requisition as the vendor quality has been deemed acceptable by ECCS.

Procedure
Supplies received are reconciled against the packing list and inspected for damage. Supplies, reagents, and chemical standards are checked-in and distributed to the appropriate individuals, departments or storage areas. Reagents and standards are logged into LIMS and then labeled appropriately for documentation purposes. Documentation requirements for reagents and standards include:

- date of receipt;

- expiration date, if applicable;
- source;
- lot or serial number;
- calibration and verification records; and
- certifications.

Procedure
Supplies received are stored according to manufacturer's instructions, laboratory SOP, or test method specifications.

9.0 SERVICE TO THE CLIENT

Clients seek the on-site and fixed facility laboratory services that ECCS offers.

9.1 Client Communication

ECCS maintains and documents communication with clients and/or their representatives for the purposes of seeking feedback, both positive and negative, and clarifying requests. Each request is reviewed to determine the nature of the request and the laboratory's ability to comply with the request within the confines of prevailing statutes and/or regulations without risk to the confidentiality of other clients.

On-site laboratory projects require exceptional upfront communication with the client and documentation to ensure that project-specific objectives are known and met. Refer to SOP "GEN-013, On-site Testing" for more information about upfront communication.

Policy
Client feedback is analyzed to improve the QS, testing activities and service to the client.

9.2 Client Confidentiality

Policy
Information cannot be divulged or released to a third party without proper authorization from the client.

Policy
All electronic data (storage or transmissions) are kept confidential, based on current electronic technology, as required by client, ECCS policy, or regulation.

Procedure
The following notice shall accompany all e-mail and facsimile correspondence with clients:

NOTICE-- This email may contain confidential and privileged information for the sole use of the intended recipient. Any review or distribution by others is strictly prohibited. If you are not the intended recipient, please contact the sender immediately and delete all copies.

10.0 COMPLAINTS

The purpose of this section is to assure that customer complaints are addressed and corrected, if required.

Policy
All customer complaints are documented by the person receiving the complaint and addressed by appropriate personnel. If it is determined that a complaint is without merit, it is documented, and the client is contacted. If the complaint has merit the issue is resolved.

Policy
The Project Manager or Project Assistant reviews all complaints and determines appropriate action after discussing with the QM and/or OM. Corrective action is initiated in cases where the complaint is the result of a laboratory error. See Section 12 for corrective action procedures.

11.0 NON-CONFORMING WORK

Non-conforming work is work that does not meet acceptance criteria or requirements. Non-conformance can include unacceptable quality control results or departures from standard operating procedures or test methods. Requests for departures from laboratory procedures are approved by the QM and documented.

Policy
Employees should identify the non-conformance, determine if it will be permitted, and take appropriate action. All employees have the authority to stop work on samples when any aspect of the process does not conform to SOP requirements.

- The responsibilities and authorities for the management of non-conforming work are detailed in Section 12 – Corrective Action. The procedures for investigating and taking associated corrective actions for non-conformance are also described.
- The OM, QM, and/or PM evaluate the significance of the non-conforming work, and take corrective action immediately. If their data has been impacted, the client is notified within five working days.
- When an investigation of non-conformance indicates that the root cause of the non-conformance requires that a method be restricted or not used until modifications are implemented, the QM will immediately notify all personnel of the suspension/restriction. The QM will notify staff when resumption of work is authorized.

12.0 CORRECTIVE ACTION

Corrective action is taken to eliminate the root cause of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Policy
Nonconformities cited in external assessments, internal quality audits, data reviews, complaints, or managerial reviews are documented and require corrective action. Corrective actions taken are appropriate for the magnitude of the problem and the degree of risk.

Procedure
The data reviewer is responsible for initiating corrective action on routine data reviews. The QM is responsible for monitoring and recording corrective actions.

Policy
Nonconformities are investigated and a corrective action plan is developed and implemented, if deemed necessary. The implementation is monitored for effectiveness with discussion at weekly staff meetings.

Procedure
Corrective action reports for routine, non-recurring nonconformance can be records in logbooks, email, or other informal documents. More serious corrective actions require a more formal corrective action report.

- Sample data associated with failed quality control results are evaluated by the analyst for the need to be reanalyzed or qualified.
- Corrective action protocols specified in test methods may over-ride general corrective action procedures specified in this manual.
- Root cause is the condition or event that, if corrected or eliminated, would prevent the recurrence of a nonconformance.

Policy
Once a nonconformance is noted, the first action is an investigation to determine the root cause. Records are maintained of nonconformance requiring corrective action to show that the root cause(s) was investigated, and includes the results of the investigation.

Procedure
Where uncertainty arises regarding the best approach for analysis of the root cause of a nonconformance that requires corrective action, senior analysts are consulted to determine the corrective actions to be initiated. The QM ensures that corrective actions are discharged within the agreed upon time frame and documented as described in Section 12.3.

12.1 Monitoring of Corrective Action

Policy
The QM, or his designee, will monitor implementation and documentation of the corrective action to assure that the corrective actions were effective.

Procedure
All laboratory corrective actions are documented and implemented according to Section 12.3.

12.2 Documentation of Corrective Action

Procedure
Unacceptable quality control results are documented, and if the evaluation requires cause analysis, the cause is recorded and the solution implemented. When clear documentation of analytical events leading to problem resolution occurs, the information can be recalled to provide insight into the same or similar situations at a later date.

Procedure
Analysts are responsible for initiating or recommending corrective actions and ensuring that an exceedance of quality control acceptance criteria is documented. When a quality control limit is exceeded, a systematic approach to problem resolution is taken. A two-part Corrective Action Form (CAF), See Appendix D, is initiated that documents the corrective action process to its completion. When the out-of-control situation has been resolved, the completed form is maintained with the data until approval by the data reviewer. Once approved, the carbon copy of the CAF is routed to the QM to monitor trends and on-going analyst performance. The original is kept with the analytical run raw data file. Also, a CAF documents the disposition of any data that doesn't meet the requirements of the quality control program.

Procedure
Analysts routinely implement corrective actions for data with unacceptable quality control measures. First level correction may include re-analysis without further assessment. If the test method SOP addresses the specific actions to take, they are followed. Otherwise, corrective actions start with assessment of the cause of the problem.

Procedure
Data reviewers and the QM review corrective action reports and suggest improvements, alternative approaches, and procedures where needed. The review of CAFs is performed prior to the data being reported.

Procedure
If the data to be reported are affected adversely by the non-conformance, the data is qualified and a sample narrative is included with the final report.

Procedure
The discovery of a non-conformance for results that have already been reported to the client must be immediately evaluated for the significance of the non-conformance, its acceptability to the client, and determination of the appropriate corrective action. If the data reported to the client are affected adversely by the non-conformance, the client is notified.

12.3 Exceptionally Permitting Departures from Documented Policies and Procedures

An example of a planned departure is: The initial sample volume or final extract volume is adjusted to meet project reporting limit requirements. This change to the SOP requirements would be documented in the project file and in the case narrative as an exception to our SOP.

Policy
The laboratory allows the release of non-conforming data only with approval by the QM or his designee on a case-by-case basis. Project specific planned departures from procedures or policies are documented and do not require audits or investigations.

13.0 PREVENTIVE ACTION

Preventive action includes, but is not limited to, review of quality control data to identify quality trends, discussion of quality related issues at the regularly scheduled staff meetings, annual budget reviews, annual managerial reviews, scheduled maintenance, routine preventive maintenance, validating LIMS and spreadsheet calculations, and other actions taken to prevent problems. Preventive action, rather than corrective action, aims at minimizing or eliminating inferior data quality or other non-conformance through scheduled maintenance and review, before the non-conformance occurs.

Policy
All employees should be actively involved in performing preventive maintenance on their instruments. Preventive maintenance is addressed in the laboratory methods and procedures. Employees also have the authority and responsibility to recommend new or additional preventive action procedures. Management is charged with implementing preventive actions to improve laboratory operations, data quality, and maintain laboratory equipment.

Policy
Preventive maintenance to all instruments is documented in the instrument maintenance logs.

Policy
The QM, or his designee, periodically reviews logbooks and documents the review by initialing and dating the logs.

14.0 CONTROL OF RECORDS

Records are a subset of documents, usually data recordings that include annotations, such as daily refrigerator temperatures posted to a laboratory form, lists, spreadsheets, or analyst notes on a chromatogram. Records may be on any form of media, including electronic and hard copy. Records allow for the historical reconstruction of laboratory activities related to sample-handling and analysis.

Policy
The laboratory maintains a record system appropriate to its needs, documents all laboratory activities, and complies with applicable standards or regulations as required.

Procedure
The laboratory retains all original observations, calculations and derived data, calibration records, and reports for a minimum of five years. Observations, data, and calculations are recorded at the time they are made.

Policy
When mistakes are made in technical records, each mistake is crossed out with a single line (not erased, made illegible, or deleted) and the correct value entered alongside. Corrections are signed or initialed by the person making the correction. For electronic systems, all changes are tracked by the audit trail or by added notes. When changes are made to technical records for reasons other than for correction of transcription errors, the reason for the change is recorded on the document.

Policy
Records of all procedures to which a sample is subjected while in the possession of the laboratory are kept.

14.1 GC and LC Raw Instrument Data

Policy
Raw instrument data and any reprocessed chromatograms for GC and LC systems are stored electronically so that a complete trail of data reports can be reproduced. The electronic data in original or reprocessed format are considered the official record. Instrument sequence data packages contain the final processed data used to calculate the results presented in the final report.

14.2 GC/MS Raw Instrument Data

Policy
Raw instrument data for GC/MS systems are collected and stored electronically in original format so that the original report or chromatogram, before any manual manipulation or integration, can be reproduced. The electronic data in original format are considered the official record and need not be printed once manually integrated. Instrument raw data packages contain

the final processed data used to calculate the results presented in the final report.

14.3 Records Management and Storage

Policy
Records, including electronic records, are retrievable, legible, protected from deterioration or damage, held secure and in confidence, and are available for a minimum of five years.

Policy
The QM shall maintain a log of names, initials, and signatures for all individuals responsible for signing or initialing any hard copy laboratory record. LIMS also maintains a complete audit trail of changes made by any staff member.

Policy
The laboratory maintains a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting.

Policy
Archived information and access logs are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

Policy
In the event that the laboratory transfers ownership or goes out of business, records will be maintained or transferred according to client instructions.

Procedure
ECCS network drives are backed-up on a daily basis. Access to protected records is limited to ECCS staff to prevent unauthorized access or amendment.

14.4 Chain-of-Custody

Chain-of-Custody is the documentation trail of sample possession that accompanies samples submitted to the laboratory. Documentation is critical to providing legal evidence to support the maintenance of sample integrity. Authoritative sources suggest the following guidelines. See Appendix E for the ECCS chain-of-custody. A sample is in your custody if:

- it is in your possession, or
- it is within your view after being in your possession, or
- it was in your possession and you locked it up, or
- it is in a designated secure area.

Procedure
SOP "GEN-003 Chain-of-Custody, Log-in, Tracking Procedures and Sample Containers" provides for chain-of-custody procedures.

14.5 Evidentiary Chain-of-Custody Records

Evidentiary Chain-of-Custody Records including complete custody tracking of a sample throughout the laboratory testing process can be used as legal evidence. At the time of this revision, the laboratory has not handled evidentiary samples. Therefore, this subject is currently not applicable.

15.0 AUDITS AND MANAGEMENT REVIEW

The QS requires continuous monitoring by the QM and periodic review by executive management.

15.1 Types of Audits

Audits measure laboratory performance and verify compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the QS. They are also instrumental in identifying areas where improvement in the QS will increase the reliability of data. Audits are of four main types: internal, external, performance, and management reviews.

15.1.1 Internal Audits

Policy
The laboratory conducts internal audits of its QS activities for all operational areas, including data integrity and routine personnel training at least annually. Personnel may not audit their own activities except when it can be demonstrated that an effective audit will be carried out.

Procedure
Annually, the QM prepares a schedule of internal audits to be performed during the year. These audits verify compliance with the requirements of the QS, including analytical methods, SOPs, ethics policies, other laboratory policies, and accrediting authority Standards.

- It is the responsibility of the QM to plan and organize internal audits as required by the schedule and requested by management.
- The area audited, the audit findings, and corrective actions are recorded.
- All investigations that result in findings of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.
- Clients are notified with-in five days, in writing, when audit findings cast doubt on the validity of data.
- Audits are reviewed after completion to assure that corrective actions were implemented and effective.

15.1.2 External Audits

Policy
It is the laboratory's policy to cooperate and assist with all external audits, whether performed by clients or an accrediting authority.

Policy
All external audits are fully documented and tracked to closure.

Procedure
Management ensures that all areas of the laboratory are accessible to auditors as applicable and that appropriate personnel are available to assist in conducting the audit. Any findings related to an external audit follow corrective action procedures. Management ensures that corrective actions are carried out within the timeframe specified by the auditor(s).

15.1.3 Performance Audits

Performance audits may be proficiency test samples, internal single-blind samples, double-blind samples through a provider and/or client, or anything that tests the performance of the analyst and method.

The policy and procedures for proficiency test samples are discussed in Section 25.4.

15.2 Management Reviews

Policy
The President and the management team of ECCS reviews the QS on at least a yearly basis. The QM maintains records of the management review findings and any resulting actions.

Procedure
All components of the QS are reviewed over the course of each year, and findings are recorded. Management review of QS items will take place at one or more of the regularly scheduled operations team meeting. The reviews will be planned so that each item listed below will be addressed at least once per year. The management team assures that actions are performed within agreed time frames. The following is a list of review items:

- The suitability of policies and procedures
- Reports from managerial and supervisory personnel
- The outcome of recent internal audits
- Corrective and preventive actions
- Assessments by external bodies
- The results of inter-laboratory comparisons or Proficiency Tests
- Changes in the volume and type of the work
- Client feedback
- Complaints
- Other relevant issues

- Quality control activities
- Resources
- Staff training

16.0 PERSONNEL

ECCS has a unique personnel culture, because of its seamless on-site and fixed facility laboratory operations. Some personnel have day to day position descriptions that remain the same. However, chemists often have several position descriptions based on their current work assignment. Position descriptions are presented in Appendix B.

16.1 Position Descriptions

The President provides leadership to position the company at the forefront of the industry; develops a strategic plan to advance the company's mission and objectives and to promote revenue, profitability and growth as an organization; leads the marketing/sales efforts; and oversees company operations to ensure production efficiency, quality, service, and cost-effective management of resources. The president signs SOPs.

The operations manager manages operational issues associated with all on-site and fixed facility laboratory activities. The OM signs SOPs.

The quality manager has the authority and responsibility for ensuring that the QS is implemented and followed. The QM has direct access to the OM and is independent of operations. The QM signs SOPs.

Senior chemists provide solution-oriented and quality objective-oriented service to our clients; have the skills to be the entire laboratory staff (for example, in an on-site laboratory environment); provide for technical training of staff; and are the primary day-to-day analysts in the on-site and fixed facility laboratories.

Chemists and laboratory technicians primarily perform support work required to produce analytical results while adhering to quality control procedures specified in laboratory SOPs and the QSM.

The lead chemist provides for the day-to-day operations management of one of ECCS' on-site laboratory projects. The lead chemist determines project requirements and client interaction roles; prepares necessary equipment, supplies and vehicles (if required); conducts/supervises (when required) on-site testing operations; decommissions the project's on-site laboratory unit; prepares the final report; and provides feed back to the laboratory management.

The project manager provides support to sales/marketing staff in design and procurement of new projects; program management of all projects assigned.

The project assistant role provides for the day-to-day cradle to grave peripheral support of on-site and fixed facility laboratory projects by working with the project manager, client services, the OM, and/or lead chemists to coordinate typically non-technical logistical issues during project planning and initiation; raw data downloads to LIMS; final report preparation; file review and closure; and client feedback episodes.

The technical director role is not defined at this time. Currently, senior chemists support staff in the typical role of technical director.

17.0 DATA INTEGRITY AND ETHICS

Data integrity is the result of the processes that together assure valid data of known and documented quality. Data integrity and ethics procedures in the laboratory include training, signed and dated data integrity documentation for all laboratory employees, periodic monitoring of data integrity, and documented data integrity procedures.

Policy
Managers uphold the spirit and intent by supporting and enforcing data integrity procedures, and by signing and dating the data integrity procedure training forms.

Policy
Data integrity procedures are reviewed annually and are updated as needed. Data integrity procedures are periodically monitored through regularly scheduled internal audits, in-depth data review, records review, or other thorough check processes.

Procedure
SOP GEN-008 addresses Laboratory Ethics and Data Integrity.

17.1 Data Integrity and Ethics Training

Policy
Data integrity training is provided for all (including temporary) employees initially upon hire and annually thereafter.

Procedure
Attendance at an initial data integrity training (part of new employee orientation) and the annual refresher training is recorded with a signature attendance sheet or other form of documentation that demonstrates all staff members have participated and understand their obligations related to data integrity.

17.2 Detecting and Deterring Improper, Unethical or Illegal Actions

Procedure
SOP “GEN-008, Laboratory Ethics and Data Integrity,” provides for procedures to detect and deter improper, unethical or illegal actions.

18.0 GENERAL TRAINING

Policy
All personnel are appropriately trained and competent in their assigned tasks before they contribute to functions that can affect data quality. It is management's responsibility to assure personnel are trained.

Policy
Only trained personnel are authorized to perform specific tasks.

Policy
SOP GEN-009 addresses documentation of training.

Procedure
New staff members are given introductory training and orientation upon arrival. This training is documented.

18.1 Attendance

Attendance at training sessions is documented on signature sheets that are maintained on file by the QM. Training also occurs through email correspondence from the QM. Documentation occurs with a reply from the trainee that they have completed the training assignment.

18.2 Initial Training

The initial training for a new task consists of:

- All documentation involved with a new and unfamiliar task is read and understood by the trainee.
- Training is performed under the direct supervision of a qualified analyst or technician. During the time the analyst is training, the trainee may sign laboratory notebooks or logbooks, but laboratory notebooks must be cosigned by the senior analyst, who is responsible for the data generated.
- The trainee demonstrates competency in the new task before they can operate independently. The competency for a test method is accomplished by a demonstration of capability as indicated in Section 20.1. Approval of competency is noted by the initials or signature of the qualified senior analyst on the training form.
- Each step of the training process is documented.

18.3 Ongoing Training

Ongoing training consists of the following:

- The employee attests, through signature, email correspondence (18.1.2), or equivalent that they have read, understood, and agreed to follow the latest version of the QSM and any method SOPs.
- Annually, the analyst/technician shows ongoing proficiency in each method they perform. Proof of acceptable on-going training is documented by the annual demonstration of capability (DOC) for each analyst and each method. See SOP "GEN-021, Demonstration of Capability (DOC) Determinations," for the initial and ongoing DOC procedures**.**
- Other training as determined by management.

19.0 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

Policy
ECCS laboratory facilities are designed and organized to facilitate testing of environmental samples. Environmental conditions are monitored to ensure that conditions do not invalidate results or adversely affect the required quality of any measurement.

Policy
In the event of a power failure or other interruption of normal laboratory operations, all criteria of the analytical method SOP (instrument tuning, calibration, etc.) must be demonstrated prior to resuming sample analysis. All method requirements shall apply.

Policy
Environmental tests are stopped when the environmental conditions jeopardize the results.

Policy
Access to, and use of areas affecting the quality of the environmental tests is controlled by restricting areas to authorized personnel only.

Policy
The laboratory work spaces are adequate for their use, and appropriately clean to support environmental testing and ensure an unencumbered work area.

Procedure
Laboratory space is arranged to minimize cross-contamination between incompatible areas of the laboratory. In the fixed facility, volatile organics analysis is performed and samples and sample extracts are stored in a separate building, away from the main lab where solvent extractions occur to prevent contamination.

Policy
When a sample(s) is suspected to contain high concentrations of target analytes, great care is taken to ensure cross contamination does not occur.

Procedure
When a sample(s) is suspected to contain high concentrations of target analytes, a note is added to the comment section in LIMS. In the both the VOC and SVOC area, known highly contaminated samples are segregated from other samples in a separate area of cold storage. In the VOC area, water and methanol storage blanks are used to monitor the potential for cross contamination. Action limits for storage blanks are the same as for method blanks.

Procedure
If the laboratory conditions are required to be controlled by method or regulation, the adherence is recorded.

19.1 Availability of Equipment and Reference Materials

All equipment and reference materials required for the accredited tests are available in the laboratory. Records are maintained for all equipment, reference measurement materials, and services used by the laboratory.

Reference materials traceable to national standards of measurement or to national standard reference materials are stored away from heavy use areas. Certificates of Traceability are available for thermometers and the Class 1 weights. The reference materials are used only for calibration to maintain the validity of performance.

20.0 TEST METHODS AND METHOD VALIDATION

Policy
A method is validated before it is put into use. All methods validations are documented.

20.1 Demonstration of Capability

A DOC is a procedure to establish the ability of the analyst to generate data of acceptable accuracy and precision. See SOP "GEN-021, Demonstration of Capability (DOC) Determinations," for initial and ongoing DOC procedures.

ECCS defines an initial DOC as four spiked samples in a control matrix at the level of the LCS. A method blank is also included.

DOCs that are prepared by a technician and then analyzed by an analyst are applied to the training files of each individual. The sample preparation applies to the technician and the analysis applies to the analyst.

Analysts need not perform DOCs for similar methods (*i.e.*, 8081 and 8082).

New or inexperienced technicians/analysts work under the direct supervision of an experienced analyst who has completed acceptable DOCs.

DOCs are evaluated for most analytes based on simple pass/fail guidelines. The percent recovery must be with 70% to 130%, and the % RSD ≤20%. There are exceptions to the above criteria, as a test method may have an analyte(s) with typical poor performance. These analyte(s) are treated separately as defined in method SOPs.

Policy
The laboratory confirms that it is capable of generating data of acceptable accuracy and precision on all methods before employing them.

Procedure
The DOCs are documented and approved by the OM or the QM. Completed forms are kept in the training files for each analyst. DOCs are performed for each analyte whenever the method, analytes, or instrument is changed.

20.2 On-Going (or Continued) Proficiency

On-going (or continued) proficiency is maintained and demonstrated at least annually by the successful analysis of:

- either a single-blind or a performance testing (PT) samples,
- an MDL study,

- a new initial DOC, or
- four consecutive LCS results.

Demonstration of proficiency is documented in the training file of each analyst.

20.3 Work Cells

On-site laboratory projects are considered a work cell. When the members of the work cell change, the new members must demonstrate proficiency as described above.

20.4 Initial Test Method Evaluation

Initial test method evaluation involves the determination and documentation of the calibration range, interferences, (LOD), confirmation of the limit of quantitation (LOQ), an evaluation of precision and bias, and an evaluation of the selectivity of the method.

The MDL is an estimate of the minimum amount of a substance that an analytical process can reliably detect. ECCS equates the terms MDL and LOD.

Procedure
MDLs (LODs) are determined according to SOP "GEN-019, Method Detection Limit (MDL) Determinations."

The limit of quantitation is an estimate of the minimum amount of a substance that can be reported with a specified degree of confidence. ECCS equates the terms LOQ and reporting limit.

Policy
The lowest calibration standard is defined as the RL unless otherwise specified by a regulatory agency or client.

Policy
The RL for water samples are adjusted for each sample based on volume of water used. The RL for soil samples are adjusted for the weight of soil used and percent solids.

Policy
If results are not reported below the calibration range (lowest calibration standard), the MDL determination is not required. If an MDL study is not performed, reporting unqualified concentrations less than the lowest calibration standard is not allowed.

20.4.1 Precision and Bias

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, and RSD based on the mean value of the replicates.

Bias is the systematic error that contributes to the difference between the mean of a statistically significant number of test results and the theoretical value.

Policy
Precision and bias are evaluated for standard and non-standard methods.

Procedure
Precision and bias are evaluated for standard methods through the performance of DOCs, MDLs, and method specific quality control samples.

When using non-standard, modified standard, or laboratory-developed methods, precision is compared to the criteria established by the client (when requested), the method, or the laboratory. When established criteria are not available, the assessment of precision and bias should be based on the science of the method and practical experience.

20.4.2 Selectivity

Selectivity is the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.

Policy
The laboratory evaluates selectivity through procedures defined in the test method SOPs.

20.5 Estimation of Uncertainty

Estimation of Uncertainty is the sum (combining the components) of the uncertainties of the numerous steps of the analytical process, including, but not limited to, sample plan variability, spatial and temporal sample variation, sample heterogeneity, calibration/calibration check variability, extraction variability, weighing variability, and analytical system variability.

Procedure
The laboratory estimates uncertainty using the percent recovery calculated from routine quality control samples.

20.6 Laboratory-Developed or Non-Standard Method Validation

Policy
Laboratory developed, modified standard methods, and non-standard methods require method validation as described above.

Procedure
Laboratory method validation consists of performing DOCs and MDLs. Evaluation of DOCs is addressed in Section 20.1.6. MDLs are evaluated according to SOP "GEN-019, Method Detection Limit (MDL) Determinations."

Policy
Where applicable, the laboratory validates non-standard methods, laboratory-designed/developed methods, standard methods used outside their published scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use.

Policy
The range and accuracy of the values obtainable from validated methods (*e.g.*, the uncertainty of the results, detection limit, selectivity of the method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), is assessed for the intended use and whether it is relevant to the clients' needs.

20.7 Control of Data

Policy
All calculations and all relevant data are subject to appropriate checks in a systematic manner.

Policy
Commercial off-the-shelf software (*e.g*., word processing, database, data acquisition and statistical programs) used within the designed application range is considered sufficiently validated when in-house programming is not used. However, at a minimum a sample data set is used to verify automated data reduction processes.

Procedure
The laboratory assures that computers and software are protected, maintained, and secure through measures such as documentation, locked access, and control of the laboratory environment.

Procedure
The laboratory procedure to insure that reported data are free from transcription and calculation errors and that all quality control measures are reviewed and evaluated before data are reported is found in SOP “GEN-016, Data Review Procedures.”

The laboratory assures that computers, user-developed computer software, automated equipment, or microprocessors used for the acquisition, processing, recording, reporting, storage, or retrieval of environmental test data are:

- documented in sufficient detail and validated as being adequate for use;
- protected for integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;
- maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data; and

- held secure including the prevention of unauthorized access to, and the unauthorized amendment of computer records.

21.0 EQUIPMENT

The laboratory provides all the necessary equipment required for the correct performance of the scope of environmental testing presented in this QSM.

21.1 General Equipment Requirements

Policy
All equipment and software used for testing and sampling is capable of achieving the accuracy required and complies with the specifications of the environmental test method as specified in the laboratory method SOP.

Policy
Equipment is operated by authorized personnel only.

Policy
Manufacturers' manuals and maintenance/calibration logs are readily available for use by laboratory personnel.

Policy
All equipment is calibrated or checked before being placed into use to ensure that it meets laboratory specifications and the relevant standard specifications.

Policy
Test equipment, including hardware and software, are safeguarded from adjustments which would invalidate the test results by limiting access to the equipment and using password protection where possible.

Policy
Equipment that has been subject to overloading, mishandling, produced suspect results, or been shown to be defective or outside specifications is taken out of service, isolated to prevent its use, or clearly labeled as being out of service until it has been shown to function properly. If it is shown that previous tests are affected, then procedures for non-conforming work are followed.

Policy
When equipment is needed for a test that is outside of permanent control of the laboratory, the lab ensures the equipment meets the requirements of this manual prior to its use by inspecting or otherwise testing it.

Policy
Each item of equipment and the software used for testing and significant to the results is uniquely identified, and records of equipment and software are maintained in the maintenance log and/or computer system for each instrument. This information includes the following:

- identity of the equipment and its software;
- manufacturer's name, type identification, serial number or other unique identifier;
- checks that equipment complies with specifications of applicable tests;
- dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration, where applicable;
- maintenance plan where appropriate, and maintenance carried out to date; documentation on all routine and non-routine maintenance activities and reference material verifications;
- any damage, malfunction, modification or repair to the equipment;
- date received and date placed into service (if available); and
- condition when received, if available (new, used, reconditioned).

21.2 Support Equipment

Support equipment includes, balances, ovens, refrigerators, freezers, temperature measuring devices, and volumetric dispensing devices.

Policy
All support equipment is maintained in proper working order and records are kept of all repair and maintenance activities, including service calls.

Procedure
All raw data records are retained to document equipment performance. These records include logbooks, data sheets, or equipment computer files.

Procedure
All support equipment is calibrated or verified annually over the entire range of use using NIST traceable references where available. The results of the calibration of support equipment are within specifications or the equipment is removed from service until repaired.

Procedure
Support equipment such as balances, ovens, refrigerators, freezers, and water baths are checked according to SOP GEN-005, Daily Equipment Checks, to ensure they are operating within the expected range for the application for which the equipment is to be used.

Policy
Class B volumetric glassware is check for accuracy by lot before use. Class A and B glassware is checked for accuracy upon evidence of deterioration.

Policy
Mechanical volumetric dispensing equipment is checked for accuracy quarterly according to

SOP "GEN-014, Quarterly Calibration of Air Dispensing Pipette Devices." Bottle-top Optifix volumetric dispensers are checked prior to use for each batch.

21.2.1 Support Equipment Maintenance

Policy
Regular maintenance of support equipment, such as balances and fume hoods is conducted at least annually. Maintenance on other support equipment, such as ovens, refrigerators, and thermometers is conducted on an as-needed basis.

Policy
Records of maintenance to support equipment are documented in Instrument Maintenance Logs. Each piece of support equipment does not necessarily have its own logbook. Maintenance logbooks may be shared with equipment that is housed in the same laboratory area.

21.2.2 Support Equipment Calibration and Performance Checks

Calibration and performance check requirements for analytical support equipment are found in the Table 21.2.2 below. For analytical instrumentation, the calibration requirements are found in the SOP for each analytical method.

Calibration date and expiration date (when recalibration is due) is recorded for equipment requiring calibration, where applicable.

21.3 Analytical Equipment

Policy
All equipment is properly maintained, inspected, and cleaned.

Procedure
Maintenance of analytical instruments and other equipment may include regularly scheduled preventive maintenance or maintenance on an as-needed basis due to instrument malfunction and is documented in Instrument Maintenance Logs, which become part of the laboratory's permanent records. Maintenance for analytical equipment is included in the SOP for each analytical method.

Table 21.2.2 Calibration and Maintenance of Support Equipment

Instrument	Activity	Frequency	Documentation
Balance	1. Calibration check 2. Clean 3. Check alignment 4. Weighing	1. Annually 2. Before use 3. Before use 4. Daily before use	1. Date on balance post annual service in logbook 4. Logbook
ASTM Class 1 Weights	1. Only use for the intended purpose 2. Use plastic forceps to handle 3. Keep in case 4. Calibration check	4. Once every 5 years	4. Keep certificate in analytical balance logbook
Working Class Weights	1. Use plastic forceps to handle 2. Keep in case 3. Calibration check	3. Yearly	3. QA electronic log
Digital Thermometers	Verify or replace annually	Annual check vs. current traceable or purchase new	Calibration information and certificates in the log book or on file
Automatic or digital pipettes	Calibrate for accuracy and precision using reagent water and analytical balance	Quarterly	Bench sheet or logbook
Refrigerators and Freezers	1. Thermometers are immersed in liquid to the appropriate immersion line 2. Thermometers are graduated in increments of 1°C or less	Temperatures are recorded each day in use	Logbook
Bottle-top Volumetric Dispensers	1. Clean 2. Verify accuracy	1. When switching solvents. 2. Prior to each batch.	2. Bench sheet or logbook
Glass Microliter Syringes	1. Clean 2. Verify accuracy	1. Before each use 2. Upon Receipt	1. not required 2. logbook

22.0 CALIBRATION

Initial calibration (ICAL) and continuing calibration verification (CCV) are an important part of ensuring data of known and documented quality. If more stringent calibration requirements are included in a mandated method or by regulation, those calibration requirements override any requirements outlined here or in laboratory SOPs. Specifics for conducting instrument calibrations are provided in test method SOPs.

Policy
ICALs are verified with a standard obtained from a second source traceable to a national standard when commercially available. If a second source is not available, a standard prepared from a separate lot may be used as long as the manufacturer can demonstrate the lot was prepared independently from other lots purchased.

Procedure
ICAL standards prepared from new stock standards are verified and documented with a second source standard. The second source is also used as the LCS spiking solution. LCS recovery trends are monitored to assure ongoing standards integrity. Initial and second source standards must agree within method specified control limits.

Policy
Any samples that are analyzed after an unacceptable ICAL are re-analyzed or the data are reported with qualifiers, appropriate to the scope of the unacceptable condition.

Policy
Quantitation is always determined from the ICAL unless the test method or applicable regulations require quantitation from the continuing calibration.

Policy
The lowest calibration standard is the lowest concentration for which quantitative results can be reported without qualification except where defined by a regulatory agency or client.

Policy
The highest calibration standard is the highest concentration for which quantitative results can be reported without being qualified.

Procedure
The ICAL includes calculations, integrations, acceptance criteria, and associated statistics referenced in the test method SOP. Acceptance criteria for initial calibration are listed in the test method SOPs.

Sufficient raw data records are collected to allow reconstruction of the ICAL. These include, at a minimum, calibration date, test method, instrument, analysis date, analyte names, analyst's signature or initials, concentration and response, calibration curve or response factor, or unique

equation or coefficient used to reduce instrument responses to concentration.
Under Method 8000B, corrective actions must be performed when the ICAL results are outside the method acceptance criteria. For any ICAL that does not meet the method criteria, the cause of the failure and any corrective action must be documented on a CAF. The analyst should review the calibration results to ensure that the problem is not associated with just one of the ICAL standards. If the problem is associated with just one of the standards, that standard may be reanalyzed and the RSD recalculated (Note: this reinjection is only allowed once). If the cause cannot be determined, and the condition resolved, the calibration curve must be re-prepared and any samples analyzed after the ICAL must be reanalyzed.

Normally standards should not be dropped from an initial calibration. If a standard to be dropped is the upper point of the curve, then the concentration of the next lower standard becomes the high calibration point. Any samples with analyte concentrations above that point must be diluted, or the analytes that are greater than the curve must be qualified as estimates.

Dropping a mid-point standard is not allowed unless there is justification (e.g., a bad purge for VOCs, internal standards were not added, an obvious syringe error, etc.) The dropping of a mid-point standard without a valid reason (i.e., simply to meet calibration curve acceptance criteria) is prohibited. If the lowest standard is dropped, the analyst has the following options: 1) raise the reporting limit up to the level of the next lowest acceptable standard, or 2) as discussed above, reinject the lowest standard later in the run and include the reinjection in the standard curve (Note: this reinjection is only allowed once). As stated above, any corrective action must be documented on a CAF that is included with the raw data.

- Results that are less than the lowest calibration standard are considered to have increased uncertainty, and are reported with qualifier codes. See SOP "GEN-015, Qualification of Data."
- Results that are greater than the highest calibration standard are either diluted to within the calibration range or considered to be an estimate, and reported with a qualifier code and explained in the case narrative.

22.1 Continuing Instrument Calibration

Policy
The validity of the initial calibration is verified prior to sample analysis by use of an initial calibration verification (ICV) standard.

Policy
The validity of continued calibration is verified during sample analysis by use of CCV standards.

Policy
Corrective action is initiated for CCV results that are outside of acceptance criteria. Samples are re-analyzed and/or results are qualified.

Procedure
CCV requirements are specified in the test method SOPs.

- The calculations and associated statistics for continuing calibration are included or referenced in the test method SOP.
- Sufficient raw data records are retained to allow reconstruction of the CCV and the associated ICAL.

22.1.1 Unacceptable Continuing Calibration Verification

Policy
Corrective action is required following an unacceptable CCV. If following the corrective action, the instrument cannot produce a CCV within the acceptance criteria, a new initial calibration is performed and/or the associated data is qualified. See SOP "GEN-015, Qualification of Data," for additional guidance.

23.0 MEASUREMENT TRACEABILITY

Measurement traceability is another factor that determines the correctness and reliability of the environmental test measurements performed.

23.1 Measurement Quality Assurance

Policy
All equipment used that affects the quality of test results are calibrated prior to being used to obtain analytical results and on a continuing basis. These calibrations are traceable to national standards of measurement where available.

Policy
If traceability of measurements to The International System of Units (SI) is not possible or not relevant, evidence for correlation of results through inter-laboratory comparisons, proficiency testing, or independent analysis is provided.

Procedure
All equipment that affects the quality of test results are calibrated according to the minimum frequency suggested by the manufacturer, by regulation, by method, or as needed.

- Clients can verify that required uncertainty is achieved by reviewing the internal quality control data provided with each report.

23.2 Reference Standards

Reference standards are standards of the highest quality available at a given location, from which measurements are derived.

Policy
Reference Standards, such as ASTM Class 1 weights, are used for calibration only and for no other purpose unless it is shown that their performance as reference standards will not be compromised.

Procedure
Reference standards, such as ASTM Class 1 weights, are calibrated by an entity that can provide traceability to national or international standards. Class 1 weights are sent out every five years for calibration to a national standard.

23.3 Reference Materials

Reference materials are substances that have concentrations that are sufficiently well established to use for calibration or as a frame of reference.

Policy
Reference materials, where commercially available, are traceable to national standards of measurement, or to Certified Reference Materials, usually by a Certificate of Analysis.

Policy
Internal reference materials, such as working standards or intermediate stock solutions, are checked as far as technically and economically possible.

Procedure
Purchased reference materials require a Certificate of Analysis where available. Otherwise, purchased reference materials are verified by application to a certified reference material, inter-laboratory comparison, and/or demonstration of capability.

Procedure
Internal reference materials, such as working standards and intermediate stock solutions, are checked by comparison with second source standards or materials that have been compared to second source standards.

Procedure
Internal thermometers are checked against the NIST certified reference thermometer or are replaced annually.

Procedure
Working class weights are checked against Class 1 weights annually.

23.4 Transport and Storage of Reference Standards and Materials

Policy
The laboratory handles and transports reference standards and materials in a way that protects their integrity.

Procedure
Reference standards and materials are protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials.

Policy
Reference standards and materials are stored according to manufacturer's recommendations and separately from working standards or samples.

Policy
On-site laboratory standards are transported under cool/refrigerated conditions.

In an on-site laboratory it may be necessary to store working standards in the same refrigerator/freezer as unknown samples. However, the unknown samples are typically prepared

and/or analyzed upon receipt and prior to storage in the refrigerator. Where possible the standards are stored in a separate area.

23.5 Labeling of Reference Standards, Reagents, and Materials

Policy
Reference standards and materials are tracked from purchase, receipt, and storage through disposal.

Policy
Reagent quality is verified during routine blank analysis and is tracked by manufacturer's lot number. Reagents are entered into and tracked by the LIMS.

Procedure
The procedures for receipt, documentation, handling, and disposal of reference standards are addressed in SOP GEN-004, Analytical Standards and Reagents.

24.0 SAMPLE MANAGEMENT

While ECCS does not participate in the collection of samples and has little control over its client's sampling activities, documented sample management techniques are important in maintaining the validity of the sample while in the confines of its laboratories.

24.1 Sample Receipt

Procedure
When samples are received at the laboratory, their condition is documented, they are given unique identifiers, and they are logged into the ECCS LIMS system. See SOP "GEN-003, Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers."

24.2 Sample Acceptance

Policy
For the minimum conditions a sample must meet upon receipt, see SOP "GEN-003, Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers." If these conditions are not met, the client is contacted prior to any further processing.

The sample acceptance policy, including specific instructions, is provided to sample collection personnel with the bottle order. These instructions emphasize the need for use of water resistant ink, use of appropriate containers, adherence to holding times, sample volume requirements, sample preservation, and what to do with compromised samples.

Procedure
SOP GEN-003 contains the various checks that are performed on samples at the time of receipt and the required actions and documentation in the event that the criteria of the SOP are not met.

All information provided with samples is maintained by administrative staff in the original project folders. A copy of the information is attached to the LIMS work order provided to the analyst.

24.3 Sample Identification

Policy
Samples including subsamples, extracts, and digestates, are uniquely identified in a permanent chronological record (ECCS LIMS) to prevent mix-up and to document receipt of all sample containers.

Procedure
SOP GEN-003 addresses the sample receipt and Log-In procedure.

24.4 Sample Storage

Procedure
Storage conditions (i.e., temperature) are monitored as required by SOP GEN-005, Daily Equipment Checks.

- Samples are held secure and stored apart from contaminating sources such that cross-contamination between samples is minimized. All portions of samples, including extracts, digestates, leachates, etc. are maintained according to method required conditions.
- For on-site laboratory projects, where only one refrigerator may be available, standards should be isolated from samples through the use of a secondary container.

24.5 Sample Disposal

Policy
Samples are disposed of according to federal, state and local regulations. Procedures are available for the disposal of samples, digestates, leachates, and extracts.

Procedure
SOP GEN-003 provides detailed information regarding sample disposal.

24.6 Sample Transport

Policy
Samples that are transported under the responsibility of the laboratory, where necessary, are done so safely and according to storage conditions. This includes moving bottles within the laboratory. Specific safety required are addressed in the ECCS Chemical Hygiene Plan and in method SOPs.

24.7 Sampling Records

Policy
ECCS does not collect samples except for our SDWA compliance monitoring business. These samples are collected according to SDWA guidelines. The samples are then subcontracted to SDWA certified labs for analysis.

24.8 Subsampling

Policy
Subsampling is performed in such a manner to obtain a representative sample. See SOP "GEN-023, Sub-sampling Soil Samples" for sub-sampling procedures.

Policy
When an unusual sample matrix is noted or the presence of an inordinate amount of extraneous material exists in a sample, the condition is noted in LIMS.

Procedure
"GEN-022, Compositing Soil Samples" provides the procedure for compositing soil samples, when required by our clients.

25.0 QUALITY OF TEST RESULTS

This section provides for the policies and procedures needed to assure applicable quality control principals are implemented. Method-specific quality control procedures are found in the individual method SOPs.

25.1 Essential Quality Control Procedures

Policy
All essential quality control elements are collected and assessed on a continuing basis.

Policy
The qualities of test results are recorded in such a way that trends are detectable, and where practicable, are statistically evaluated.

Policy
For test methods that do not provide acceptance criteria for an essential quality control element or where no regulatory criteria exist, acceptance criteria are developed based on practical experience.

Policy
The quality control procedures specified in test methods are followed by laboratory personnel. The most stringent of control procedures is used in cases where multiple controls are offered. If it is not clear which is the most stringent, that mandated by test method or regulation is followed.

Procedure
To monitor the validity of environmental tests performed, review includes any one or combination of the techniques below:

- use of certified reference materials and/or internal quality control using secondary reference materials;
- participation in proficiency testing programs;
- replicate testing using the same or different methods;
- retesting of retained samples.

Written procedures to monitor quality controls including acceptance criteria are located in the test method SOPs, except where noted, and includes:

- use of laboratory control samples (LCS) and blanks to serve as positive and negative controls;
- use of LCSs to monitor test acceptability of laboratory results;
- use of matrix spike samples and/or matrix spike duplicate samples to monitor precision and accuracy associated with a specific matrix;

- use of duplicate samples to monitor precision associated with a specific matrix;
- use of ICALs, CCVs, certified reference materials, and PT samples to monitor accuracy of the test method;
- measures to monitor test method capability, such as limit of detection, limit of quantitation, and/or range of test applicability, such as linearity;
- use of regression analysis, internal/external standards, or statistical analysis to reduce raw data to final results;
- use of reagents and standards of appropriate quality per method SOP; and,
- procedures to ensure the selectivity of the test method.

25.2 Internal Quality Control Practices

Analytical data generated with quality control samples that fall within prescribed acceptance limits indicate the test method is "in control."

Quality control samples that fall outside acceptance limits indicate the test method is "out of control" (non-conforming) and that corrective action is required or that the data are qualified.

Policy
Detailed quality control procedures are included in method SOPs or as general guidelines set forth in this section.

Policy
All quality control measures are assessed and evaluated on an on-going basis so that trends are detected.

Acceptance or rejection criteria are created according to laboratory policy where no method or regulatory criteria exist. Acceptance criteria define the boundary for the appropriate response from laboratory personnel, such as corrective action, reporting with qualifiers, reanalysis, review, and others.

Procedure
The following general controls are used:

Positive and negative controls such as:

- Blanks (negative)
- LCS (positive)

Selectivity is assured through:

- absolute and relative retention times in chromatographic analyses;

- second-column confirmation when using non-selective detectors; and
- use of acceptance criteria for mass-spectral tuning (found in test method SOPs);
- use of the correct method according to its scope assessed during method validation.

Consistency, variability, repeatability, and accuracy are assured through:

- proper installation and operation of instruments according to manufacturer's recommendations or according to the processes used during method validation;
- monitoring and controlling environmental conditions (temperature, access, proximity to potential contaminants);
- selection and use of reagents and standards of appropriate quality; and
- cleaning glassware appropriate to the level required by the analysis. Cleaning procedures are specified in SOP "GEN-006, Glassware Cleaning."
- following SOPs and documenting any deviation, assessing for impact, and treating data appropriately;
- testing to define the variability and/or repeatability of the laboratory results, such as replicates or MS/MSD samples;
- use of measures to assure the accuracy of the test method, including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

Test method capability is assured through:

- establishment of the limit of detection or MDL where appropriate;
- establishment of the limit of quantitation or reporting level;
- establishment of the range of applicability such as linearity or acceptable regression analysis;
- analysis of a set of acceptable MDLs; and
- analysis of a set of DOC samples.

Data Reduction is assured to be accurate by:

- selection of appropriate formulae to reduce raw data to final results such as regression;
- periodic review of data reduction processes to assure applicability; and
- data reduction, and statistical interpretations specified by each test method.

Table 25.2 summarizes the key elements of the ECCS quality control system. Corrective actions for items in Table 25.2 below are specified in the method SOP and/or according to SOP GEN-015, Data Qualification.

25.3 Quality Control Samples

Method Blanks are prepared from analyte free water or sand.

Policy
Blank test results are reviewed according to the acceptance limits in the test method SOPs or laboratory documentation.

Policy
Samples associated with a blank that exceeds acceptance limits are evaluated as to the appropriate corrective action for the samples (e.g. reprocessing or data qualification).

Procedure
Criteria for acceptance of method blanks are included in the method SOPs.

Table 25.2 Key Elements of Quality Control Program

Item	Frequency	Acceptance Criteria
Method Blank (Negative Control)	1/ batch of 20 or less or 1/day, whichever is greater.	Less than the reporting limit
Laboratory Control Sample (Positive Control)	1/ batch of 20 or less or 1/day, whichever is greater.	Laboratory control limits
Matrix Spike and/or Matrix Spike Duplicate Samples	1/ batch of 20 or less or 1/day, whichever is greater.	Laboratory control limits
Duplicate Samples	1/ 10 or less or 1/day, whichever is greater.	Laboratory control limits
Surrogates	Per method requirement	Laboratory control limits
Continuing Calibration Verification	Per method requirement	Method specific
Initial Calibration Verification	Following each initial calibration.	Method specific

NOTE: See method SOPs for specific criteria.

When a blank is determined to exceed acceptance limits, the cause must be investigated and measures taken to minimize or eliminate the problem.

Data that are unaffected by the blank test results (non-detects or other analytes) are reported unqualified.

Sample data that are suspect due to the presence of a blank exceedance are reanalyzed, and/or qualified.

Laboratory control samples are prepared from analyte free water or sand, and spiked with verified and known amounts of analytes for the purpose of establishing precision or bias measurements.

Policy
LCSs are analyzed at a frequency specified in method SOPs.

Procedure
The results of LCSs are calculated in percent recovery. Matrix specific LCS control limits for each method are calculated based on the mean and standard deviation of recoveries from a significant pool (minimum of 20 points) of data collected over time. Acceptance limits are based on the 99% confidence limit (±3 standard deviations) generated from the historical data. Where there are not established method criteria, or there are not 20 points available to calculate limits, interim limits are used.

Corrective actions for out of control recoveries are given in the method SOP and SOP GEN-015 Data Qualification.

Matrix Spikes and/or matrix spike duplicates are samples fortified with a known amount of analyte to help assess the effect of the matrix on method performance.

Policy
Matrix spike results are used to help assess the effect of the sample matrix on method performance.

Procedure
The laboratory procedure for matrix spikes includes spiking appropriate analytes at appropriate concentrations, calculating percent recoveries and relative percent difference (RPD) for MS/MSDs, and evaluating and reporting the results.

Procedure
Matrix specific matrix spike control limits for each method are calculated based on the mean and standard deviation of recoveries from a significant pool (minimum of 20 points) of data collected over time. Acceptance limits are based on the 99% confidence limit (±3 standard deviations) generated from the historical data. Where there are not established method criteria, or there are not 20 points available to calculate limits, interim limits are used.

The RPD control limit is also statistically established.

Policy
If a sample that is spiked contains a spiked analyte equal to or greater than the amount added, the acceptance criteria do not apply.

Corrective actions for out of control recoveries are found in method SOPs and "GEN-015, Qualification of Data."

Surrogates are substances with chemical properties and behaviors similar to the analytes of interest used to assess method performance in individual samples.

Policy
Surrogates are added to all samples (in test methods where surrogate use is appropriate) prior to sample extraction.

Procedure
Surrogate recovery results are compared to the acceptance criteria calculated by LIMS on at least a yearly basis.

Procedure
Matrix specific surrogate control limits for each method are calculated based on the mean and standard deviation of recoveries from a significant pool (minimum of 20 points) of data collected over time. Acceptance limits are based on the 99% confidence limit (±3 standard deviations) generated from the historical data. Where method criteria are not established, or there are not 20 points available to calculate limits, interim limits listed in method SOPs are used.

Corrective actions for out of control surrogate recoveries are found in method SOPs and "GEN-015, Qualification of Data."

25.4 Proficiency Test Samples or Inter-Laboratory Comparisons

Policy
The laboratory participates in PT studies a minimum of twice per matrix per year as required by NELAP. The results of these PT studies are also used for maintenance of other certifications/accreditations held by ECCS.

Policy
The laboratory may utilize internal PT samples at any time for quality monitoring and quality improvement as determined by the QM.

Procedure
Proficiency test samples may be analyzed in either the fixed-based laboratory or in an on-site laboratory. The laboratory performing the analysis is determined by the OM and the QM.

Policy
The laboratory implements corrective action procedures for failed PT samples.

Procedure
The corrective action in the event of a PT failure should be a thorough review of the raw data from which the result was generated. Some of the items to investigate are instrument initial and continuing calibrations, results of the LCS prepared/analyzed with the PT sample(s), and results of any other QC indicators or instrument performance issues which may provide insight into the root cause of failure. A CAF is prepared which summarizes the PT failure(s) and the result of the investigation by the original analyst. If a problem is identified, the root cause of the problem and any corrective action taken is documented. The documentation is reviewed by the OM and submitted to the QM for review and approval. This documentation is maintained on file with the PT sample results.

Policy
The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

Procedure
PT samples are treated as typical samples in the normal laboratory process where possible, including the same preparation, calibration, quality control and acceptance criteria, sequence of analytical steps, number of replicates, and sample Log-In. In the event that the laboratory is certified/accredited for the same analyte(s) by more than one method, the PT sample may be split and analyzed by each method, (*i.e.*, 8270 Pesticides by GC/MS and 8141 Pesticides by GC/NPD).

25.5 Data Review

Policy
All data generated by the laboratory is reviewed for compliance with method, laboratory and, where appropriate, client requirements.

Procedure
Data review procedures and documentation of the review are specified in SOP "GEN-016, Data Review Procedures."

26.0 REPORTING OF RESULTS

Policy
The result of each test carried out is reported accurately, clearly, unambiguously, and objectively and complies with all specific instructions contained in the test method.

Policy
Procedures for the required steps and responsibilities for report generation, report review, and final authorization of reports are specified in SOP GEN-017, Report Preparation and Authorization.

Policy
For projects requiring NELAP accreditation, ECCS will report the data from each NELAP accredited test according to the requirements of the NELAP standard (See Table 26-1, Detailed Report Format). In addition, the test reports will comply with any specific reporting requirements of a state or regulatory jurisdiction, *i.e.*, Louisiana requires a Level III Expanded Report (See Table 26-1). Reports may also be customized based on a specific request made by the client.

Policy
Data are reported without qualification if they are greater than the lowest calibration standard, lower than the highest calibration standard, and without compromised sample or method integrity.

Policy
All results associated with the use of the NELAP accreditation must meet both the QC criteria of the method/SOP and the QA criteria of Section 25 of this QSM prior to release as a "Final Report." As required by SOP GEN-017 Report Preparation and Authorization, data released without the second party review must be annotated as a "Draft Report." With this proper documentation on the report, the data can be released as "NELAP accredited" prior to the second party review.

Policy
Technical justification is required any time that a test result is not used, and the justification must be documented. If a sample is reanalyzed, *i.e.*, to confirm prior results, and the results of both the initial and reanalysis are considered to be valid, the actual final value reported shall be determined by a documented procedure.

Procedure
If the RPD between the results of the initial analysis and the reanalysis is ≤ 20, report the result from the original analysis with no qualification. If the RPD between the results of the initial analysis and the reanalysis is >20, report the original result qualified as estimated. The origin of the reported value must be clearly documented in the raw data and in the sample narrative. RPD shall be calculated as follows:

$$\text{RPD} = \frac{|\text{R1} - \text{R2}|}{\left(\frac{\text{R1} + \text{R2}}{2}\right)} \times 100$$

Where: R1 = Result from the initial analysis
R2 = Result from the reanalysis

26.1 Test Reports

Policy
The report format has been designed to accommodate each type of test performed and to minimize the potential for misunderstanding or misuse of the report.

Procedure

- Each report shall have a unique identification, such as the LIMS work order number, on each page and a pagination system that ensures that each page is recognized as part of the test report and a clear identification of the end of the report, such as "Page 3 of 10."
- The reporting limits shall be adjusted for sample volume, sample dilution, sample percent solids, etc.
- Results for analytes determined from a dilution shall be clearly identified.
- The reporting basis, "wet weight" or "dry weight" shall be clearly identified on each report.
- A sample narrative shall be prepared, if applicable, which is specific to the work order that discusses analytical anomalies, QC sample results that fall outside of the control limits in the ECCS method, deviations from either the method or procedures in this QSM. The format of the sample narrative may change depending on the deliverables formats described below.
- The origin of the data, either the fixed-base laboratory or an on-site laboratory shall be clearly stated in the sample narrative.

26.1.1 ECCS offers four levels of reports to meet the specific reporting needs of each project. Table 26-1 contains the specific contents of each report format.

Policy
Test results shall be qualified in accordance with the SOP GEN-015 Data Qualification, unless other instructions are received via a site-specific quality assurance project plan (QAPP), the prevailing regulatory jurisdiction, or the client.

Policy
The final report contents shall be reviewed by the project manager or his designee prior to release.

Policy

For NELAP accredited work, each sample narrative will contain a statement to the effect that the results relate only to the samples and a certification that the results are in compliance with the NELAP Standards or, provide reasons and/or justification if they do not comply.

Table 26.1 ECCS Report Deliverable Formats

Summary Report
Analytical results (Summary LIMS Report Format) Chain of custody documents Surrogate recoveries- where applicable
Detailed Report (Standard Format; Used for NELAP Accredited Test Results[1])
Analytical results (Detailed LIMS Report Format) Sample narrative Chain of custody documents Surrogate recoveries- where applicable Method blank results[2] Blank spike results[2] Blank spike duplicate results- when applicable[2] Matrix spike/matrix spike duplicate summary[2] Sample duplicate results, where appropriate[2]
Expanded Detailed Report
Detailed report plus: GC/MS tuning- where applicable Internal standard area summary- where applicable Initial calibration summary Continuing calibration summary Other method related QC results, e.g. serial dilution results for metals.
Full Reportables
Expanded detailed report plus: All raw data including chromatograms, quantitation reports and spectra (where applicable) for each sample, QC sample, and standard. Miscellaneous data including instrument sequences, extraction/digestion logs, etc.

Notes:

[1] Specific state or federal regulatory jurisdictions may require additional information, i.e. Louisiana requires the Expanded Report.

[2] Quality control samples are reported on a per batch basis.

When necessary for interpretation of the results or when requested by the client, test reports may include the following additional information:

- deviations from, additions to, or exclusions from the test method, information on specific test conditions, such as environmental conditions, and any non-standard conditions that may have affected the quality of the results, and any information on the use and definitions of data qualifiers
- a statement of compliance/non-compliance when requirements of the QS are not met, including identification of test results that did not meet regulatory sample acceptance requirements, such as holding time, preservation, etc.
- when opinions and interpretations are included, the basis for the opinions and interpretations are documented. Opinions and interpretations, if applicable, are included in the Sample Narrative and are clearly identified as such
- identification of non-target analytes present in the sample, if any is noted as present by the analyst
- additional information which may be required by specific methods or client

Test reports obtained from subcontractors are provided as hardcopy or electronically as a PDF file. A copy of the subcontractor's report is delivered to the client.

26.2 Electronic Transmission of Results

Policy
All test results transmitted by telephone, fax, e-mail, or other electronic means comply with the requirements of this QSM and associated procedures to protect the confidentiality and proprietary rights of the client.

26.3 Amendments to Test Reports

Policy
Amendments or changes to a test report after it has been issued are made only in the form of a "Revised Report." Revised reports must meet all the requirements for the initial report and the requirements of this QSM.

Procedure
Test reports can be revised at the client's request, such as to add a previously unrequested analyte, or because of an error on the part of the laboratory. In both cases the original report is referenced.

Procedure
Revised Reports require the approval of the OM, the QM, or their designee. The QM maintains a list of Revised Reports, including the reason for the reissue and any corrective measures taken

to avoid similar occurrences in the future, if applicable.

Appendix A

Summary of ECCS

Validated Test Methods

Summary of ECCS Validated Test Methods

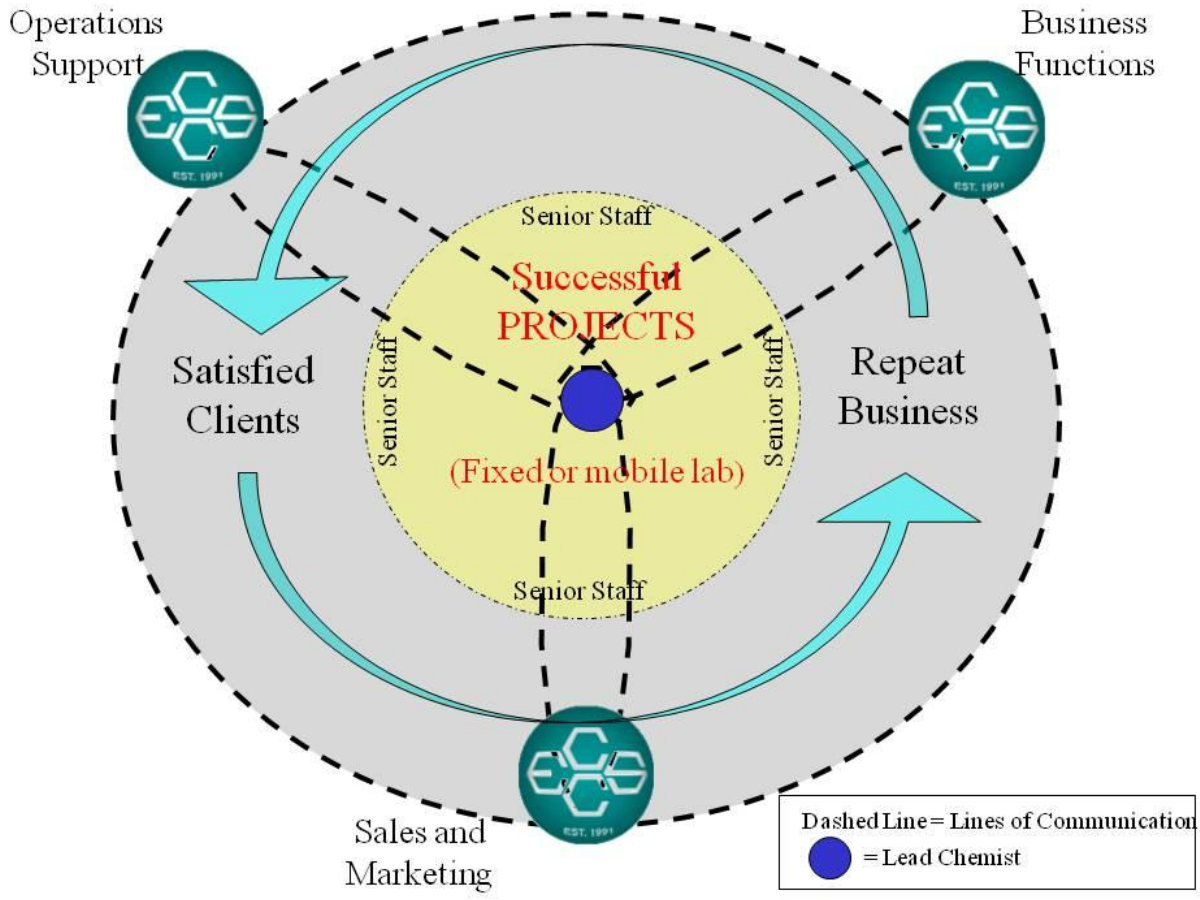
Description	EPA Method Reference	ECCS Reference	Certification/Accreditation					
			WI	LA	IL	KS	NC	NJ
Volatile Organics								
VOCs [GC/MS, Purge & Trap]	EPA 8260B	LAM-004	X	X	X	X	X	X
VOCs [GC/MS, Dir. Inject]	EPA 8260B	LAM-017	X	X	X	X	X	X
VOCs [GC/MS, Air]	EPA 8260B	NC						
Gasoline Range Organics [FID]	8015B	NC						
Diesel Range Organics [FID]	8015B	NC						
1,4-Dioxane [GC/MS SIM]	ECCS SOP	NC						
SVOCs								
Alkylated PAHs [GC/MS, SIM]	EPA 8270C	LAM-008	X	X	X	X	X	X
PAHs [GC/MS, Full Scan]	EPA 8270C	LAM-013	X	X	X	X	X	X
PAHs [GC/MS, SIM]	EPA 8270C	LAM-008	X	X	X	X	X	X
Organochlorine Pesticides [GC/ECD]	EPA 8081A	LAM-003	X	X	X	X	X	X
Neutral Extractable Pesticides [GC/MS]	EPA 8270C Modified	LAM-006	X	X	X	X	X	X
Organophosphate Pesticides [GC/NPD]	EPA 8141A	NC	X	X	X	X	X	X
Acid Extractable Herbicides [HPLC]	EPA 8321	LAM-001	X					
Acid Extractable Herbicides [GCMS]	EPA 8270C Modified	LAM-014	X	X	X	X	X	X
Pyrethroids [GC/MS]	EPA 8270C Modified	NC						
N-methylcarbamates [HPLC]	EPA 8318	NC	X					
Urea Carbamates [HPLC]	EPA 8321	NC	X					
PCBs [GC/ECD]	EPA 8082	LAM-005	X	X	X	X	X	X
Explosives [GC/NPD, GC/ECD]	EPA 8095	NC		X				
Explosives [HPLC/UV]	EPA 8330A	LAM-009						
Guanadine Nitrate [HPLC/EC]	ECCS SOP	NC						
Nitrocellulose	ECCS SOP	NC						
Perchlorate [LC/MS/MS]	EPA 6850	NC						
PETN [GC/MS, GC/ECD]	ECCS SOP	LAM-018						
Inorganics								
Metals [XRF]	EPA 6200	LAM-011						
Metals [ICP/MS, AA, GFAA]	EPA 6010B, 7000 Series	LAM-007		X				
Nitrate, Colorimetric [FIA]	EPA 353.2	LAM-002	X	X	X	X		
Nitrate+Nitrite, [ISE]	SM4500-NO3-G (17th Ed.)	LAM-015	X					
Nitrate+Nitrite, Colorimetric [FIA]	EPA 353.2	LAM-002	X	X	X	X		
Nitrite, Colorimetric [FIA]	EPA 353.2	LAM-002	X	X	X	X		
Ammonia, Colorimetric [FIA]	EPA 350.1	LAM-002	X	X	X	X		
Ammonia [ISE]	SM4500-NH3-D (20th Ed.)	LAM-015	X					
Anions by unsuppressed HPLC/EC	EPA 300.0	NC						
Misc.								
pH	EPA 9045C	NC						
Solids, Dry Weight	Solids, Dry Weight	GEN-007	X	X				
Total Suspended Solids	EPA 160.1	LAM-012	X					
TCLP Extraction Procedure	EPA 1311	NC						

NC = SOP not converted to LAM format at this time

Appendix B

Lead Chemist Relationships to Operations

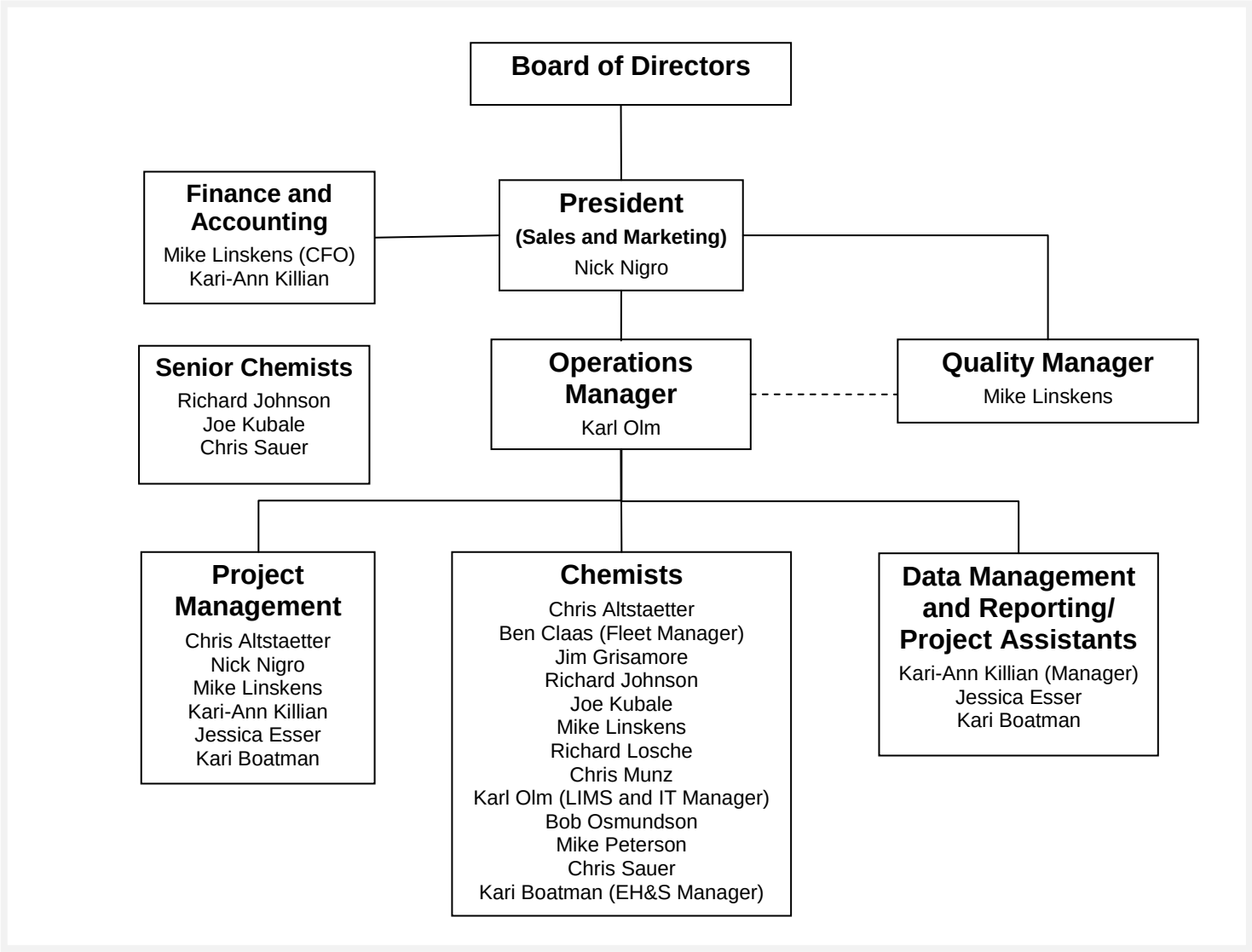
Lead Chemist Relationships to Operations



Appendix C

Organizational Chart

Organizational Chart



Appendix D

Corrective Action Form

CORRECTIVE ACTION FORM (CAF)

INITIALS:_____ DATE: _____

PROBLEM/DEFICIENCY: Test Method: _____

LIMS Work Order/

Sequence/Batch #	CCV(s)	Blank(s)	LCS(s)	MS/MSD	Surrogate(s)
_____	____	____	____	____	____
_____	____	____	____	____	____
_____	____	____	____	____	____

Other:_____

_____

_____

_____

Root Cause (Why or how did this happen?):_____

_____

_____

_____

Corrective Action:_____

_____

_____

_____

_____

Reviewer Approval:_____

PLEASE KEEP BOTH COPIES ATTACHED. They will be separated during the review.
White copy of form is to be filed with Batch raw data. Yellow copy of form goes to QA Officer.

Appendix E

Chain-of-Custody Form

Environmental Chemistry Consulting Services, Inc.
2525 Advance Road
Madison, WI 53718
608-221-8700 (phone)
608-221-4889 (fax)

CHAIN OF CUSTODY

Page ____ of ____

Lab Work Order #:

Mail Report To:
Company:
Address:
E-mail Address:
Invoice To:
Company:
Address:

Project Number:
Project Name:
Project Location:
Turn Around (circle one): Normal Rush
If Rush, Report Due Date:
Sampled By (Print):

Analyses Requested
Preservation Codes

Sample Description	Collection Date	Collection Time	Matrix	Total # of Containers							Comments	Lab ID	Lab Receipt Time

Preservation Codes
A=None B=HCL C=H_2SO_4
D=HNO_3 E=EnCore F=Methanol
G=NaOH O=Other (Indicate)

Relinquished By: Date: Time: Received By: Date: Time:
Relinquished By: Date: Time: Received By: Date: Time:

Matrix Codes
A=Air S=Soil W=Water O=Other

Custody Seal: Present/Absent Intact/Not Intact Seal #'s
Shipped Via:
Receipt Temp:
Temp Blank Y N

Download this form at www.eccsmobilelab.com. WHITE - REPORT COPY YELLOW - LABORATORY COPY PINK - SAMPLER/SUBMITTER Rev. 11/08

Appendix F

Job and Role Descriptions

Position Description

Title: President

Job Description Summary:
Provide leadership to position the company at the forefront of the industry. Develop a strategic plan to advance the company's mission and objectives and to promote revenue, profitability and growth as an organization. Oversee company operations to insure production efficiency, quality, service, and cost-effective management of resources.

Principal Accountabilities: (essential job functions and responsibilities)
1. Develop a strategic plan to advance the company's mission and objectives and to promote revenue, profitability, and growth as an organization.
2. Oversee company operations to insure production efficiency, quality, service, and cost-effective management of resources.
3. Plan, develop, and implement strategies for generating resources and/or revenues for the company.
4. Lead and/or oversee all company sales and marketing efforts.
5. Identify acquisition and merger opportunities and direct implementation activities.
6. Approve company operational procedures, policies, and standards.
7. Review activity reports and financial statements to determine progress and status in attaining objectives and revise objectives and plans in accordance with current conditions.
8. Evaluate performance of operations team and senior staff for compliance with established policies and objectives of the company and contributions in attaining objectives.
9. Promote the company through written articles and personal appearances at conferences and on radio and TV.
10. Represent the company at legislative sessions, committee meetings, and at formal functions.
11. Promote the company to local, regional, national, and international constituencies.
12. Other duties as assigned.

Required Experience and Skills:
1. Experience in strategic planning and execution. Knowledge of contracting, negotiating, and change management. Skill in examining and re-engineering operations and procedures. Experience in formulating policy, and developing and implementing new strategies and procedures. Ability to develop financial plans and manage resources. Ability to analyze and interpret financial data. Knowledge of public relations principles and practices. Knowledge of communication and public relation techniques. Ability to develop and deliver presentations. Ability to identify and secure funding/revenue sources.
2. Work requires professional written and verbal communication and interpersonal skills. Ability to communicate and interact with officials at all levels of government and to work effectively with a wide range of constituencies in a diverse community. Ability to motivate teams and simultaneously manage several projects.
3. This is normally acquired through a combination of the completion of a Masters Degree in Business Administration, Finance or Accounting and ten years of experience in a leadership role for a large division or company.

Position Description

Title: Operations Manager

Reports To:
President

Job Description Summary:
Provide for the day-to-day operations management of our Madison based laboratory; oversight/direction of Madison based rotating field staff; and maintenance and preparation of equipment and fleet (vehicles/trailers) in support of both Madison and field operations.

Supervisory Responsibilities:
Direct reports include the full-time laboratory staff. Log-In and reporting technicians and rotating field chemists are indirect reports.

Principal Accountabilities: (essential job functions and responsibilities)
1. Supervises laboratory operations to maintain quality and efficiency.
   a. Provides day to day direction to full time laboratory staff and rotating field staff to assure client and regulatory standards are being met.
   b. Strives to meet or exceed client turnaround time and quality expectations.
   c. In concert with QA officer, maintains QS and appropriate laboratory certifications and approvals.
   d. Actively seeks improvement to standard operating procedures and laboratory protocols.
   e. Reviews work, documents and provides both positive and developmental feedback, performance appraisals, on-going advice, council and recommendations for staff development and training of direct reports.
2. Provides day-to-day direction of rotating field staff.
   a. Maintains successful laboratory operations by scheduling available rotating field staff for laboratory work.
   b. Maintains a list of tasks to be accomplished in Madison and schedules available rotating field staff to complete the tasks.
   c. Provides on-going advice, council and recommendations for staff development and schedules cross-training of rotating field staff when applicable.
3. Maintains and prepares equipment/vehicles/trailers in support of both Madison and field operations.
   a. Maintains an adequate supply of equipment and supplies to support Madison and field operations including:
      (1) computer equipment and supplies
      (2) laboratory equipment and supplies
      (3) field operations infrastructure equipment and supplies
   b. Maintains and prepares vehicle and trailer fleet
   c. Implements fleet maintenance schedules including remote field vehicles
   d. Prepares vehicles/trailers for field operations as needed

Required Experience and Skills:
1. Bachelor of Science Degree
2. 15+ years of laboratory experience with an emphasis in pesticide residue analyses.
3. 10+ years of analytical project management experience.
4. 5+ years of ECCS on-site laboratory experience.

5. Excels in client management and service.
6. Strong interpersonal and oral and written communication skills.
7. Effective business management knowledge.

Position Description

Title: Quality Manager

Reports To:
President

Job Description Summary:
This position is responsible for developing, directing and coordinating a comprehensive QS program for use in ECCS' on-site and fixed-base laboratory operations; maintaining appropriate agency certifications and approvals; and daily review of fixed-base laboratory final reports.

The principal accountabilities described below are expected to require approximately 50% of the Quality Manager's time. The remainder of the time is expected to be filled with on-site and fixed-base laboratory chemist's duties. This position requires approximately 25% travel to on-site laboratory operations.

Supervisory Responsibilities:
No direct supervisory responsibilities. Indirect responsibility to work closely with staff in a mentoring role.

Principal Accountabilities: (essential job functions and responsibilities)

1. Develops, directs and coordinates the on-site and fixed-base laboratories' quality assurance programs.
   a. Monitors laboratory practices to promote a continuous quality improvement process.
   b. Coordinates review and approval of, or develops standard operating procedures and QA Manuals.
   c. Coordinates quarterly and yearly PE sample requirements.
   d. Performs quality audit of approximately 10 % of on-site and fixed-base laboratory data.
   e. Performs clear trail audits on a minimum of one fixed-base laboratory raw data file for each method on a yearly basis and a minimum of one on-site laboratory raw data file for each lead chemist on a yearly basis.
   f. Maintains organized quality assurance records by method and by analyst.
2. Establishes and maintains appropriate agency certifications and approvals.
   a. Maintains current knowledge of and is the authoritative source for various agency certification programs and rules.
   b. Maintains certifications and approvals with timely submittal of each program's yearly requirements such as: program fees; yearly PE results; changes to methods or updates to SOPs, etc.
   c. Coordinates any agency audit or visit.
3. Monitors fixed-base data quality by reviewing and signing each individual client report. Monitoring process should include at a minimum: review of associated raw data quality control results; check of sample reports for client provided field quality control samples (blanks, field duplicates); comparison to previous data, where possible; check appropriateness of test results in conjunction with likely DQO (potential false positives in a water sample).
4. Assists sales/marketing staff and project assistant with client development and proposal preparation.
5. Contributes to and monitors staff development and training protocols, competency standards and documentation requirements.
6. Provides semi-annual reports to management summarizing audit findings, agency issues, etc.

Required Experience and Skills:

1. Bachelor of Science Degree
2. 10+ years of laboratory experience with an emphasis in organics analyses.
3. 3+ years of ECCS on-site laboratory experience.
4. Excels in client interactions and service.
5. Strong interpersonal and oral and written communication skills.

Position Description

Title: Senior Chemist

Reports To:
Operations Manager

Job Description Summary:
This position requires the ability to function in several roles; as an analytical chemist; as a project manager; as a lead chemist; as a technical expert; as a mentor; while providing solution-oriented and quality objective-oriented analytical services to our clients in our fixed and on-site laboratories. This position requires approximately 50% travel to on-site laboratory operations.

Supervisory Responsibilities:
No direct supervisory responsibilities. Indirect responsibility to work closely with junior staff in a mentoring role.

Principal Accountabilities: (essential job functions and responsibilities)
1. Performs routine and non-routine laboratory testing of samples in accordance with the ECCS QS.
2. Maintains current knowledge and principles of EPA test methods and associated federal, state, and local regulations.
3. Maintains/repairs laboratory instrumentation and associated equipment .
4. Develops and validates new fixed or on-site laboratory methods and prepares SOPs.
5. Keeps equipment operating by following operating instructions; troubleshooting breakdowns; maintaining supplies; performing preventive maintenance.
6. Maintain daily logs, equipment record books, and computer database of sample results.
7. Complete projects by assisting project team; attending and participating in group and project meetings.
8. Interacts with clients in a positive manner.
9. Serves as a technical expert and mentor to junior staff.
10. Actively promotes quality improvement.
11. Maintains awareness of occupational safety hazards and is skilled in implementing appropriate safety practices and recognizing and communicating hazards.
12. Functions in the role of project manager (see Project Manager role description).
13. Functions in the role of lead chemist (see Lead Chemist role description).

Required Experience and Skills:
1. Bachelor of Science Degree
2. 10+ years of laboratory experience with an emphasis in instrumental analyses.
3. Excels in client interactions and service.
4. Strong interpersonal and oral and written communication skills.
5. Skilled in various instrumental computer applications

Position Description

Title: Chemist

Reports To:
Operations Manager

Job Description Summary:
This position requires the ability to function as an analytical chemist in our fixed and on-site laboratory operations while providing solution-oriented and quality objective-oriented analytical services to our clients. This position requires approximately 50% travel to on-site laboratory operations.

Supervisory Responsibilities:
No direct supervisory responsibilities.

Principal Accountabilities: (essential job functions and responsibilities)
1. Performs routine testing of samples in accordance with the ECCS QS.
2. Maintains current knowledge and principles of EPA test methods and associated federal, state, and local regulations.
3. Keeps equipment operating by following operating instructions; troubleshooting breakdowns; maintaining supplies; performing preventive maintenance.
4. Maintain daily logs, equipment record books, and computer database of sample results.
5. Complete projects by assisting project team; attending and participating in group and project meetings.
6. Interacts with clients in a positive manner.
7. Actively promotes quality improvement.
8. Maintains awareness of occupational safety hazards and is skilled in implementing appropriate safety practices and recognizing and communicating hazards.
9. Functions in the role of Lead Chemist, where appropriate (see Lead Chemist role description).

Required Experience and Skills:
1. Bachelor of Science Degree
2. 2+ years of laboratory experience with an emphasis in instrumental analyses.
3. Excels in client interactions and service.
4. Strong interpersonal and oral and written communication skills.
5. Ability to work with various instrumental computer applications

Position Description

Title: Technician

Reports To:
Operations Manager

Job Description Summary:
This position primarily provides support to the fixed-based laboratory in Madison, WI. Occasional support at an on-site laboratory operation may be required. This position requires approximately 10% travel to on-site laboratory operations.

Supervisory Responsibilities:
No direct supervisory responsibilities.

Principal Accountabilities: (essential job functions and responsibilities)
1. Maintain sample Log-In and tracking records.
2. Provide sample preparation support for pesticide analyses in accordance with standard operating procedures.
3. Washing laboratory glassware
4. Prepares and analyzes soil and water samples for VOCs and SVOCs in accordance with the ECCS QS.
5. Keeps equipment operating by following operating instructions; troubleshooting breakdowns; maintaining supplies; performing preventive maintenance.
6. Maintain daily logs, equipment record books, and computer database of sample results.
7. Complete projects by assisting project team; attending and participating in group and project meetings.

Required Experience and Skills:
1. Bachelor of Science Degree preferred
2. Detail oriented
3. Solid written and verbal communication capabilities
4. Interested in a career in environmental chemistry
5. Ability to work with a variety of computer applications.

Position Description

Title: Project Manager (PM)

Reports To:
President

Job Description Summary:
This position is responsible for support of sales/marketing staff in design and procurement of new projects, program management of all projects assigned. The PM will also often serve in the role of Lead Chemist and/or Project Assistant.

Supervisory Responsibilities:
The PM often has project-related team leader/supervisory responsibility for one or more lab staff and the Project Assistant.

Principal Accountabilities: (essential job functions and responsibilities)
1. Provides support to sales/marketing staff in design and procurement of new projects.
   a. Provides technical support in development of project scope of work.
   b. Offers proposal design insight by relating similar project and/or client experiences.
2. Accepts responsibility of program management of new projects.
   a. Responsible for primary project management level functions and communication with the client.
      (1) Develops working relationship with client, ensuring project success and maintaining opportunities for repeat business.
      (2) Documents all pertinent information in project file.
      (3) Tracks budget, prepares invoices and communicates with client in this regard.
   b. Understands technical and logistical needs of the project and works with the Operations Manager to select/schedule Lead Chemist(s) and project team.
   c. Understands the ECCS business model and strives to help contribute to company business success and profitability.
      (1) Schedules staff effectively to reduce airfare costs.
      (2) Makes sure project details necessary for invoicing are submitted on a timely basis.
   d. Interacts with client, Lead Chemist and other project personnel to monitor project issues and progress.
   e. Prepares and/or reviews final report to the client.
   f. Upon project completion, reviews project file and completes project assessment and QA closeout forms.
3. Provides training/mentorship of staff regarding all aspects of project development, execution and closure.

Required Experience and Skills:
1. Bachelor of Science Degree or equivalent experience.
2. 3+ years of instrumental laboratory experience.
3. Proven understanding of ECCS business model.
4. Superior client service skills.
5. Strong interpersonal and oral and written communication skills.

Position Description

Title: Lead Chemist

Reports To:
Operations Manager

Job Description Summary:
This position provides for the day-to-day operations management of one of ECCS' on-site laboratory projects. The Lead Field Chemist accepts project assignments from the operations manager; determines project requirements and client interaction role; prepares necessary equipment, supplies and vehicles (if required); conducts/supervises (when required) on-site testing operations; decommissioning the project's on-site laboratory unit; prepares final report; and provides feed back to the project manager.

Supervisory Responsibilities:
The lead chemist may have project team leader/supervisory responsibility for one or more field staff.

Principal Accountabilities: (essential job functions and responsibilities)
1. Determines project requirements and client interaction role in concert with project manager and operations manager.
2. Prepares necessary equipment, supplies and vehicles (if required).
   a. Schedule on-site laboratory and truck, as needed.
   b. Gather equipment and supplies required to meet the needs of the project. Checklists exist for some of the more common ECCS project types.
   c. Checks equipment to assure proper operation.
3. Conducts and supervises (when required) on-site testing operations.
   a. For single person short term projects, the lead chemist is required to be responsible for all aspects of the project: truck driver, analyst, sample Log-In and reporting technician, sample preparation technician, QA manager, metrology guru, maintenance department, and most importantly client services representative.
   b. Actively pursues relationship with client's field team and project manager.
   c. For multiple person short term projects, the lead chemist is also responsible for supervision of support staff.
   d. For multiple person longer term projects, the lead chemist role will fall on several individuals. In this case, the current on-site lead chemist has primary responsibilities for: on-site client interactions, coordination of on-site operational activities, being an analyst, training/mentorship of junior staff, maintaining the QS, and meeting the client's day-to-day needs.
4. Decommissioning the project's on-site laboratory unit.
   a. Properly dispose of all waste generated by the operation.
   b. Restock trailer based supplies.
   c. Clean the trailer countertops and floors.
   d. Report any equipment, trailer or truck issues to the field operations manager.
5. Prepares final report.
   a. For small projects, the lead chemist is responsible for preparing the final techmemo style report.
   b. For large projects, the lead chemist provides input to the project assistant for the final report.
6. Provides feedback to the project assistant.
   a. For any size project, the lead chemist is responsible for immediately contacting the project manager to provide feedback about any client concern.

b. For small projects, the lead chemist is responsible for scheduling a project exit interview with the project manager. The primary responsibility of the lead chemist during this interview is to pass on pertinent information about the execution of the project and the interactions with the client.
c. For large projects the lead chemist needs to communicate with the project manager on a regular basis to provide feedback about day-to-day operations, upcoming schedules, pertinent client interactions and any issue which is or could be a client concern.

Required Experience and Skills:

1. Bachelor of Science Degree or equivalent laboratory experience
2. 3+ years of instrumental laboratory experience.
3. Previous ECCS on-site lab experience.
4. Experience with or training for the need for superior client service.
5. Strong interpersonal and oral and written communication skills.

Position Description

Title: Project Assistant

Reports To:
Project Manager

Job Description Summary:
This position provides for continuous support of the Project Manager for fixed and on-site laboratory projects.

Supervisory Responsibilities:
None

Principal Accountabilities: (typical job functions and responsibilities)
1. Determines project peripheral needs with the Project Manager.
2. Provides for non-technical logistical issues during project planning, initiation and execution.
    a. Assures initial file information is complete including project set-up, analytical documentation, and client feedback forms.
    b. Acquires site location maps with driving directions.
    c. Prepares Health & Safety Plan information with directions to the nearest emergency health care facility.
    d. Makes hotel and transportation reservations.
    e. Coordinates generator rentals.
    f. Completes other tasks as assigned by the Project Manager.
3. Coordinates report generation.
4. Works with Operations Manager and Project Manager to assure project file and invoice are complete.
5. Upon direction from client services, contacts the client to obtain feedback.

Desired Experience and Skills:
1. Associates Degree or equivalent laboratory experience.
2. 2+ years of multi-dimensional project coordination experience.
3. Strong interpersonal, oral and written communication skills.
4. Proficiency in Microsoft Office suite and willingness to learn LIMS system.

Appendix C
Laboratory SOPs on CD

TRIMATRIX LABORATORIES

STANDARD OPERATING PROCEDURE

Purgeable Organic Compounds in Drinking Water by Capillary Column Gas Chromatography/Mass Spectrometry

EPA Method 524.2

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1.0 SCOPE AND APPLICATION

1.1 The identification and simultaneous measurement of purgeable organic compounds in finished potable water, raw source water or potable water at any level of treatment is performed by capillary gas chromatography/mass spectrometry.

1.2 The procedure is applicable to a wide range of volatile organics with sufficiently high volatility and/or low water solubility to be efficiently purged, as listed in Attachment 23.1.

2.0 PRINCIPAL METHOD REFERENCE

2.1 *Methods for the Determination of Organic Compounds in Drinking Water*, "Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry", Method 524.2, Revision 4.1, 1995

3.0 SUMMARY OF PROCEDURE

3.1 Volatile organic compounds and surrogates are purged from water by sparging an inert gas through the matrix.

3.2 Purged volatiles are trapped on a sorbent material which is then heated and backflushed with helium to desorb trapped components into a capillary column gas chromatograph (GC) interfaced to a mass spectrometer (MS).

3.3 The capillary column is temperature-optimized to separate and elute compounds for MS detection and identification.

3.4 An organic compound eluting from the column is identified by comparing the mass spectrum and elution time to standard spectra and elution times.

3.5 Reference spectra and retention times are obtained by measurement of calibration standards under the same conditions used for samples. The method of internal standards is used for all quantitation, including surrogate analysis.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to Attachment 23.1 for a complete list of volatile organic compounds.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-124, *Volatile Corrective Actions*, latest revision

5.2 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.3 TriMatrix SOP GR-10-115, *Manual Integrations*, latest revision

5.4 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

5.5 TriMatrix SOP GR-18-118, *Total Residual Chlorine*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Analytes with different mass spectra and non-interfering quantitation ions that coelute chromatographically can be identified and quantified from the same calibration mixture or sample. Some analytes with similar mass spectra cannot be individually evaluated from the same calibration mixture or sample unless each has a different retention time. Consequently, some structural isomers must be reported as an isomeric group or pair. For example, m-xylene and p-xylene are reported as an isomeric pair.

6.2 Volatile materials in the laboratory and impurities in the purge gas and/or concentrator can interfere with analysis.

6.2.1 Use only PTFE tubing and thread sealants.

6.2.2 Do not use flow controllers with rubber components in the purge and trap concentrator.

6.2.3 Reagent blanks must be monitored for contamination problems.

6.2.4 When interfering contaminants are observed in the laboratory blank, regenerate or replace the purge gas filter.

6.2.5 The technique of blank subtraction is not permitted.

6.3 Carryover may occur if a sample containing low volatile organic concentrations is analyzed immediately after a high concentration sample. After analysis of a high concentration sample, analyze multiple reagent blanks until carryover is not observed. Always rinse the sparge tube and injection syringes with laboratory reagent water at least twice.

6.4 Special precautions have been taken for methylene chloride analysis which includes the following:

6.4.1 The volatiles laboratory and sample storage area are isolated from the semi-volatiles extraction laboratory.

6.4.2 All volatiles GC carrier gas lines and purge gas tubing are stainless steel or copper.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity and carcinogenicity of reagents used in this procedure have not been precisely defined. Each chemical compound must be treated as a potential health hazard and exposure must be reduced to the lowest possible level, by all available means.

7.3 All neat chemicals must be handled in a hood wearing a laboratory coat, approved safety glasses, and disposable gloves.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Sample Collection, Dechlorination, and Preservation

8.1.1 **Samples known not to contain any residual chlorine**: Collect samples in 40 mL VOA vials pre-preserved with 0.5 mL (2 drops) of 1:1 HCl. Do not pre-rinse the vials prior to collection. Gently fill sample vials to almost overflowing, forming a meniscus at the top rim of the vial. To minimize volatiles loss, no air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. Invert the vial to confirm no headspace is present after sealing. If there is headspace or bubbles larger than 5-6 mm in diameter, remove the cap and slowly add additional drops of water until all air bubbles are eliminated. Tighten the vial cap, making sure the PTFE septum side is toward the sample.

8.1.2 **Samples known or suspected to contain residual chlorine, or for unfamillar samples:** Collect samples in 40 mL VOA vials containing 25 mg of ascorbic acid. Do not pre-rinse the vials prior to collection. Gently fill sample vials to the bottom of the neck of the vial. Add two drops of 1:1 HCl then continue to fill vial to almost overflowing, forming a meniscus at the top rim of the vial. To minimize volatiles loss no air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. Invert the vial to confirm no headspace is present after sealing. If there is headspace or bubbles larger than 5-6 mm in diameter, remove the cap and slowly add additional drops of water until all air bubbles are eliminated. Tighten the vial cap, making sure the PTFE septum side is toward the sample.

8.1.2.1 For these samples a third vial containing only 25 mg of ascorbic acid must be collected. Fill with sample as directed above, but do not add the HCl preservative. This vial will be used by the laboratory to test for the presence of residual chlorine in the event that trihalomethanes are detected in the sample.

8.1.3 When sampling from a water tap, open the tap and let the system flush until the water temperature has stabilized (usually about 10 minutes). Collect duplicate or triplicate samples as outlined above from the flow.

8.1.4 Collected samples must be stored and transported at 0–6° C from the time of collection to analysis. Package samples with sufficient ice to maintain 0–6° C until arrival at the laboratory.

8.2 Laboratory Sample Storage

8.2.1 Store samples at 0–6° C until analysis. The sample storage area must be free of organic solvent vapors.

8.2.2 Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and replaced unless otherwise specified.

8.3 Trip Blanks

8.3.1 Trip blanks must be handled with samples collected from the same general collection site at approximately the same time. Prepare trip blanks as follows:

8.3.1.1 At the laboratory, fill pre-preserved VOA vials with laboratory reagent water.

8.3.1.2 Seal the filled vials and transport in the cooler containing empty sample vials to the collection site.

8.3.1.3 Return to the laboratory after sample collection in the cooler containing full sample vials from the collection site.

8.3.1.4 Trip blanks must accompany collected samples during all shipping and/or storage before analysis.

8.3.2 Do not open a trip blank during sample collection, preservation, and/or storage.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Glassware and Hardware

9.1.1 Type A volumetric flasks, 10 mL, 50 mL, 100 mL, 250 mL, 1000 mL

9.1.2 Microsyringes, 10 uL, 25 uL, 50 uL, 100 uL, 1000 uL

9.1.3 Borosilicate vials with PTFE-lined septum screw caps, 20 and 40 mL

9.1.4 1.0 mL mini-inert vials for storage of prepared standards

9.1.5 Refrigerators and freezers for storage of prepared standards and samples

9.1.6 pH and chlorine test strips

9.1.7 Analytical balance capable of reading to 0.1 mg

9.1.8 Disposable Pasteur pipets and rubber pipet bulbs

9.2 Purge and trap/gas chromatograph/mass spectrometer system (procedure is applicable to the instrumentation described below).

9.2.1 Concentrator

9.2.1.1 Encon (Environmental Sample Technology) concentrator conditions:

Trap:	EST Vocarb 3000
Purge:	11 min at 38-42 mL/min
Dry purge:	1 min
Desorb preheat:	245° C
Desorb:	250° C for 2.0 min

Bake: 10 min at 260° C
Valve oven: 150° C
Transfer line: 150° C

9.2.2 Autosampler, Centurion (EST Analytical)

9.2.3 Hewlett Packard Model 6890 gas chromatograph equipped with electronic pressure control (EPC), split/splitless injection port, and capillary direct interface. Operating conditions:

Injector temperature:	200° C
Transfer line temperature:	280° C
EPC setting:	Constant flow mode at 0.8 mL/min
Ambient split ratio:	50:1
Column temperature program:	hold at 45° C for 10 min ramp to 73° C at 7° C/min hold at 73° C for 1 min ramp to 110° C at 10° C/min hold at 110° C for 1 min ramp to 220° C at 20° C/min hold at 220° C for 2 min

9.2.3.1 Chromatographic column: 20 m x 0.18 mm ID, 1 um film thickness, narrow bore capillary column, DB-624 (J&W Scientific) with capillary direct interface.

9.2.4 Mass Spectrometers

9.2.4.1 Hewlett Packard 5971A or 5973 quadrupole mass selective detector (MSD) capable of scanning from at least 35-650 amu every 2 seconds (or less) using a nominal 70 electron volts in the electron impact (EI) mode. Must produce a 4-bromofluorobenzene (BFB) mass spectrum meeting the ion abundance criteria in Attachment 23.2 when 25 ng of BFB are purged or injected onto the analytical column. Operating conditions are as follows:

9.2.4.1.1 Electron energy – 70 V nominal

9.2.4.1.2 Mass range – 35-300 amu

9.2.4.1.3 Scan time – 1.5 scans/sec

9.2.4.1.4 Source temperature – 230° C

9.2.5 Data Acquisition

9.2.5.1 The HP data acquisition system used is a DOS-based Chemstation with Enviroquant data analysis software (G1701DA Version D.1.02.16).

9.2.5.2 The system is capable of plotting EICPs and has a 120,000 compound NIST spectral library.

NOTE: Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Gas Chromatograph

10.1.1 Change gas scrubber traps yearly or as needed (record in the instrument maintenance logbook).

10.1.2 Clip or replace the column as needed (record in the instrument maintenance logbook).

10.1.3 Check the column head pressure daily (record adjustments in the instrument maintenance logbook).

10.1.4 Check the gas cylinder pressure daily.

10.1.5 Replace the injection port liner as needed (record in the instrument maintenance logbook).

10.2 Mass Spectrometer

10.2.1 Quadrupole

10.2.1.1 Drain and replace the rough pump oil every 6 months (record in the instrument maintenance logbook).

10.2.1.2 Check the diffusion pump oil annually and change if discolored or low (record in the instrument maintenance logbook).

10.2.1.3 Clean the ion source at least annually or more frequently as indicated by performance (record in the instrument maintenance logbook).

10.2.1.4 Replace the electron multiplier as needed (maximum voltage: 3000 V) (record in the instrument maintenance logbook).

10.2.1.5 Check the calibration gas and refill as necessary (record in the instrument maintenance logbook).

10.3 Purge and Trap Concentrators

10.3.1 Before initial use a Vocarb 3000 trap must be conditioned for at least one hour by baking at 260° C (record in the instrument maintenance logbook). If other trapping materials are substituted for the Vocarb 3000, follow the manufacturer's

recommendations for conditioning and submit the change to quality control for modification of the procedure.

10.3.2 Prior to daily use, the trap must also be baked for 10 minutes at 260° C. After any period of inactivity, the GC column must be run through the entire temperature program or ramped up to the final temperature and held for at least 15 minutes.

10.3.3 Check the purge flow weekly (record in the instrument run logbook).

10.3.4 Empty waste bottles and fill rinse bottles daily.

10.3.5 Check concentrator pressures weekly (record adjustments in the instrument maintenance logbook).

10.3.6 Check and if necessary fill the internal/surrogate standard module daily.

11.0 CHEMICALS AND REAGENTS

11.1 Organic free reagent water – laboratory reagent water prepared by reverse osmosis and deionization, further polished through the MilliQ System, followed by distillation and a 30-minute nitrogen purge.

11.2 Methanol, purge and trap grade

11.3 Neat standard materials with at least 96% purity or commercially purchased and certified stock solutions

11.4 Hydrochloric acid, certified ACS or better

12.0 STANDARDS PREPARATION

12.1 All standards must be recorded in the laboratory information management system (Element). Information including the vendor, lot number, concentration, and purity is recorded. Each standard is given a unique ID number. All vendors and standard concentrations provided below are recommended and subject to change; however, this procedure will be updated when a change is made.

12.2 Hold Time and Storage Requirements

12.2.1 Unopened ampules of purchased standard solutions and neat materials used in stock standards preparation, may be kept for up to 12 months from the date received, or as directed by the manufacturer.

12.2.2 Due to evaporative loss and reactivity, laboratory prepared stock and nongaseous intermediate stock standards are given an expiration date of six months. An intermediate stock gas standard requires weekly replacement. Within expiration dates, all standards must not be kept past acceptable performance.

12.2.2.1 Continuing calibration standards must meet the calculated percent difference or drift criteria of section 14.3.3 to be acceptable. This requirement applies to all analytes, both gaseous and non-gaseous. Once this criterion has been exceeded the intermediate stock and/or the stock must be replaced, unless exceedance is due to degradation of instrument performance.

12.2.2.2 Dichlorodifluoromethane will generally be the first gaseous compound to fail.

12.2.3 Store all opened ampules and prepared standards with minimal headspace, protected from light, in 1 mL mini-inert sealed screw-cap vials at -10° C or less. Purchased standards are stored as instructed by the manufacturer. Store standards separately from samples.

12.3 Stock Standard Preparation

12.3.1 Stock standards are prepared from neat materials and/or certified solutions. All purchased solutions must be accompanied by a Certificate of Analysis. All certificates of analysis must be forwarded to the quality assurance department. Two separate standard sources must be obtained. This requirement applies to both purchased standards and neat compounds. One set will be used to prepare calibration standards, laboratory fortified blanks (blank spikes), and matrix spikes, and the other will be used for second-source calibration verifications. The two must be prepared from dissimilar or entirely separate sources. It is permissible to purchase different lots from the same vendor; however, a dissimilar lot means preparation from dissimilar chemical lots, not prepared twice from the same lot. It is not necessary to purchase/prepare internal standard/surrogate mixes from separate sources.

12.3.2 When preparing stock standards from neat materials the mass weighed out may be used without mathematical correction when the compound has a purity greater than or equal to 96%. Prepare stock standard solutions in methanol.

12.3.3 Purchased Commercial Stock Standards:

12.3.3.1 The following standards are combined to make Stock Standard I

12.3.3.1.1 Mix A: Volatile Organic Compounds - Liquids at 2.0 mg/mL in methanol

12.3.3.1.2 Mix B: Volatile Organic Compounds - Gases at 2.0 mg/mL in methanol

12.3.3.1.3 Mix C: Acrolein & Acrylonitrile at 2.0 mg/mL in methanol

12.3.3.1.4 Mix D: Ketone Mix at 2.0 mg/mL in methanol

12.3.3.1.5 Mix E: Custom Additions to Method 524.2 at 2.0 mg/mL in methanol

12.3.3.1.6 Mix F: Dichlorofluoromethane at 1.0 mg/mL in methanol

12.3.3.1.7 Mix G: Internal Standard and Surrogate Standard Mix at 5.0 mg/mL in methanol

12.3.3.1.8 Mix H: BFB Standard Mix at 2.0 mg/mL in methanol

12.3.3.2 The following standards are combined to make Stock Standard II

12.3.3.2.1 Mix A: Appendix IX Standard @ 2.5 mg/mL in methanol

12.3.3.2.2 Ethyl Methacrylate at 1.0 mg/mL in methanol

12.3.3.2.3 Isopropanol at 1.0 mg/mL in methanol

12.3.3.2.4 2-Chloro-1,3-butadiene (chloroprene) at 2.0 mg/mL in methanol

12.3.3.2.5 N-butyl acetate at 2.0 mg/mL in methanol

12.3.3.2.6 Methyl Methacrylate at 2.0 mg/mL in methanol

12.4 Intermediate Stock Standards

12.4.1 Intermediate Stock Standard I

12.4.1.1 This solution is prepared in a 1.0 mL mini-inert vial. Expiration is one week from preparation. Add 650 uL of methanol to the mini-inert vial. Add the following volumes from the mixes specified in section 12.3.3.1 to produce 1.0 mL of a 100 mg/L intermediate stock standard

12.4.1.1.1 50 uL of mixes A, B, C, D, and E

12.4.1.1.2 100 uL of mix F

12.4.2 Appendix IX Intermediate Stock Standard II

12.4.2.1 This standard is also prepared in a 1.0 mL mini-inert vial. Expiration is 6 months from preparation. Add 610 uL of methanol to the mini-inert vial. From the mixes specified in section 12.3.3.2 add the following quantities to produce 1.0 mL of a 100 mg/L intermediate stock standard:

12.4.2.1.1 40 uL of Mix A

12.4.2.1.2 50 uL of 2-Chloro-1,3-butadiene

12.4.2.1.3 100 uL of Isopropanol

12.4.2.1.4 100 uL of Ethyl methacrylate

12.4.2.1.5 50 uL of Methyl methacrylate

12.4.2.1.6 50 uL of N-Butyl acetate

12.5 Working Standards

12.5.1 Internal Standard/Surrogate Intermediate Stock Standard III

12.5.1.1 This standard is prepared at a concentration of 200 mg/L. Fill a 25 mL volumetric flask approximately three-quarters full with methanol. Add 1.0 mL of mix G from section 12.3.3.1.7 and dilute to volume with methanol. Cap and invert three times to ensure proper mixing. Discard the contents in the neck of the flask and transfer the remaining contents to a 20 mL PTFE-capped vial. The purge and trap auto sampler adds this standard automatically to samples during the purge cycle.

12.5.2 BFB Intermediate Stock Standard IV

12.5.2.1 This standard is prepared in a 1.0 mL mini-inert vial. Add 990 uL of methanol to the mini-inert vial. Add 10 uL of mix H (section 12.3.3.1.8) to produce 1.0 mL of a 20 mg/L intermediate stock standard. This standard is used for performing BFB tunes.

12.5.3 Initial calibration and continuing calibration verification standards are prepared by spiking different volumes of intermediate stock standard into a volumetric flask containing reagent water. The initial calibration standards will not contain any of the preservatives used in the samples. They are prepared fresh prior to every use. Continuing calibration verification standards are typically prepared at 5.0 ug/L except for the Appendix IX compounds which are prepared at 50 ug/L. All continuing calibration verification standards will contain the same preservatives used in the samples being analyzed. Default spike volumes used in preparing the initial and continuing calibration verification standards are discussed below. Stock volumes necessary to prepare other concentrations can be calculated using the following formula:

$$V_s = \frac{C_f \times V_f}{C_s}$$

where:
V_s = Volume of stock standard to inject (uL)
C_f = Final concentration of working standard (ug/L)
V_f = Final volume of working standards (mL)
C_s = Concentration of stock standard (ug/mL)

12.5.3.1 Initial calibration standards are prepared by diluting Intermediate Stock Standard I into volumetric flasks containing reagent water as follows:

0.5 ug/L =	1 uL into 200 mL
1 ug/L =	1 uL into 100 mL
5 ug/L =	2.5 uL into 50 mL
10 ug/L =	5 uL into 50 mL
20 ug/L =	10 uL into 50 mL
40 ug/L =	20 uL into 50 mL

12.5.3.2 Appendix IX Initial calibration standards are prepared by diluting Intermediate Stock Standard II into volumetric flasks containing reagent water as follows:

5 ug/L = 2.5 uL into 50 mL
25 ug/L = 12.5 uL into 50 mL
50 ug/L = 25 uL into 50 mL
100 ug/L = 50 uL into 50 mL
200 ug/L = 100 uL into 50 mL
400 ug/L = 200 uL into 50 mL

NOTE: All continuing calibration and quality control standards **must** be prepared and transferred into a pre-preserved vial as stated in Section 12.5.3 in order to match sample preservation. Preservatives must be from the same lot as those used to preserve the samples.

12.6 Matrix Spike Standards

12.6.1 Sample matrix effects are typically not observed in drinking water samples; therefore, a matrix spike and matrix spike duplicate are not required by the method. Matrix spikes are only analyzed when specifically requested by the client.

12.6.1.1 Matrix spikes contain the same analytes, typically at the same concentration, as the CCV/blank spike. If a matrix spike is requested, use the same intermediate stock standard used to prepare the calibration standards to spike the sample.

12.6.1.2 To prepare a matrix spike and matrix spike duplicate, add 100 mL of sample to a 100 mL volumetric flask. Add 5 uL of Intermediate Stock Standard I and mix. Pour the sample into two 40 ml vials.

12.7 Blank Spike (BS) Standards

12.7.1 The primary source intermediate stock standard is used for blank spikes. Unless otherwise specified by the client, the correctly preserved continuing calibration standard analyses will also be used as the BS. The same analytes used in the matrix spike must be used for the BS. Additional analytes are reported if requested by the client. Report the same analyte list for the matrix spike and the BS.

12.7.2 To prepare a blank spike fill a 100 mL volumetric flask with reagent water. Add 5 uL of Intermediate Stock Standard I and mix. Pour the sample into a 40 mL vial.

NOTE: All continuing calibration and quality control standards **must** be prepared and transferred into a pre-preserved vial as stated in Section 12.5.3 in order to match sample preservation. Preservatives must be from the same lot as those used to preserve the samples.

13.0 SAMPLE PREPARATION

13.1 Samples analyzed on the Centurion autosampler do not require further preparation. The 40 mL vial is loaded directly into the autosampler for analysis.

13.2 If a sample dilution is required, dilute into an appropriate volumetric flask and transfer to a properly preserved 40 mL VOA vial. Keep all dilutions in the upper half of the calibration curve.

13.3 The autosampler will withdraw 5.0 mL from the 40 mL vial and add internal standard/surrogate prior to analysis.

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration

14.1.1 Initial calibration is performed through the analysis of a 6-point curve at concentrations of 0.5, 1.0, 5.0, 10, 20, and 40 ug/L. Refer to section 12.5.3 for calibration standards preparation. Do not add HCl or ascorbic acid to the standards used in the initial calibration curve.

14.1.2 Load the calibration standards into the autosampler and initiate the run in accordance with the instrument operating instructions.

14.1.3 Adhere to all instrumental instructions for purging and data acquisitions.

14.1.4 For compounds not listed in Attachment 23.1, choose a major ion that is dissimilar to potential coeluting or interfering ions as the quantitation ion. Otherwise, the ion used must be consistent with Attachment 23.1.

14.1.5 Calculate a response factor for every analyte and surrogate in the curve as specified in Section 16.1 Verify that all RFs are $\geq$0.01. RFs less than 0.01 are not acceptable and the data point must be rejected.

14.1.6 Determine the %RSD of each analyte as specified in Section 16.2. Ideally quantitation will be performed using the average response factor. The %RSD must be within ±20% to quantitate in this manner. If an analyte cannot achieve this criterion, reprocess as a linear or second-order regression curve. The coefficient of determination (r^2) value for a regression curve must be at least 0.990 to be acceptable. Also use linear or second-order regression for analytes with less than 20% RSD that obviously do not regress through origin.

14.1.7 Initial calibration curves expire seven days from the analysis date and time of the BFB analyzed with the previous initial calibration curve. Initial calibration will be required sooner if the continuing calibration verification fails.

14.2 Initial Calibration Verification

14.2.1 Initial calibration verification must be performed immediately following every initial calibration by running a second-source calibration verification (SCV) standard at 5 ug/L.

14.2.2 To prepare the SCV, fill a 100 mL volumetric flask with reagent water. Add 5 uL of the second source Intermediate Stock Standard I and mix. Pour the sample into a 40 mL vial.

14.2.3 The SCV must be run after each initial calibration.

14.2.4 The SCV must include all targeted analytes.

14.2.5 Acceptance criteria are ±30% of the expected value unless otherwise specified in the laboratory information management system. Acceptable validation of every analyte in the SCV must be achieved prior to sample analysis for that analyte.

14.2.6 The SCV does not require preservation.

14.3 Continuing Calibration

14.3.1 Continuing calibration verifies instrument stability and confirms accuracy of the initial calibration curve. Unlike the standards used in the initial calibration curve, the continuing calibration standard must contain the same preservatives as those used in the samples being analyzed in the analytical batch. Continuing calibration is accomplished through the analysis of a 5.0 ug/L standard containing every surrogate and analyte of interest.

14.3.2 Determine whether internal standard and surrogate quantitation ion areas have decreased by more than 30% relative to areas measured in the most recent calibration verification or by more than 50% relative to initial calibration areas. If any area has decreased by greater than these percentages, adjustments must be made to restore the calibration accuracy.

14.3.3 If the analyte or surrogate in the curve was processed using average RF, determine the RF in the continuing calibration standard and calculate its percent difference from the average curve RF (Section 16.3). The analyte or surrogate RF in the CCV must be within ±30% of the initial calibration RF. If the curve was processed as linear or second order regression, quantitate the CCV as a sample. The analyte or surrogate must recover within ±30% of the true value (percent drift). If either of these criteria are not met, corrective action must be taken, up to and including recalibration.

14.3.4 Calculate a response factor for every analyte and surrogate as specified in Section 16.1. Verify that all RFs are ≥0.01. RFs less than 0.01 are not acceptable and the continuing calibration data point must be rejected.

15.0 ANALYTICAL PROCEDURE

15.1 Prior to daily use, the trap must also be baked for 10 minutes at 260° C. After any period of inactivity, the GC column must be run through the entire temperature program or ramped up to the final temperature and held for at least 15 minutes.

15.2 BFB Analysis and Tuning Criteria

15.2.1 Every analytical batch must begin with the analysis of 25 ng of 4-Bromofluorobenzene (BFB). Acquisition of all subsequent analyses must begin within 12 hours of the acquisition time of BFB. No analysis may begin until a BFB spectra passing all the tuning criteria given in Attachment 23.2 is achieved.

15.2.2 Spike 25 uL of the intermediate BFB tuning standard (Section 12.5.2.1) into 100 mL of reagent water mix and pour into a 40 mL vial. The autosampler will measure 5.0 mL from the 40 mL vial to purge.

15.2.3 Use the following criteria in sequential order to evaluate the tune.

15.2.3.1 All BFB tunes must initially be evaluated using the contract laboratory program BFB tuning procedure which takes the scan average at the peak apex and scans immediately before and after the peak apex, followed by background subtraction.

15.2.3.2 If the above does not generate an acceptable BFB tune, take the scan average of the entire peak, followed by background subtraction.

15.2.3.3 If the above two options do not yield an acceptable BFB tune, use a single scan at the apex to evaluate the tune.

15.2.4 If the above procedures do not produce an acceptable BFB tune, corrective action is required.

15.2.4.1 Re-inject and reanalyze the tuning standard.

15.2.4.2 Prepare a new BFB working standard and re-inject and reanalyze.

15.2.4.3 Manually adjust the tuning acquisition parameters or by performing a new autotune followed by another BFB analysis.

15.2.4.4 The BFB analysis must pass the tuning criteria before subsequent analyses may begin (Attachment 23.2).

15.3 Calibrate or verify the calibration of the instrument as directed in Section 14. The continuing calibration standard is typically also processed as a BS.

15.4 Blank Analysis

15.4.1 The system must be demonstrated to be free from contamination and interferences by running at least one laboratory reagent water blank at this point during every 12-hour shift. Blanks will be run more frequently if contamination is suspected from a high-level sample or if laboratory contamination is in question. Blanks must be carried through every step in sample preparation and measurement and must be spiked with internal standards, surrogates, and anti-foam when used in the sample batch. The blank must also contain the same preservatives as those used in the samples being analyzed in the analytical batch.

NOTE: Blank subtraction is NOT allowed.

15.5 When samples are analyzed for Trihalomethanes, in addition to all other quality control samples and requirements a 1.0 ug/L Minimum Reporting Limit Standard must be analyzed prior to sample analysis. The standard must recover within 50 – 150%.

15.6 Only after successfully completing Sections 15.1 through 15.4 (or 15.5) can sample analysis begin.

15.7 Sample Analysis

15.7.1 All samples must be analyzed using the Centurion autosampler by placing the 40 mL vial the sample was collected in directly into the autosampler. The autosampler will remove 5.0 mL of sample and transfer it to the purge vessel. Internal and surrogate standards will be added by the autosampler. The purge sequence will start automatically. At the end of the purge cycle, analyte will have collected in the trap, ready for desorption to the gas chromatograph for separation and quantitation.

15.7.2 System operating conditions must be the same as those used for running the calibration.

15.7.3 All samples and standards must be warmed to ambient temperature before use. All samples, standards, and quality control samples must be spiked with the internal standard/surrogate standard mix.

15.7.4 Samples analyzed after a highly concentrated sample or saturated ions must be re-analyzed if carryover is suspected.

15.7.5 If any sample analysis has an analyte concentration exceeding highest calibration standard, the sample must be re-analyzed at a dilution (section 15.8) or data must be qualified.

NOTE: Regardless of how the sample was collected (with or without the addition of ascorbic acid), if any trihalomethane (bromoform, chloroform, bromodichloromethane, or dibromochloromethane) is detected, the sample must be tested for the presence of residual chlorine. Test the sample by dipping a chlorine test strip (Hach #2745050 or equivalent) into the sample and comparing to the color-comparison scale. Use the third (ascorbic acid/no HCl) vial collected specifically for this test when available. If the sample was not collected with ascorbic acid there will not be a third vial available. Use the unanalyzed HCl preserved vial. If residual chlorine is detected in the sample, LIMS data qualifier GN037 or GN038 must be applied to the sample results as appropriate.

GN037 - Sample was collected without the addition of the dechlorinating agent, ascorbic acid and residual chlorine was detected in the sample after receipt by the laboratory. All reported values, including non-detectable results are considered estimated.

GN038 - Residual chlorine was detected in the sample after receipt by the laboratory. All reported values, including non-detectable results are considered estimated.

15.8 Dilutions

15.8.1 Any sample with an analyte concentration greater than the highest calibration point must be diluted and run again. Final dilutions must contain the same preservatives as the original samples (intermediate serial dilutions do not require preservation). All dilutions must keep the peak response of previously saturated peaks in the upper half of the calibration range. All dilution steps must be performed without delay to avoid analyte loss.

15.8.2 Prepare all dilutions in 50 or 100 mL volumetric flasks. Do not use sample volumes of less than 100 uL. Serial dilutions may be required for extremely high concentrations.

15.8.3 Calculate the approximate volume of reagent water to add to the volumetric flask and add slightly less.

15.8.4 Using a clean micro syringe, Inject the appropriate sample volume into the flask. Dilute to the mark with laboratory reagent water. Cap the flask and invert three times, dispose of the contents in the neck of the flask. Carefully transfer an aliquot to a 40 mL VOA vial.

15.8.5 If the original sample contained ascorbic acid, transfer the dilution to a VOA vial that contains ascorbic acid. Fill the vial nearly to the top, add two drops of HCl, then add more sample to completely fill the vial so that it is headspace free.

15.8.6 If the original sample did not contain ascorbic acid, transfer the dilution to a VOA vial pre-preserved with HCl. Completely fill the vial so that it is headspace free.

15.8.7 In an effort to keep preservative lots consistent, obtain dilution vials and preservatives from the bottle prep laboratory area.

16.0 CALCULATIONS/DATA HANDLING

16.1 Response Factor Calculation

16.1.1 Calculate a response factor (RF) for each analyte and surrogate using the following equation:

$$RF = \frac{(Ax)(Qi_s)}{(Ai_s)(Q_x)}$$

where:

Ax = integrated abundance of the quantitation ion of the analyte
A_{is} = integrated abundance of the quantitation ion of the internal standard
Q_x = quantity of analyte purged in ng or concentration units
Q_{is} = quantity of internal standard purged in ng or concentration units

16.2 Percent Relative Standard Deviation (%RSD)

16.2.1 Using the initial calibration RFs, calculate %RSD using the following equation:

$$\%RSD = \frac{(SD)100}{X}$$

where:
RSD = Relative Standard Deviation
X = mean of the five curve points for a compound
SD = standard deviation of average RFs for a compound

16.3 Percent Difference

16.3.1 Calculate percent difference using the following equation:

$$\% \text{ Difference} = \frac{RF_{CCV} - RF_{Average}}{RF_{Average}} \times 100$$

where:
RF_{CCV} = the Response Factor determined in the CCV
$RF_{Average}$ = the average Response Factor from the curve

16.4 Qualitative Identification

16.4.1 All target analytes are identified by the one or more of the following.

16.4.1.1 Comparison of the sample mass spectrum with the mass spectrum of a standard reference spectrum.

16.4.1.2 Elution of the sample component at the same GC relative retention time (RRT) as those of the standard component retention time.

16.4.1.3 Experimentally obtained mass spectra:

16.4.1.3.1 Mass spectra for standard reference must have been obtained on the same GC/MS system. These standard reference spectra are obtained through analysis of calibration standards.

16.4.1.3.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. Relative intensities of these ions must agree within ±20% between the standard and sample spectra. For example, an ion with an abundance of 50% in the standard spectra must be between 30% and 70% abundance in the sample spectra.

16.4.1.4 Retention Time

16.4.1.4.1 The sample component relative retention time (RRT) must compare within ±0.06 RRT units of the RRT of the standard component.

16.4.1.4.2 If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles for ions unique to the component of interest.

16.5 Quantitative analysis:

16.5.1 Quantitation is performed using the same technique used during initial calibration, average response factor or regression. Use the analyte and internal standard quantitation ions as specified in Attachment 23.1. Use the following equation when quantitating with the average response factor.

$$\frac{A_x \times C_{is}}{A_{is} \times RF} \times DF = ug/L$$

where:
A_x = Area of the characteristic ion for the target compound
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard in ug/L
RF = Average Response Factor
DF = Dilution factor

16.6 Refer to TriMatrix SOP GR-10-115 for instructions on how to perform a manual integration.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for data quality and for filling in all documentation and paperwork correctly. It is important to document analysis by correctly filling in, handing in and archiving all paperwork correctly.

17.2 LIMS data transfers must be in accordance with TriMatrix SOP GR-10-123. If internal chain-of-custody is required, it is very important that a CoC form be filled in correctly and completely.

17.3 All run, maintenance, CD archival and standard logs must be filled in completely and correctly. Corrections on hardcopy must be made with a lineout (not a writeover) and must be initialed and dated. Blank lines in run logbooks must be Z'd out.

17.4 All calibration run hardcopies must be archived appropriately.

17.5 All other analysis documentation must be archived appropriately for ease in retrieval.

18.0 QUALITY ASSURANCE

18.1 Blanks

18.1.1 Acceptance criteria for the method blank is that all analytes of interest must be below the minimum reporting limit for all samples analyzed in that analytical batch, except for common lab contaminants (methylene chloride or acetone), which may be 5X the reporting limit or as allowed by the project.

18.2 Internal Standards

18.2.1 Internal standard responses and retention times in all runs following the calibration verification standard must be evaluated during or immediately after data acquisition. The retention time for all internal standards must be within ±30 seconds from the current 12-hour continuing calibration verification. The area of the quantitation ion for all internal standards must not deviate from the response in the most recent CCV by more than ±30% and must not deviate by more than ±50% from the average area measured during the initial calibration.

18.2.2 If at any time an internal standard fails the -50 to +100% area criteria, the ability to accurately quantitate an analyte is reduced. For that reason every effort must be made to prevent the failure of an internal standard, including dilution and re-analysis of the sample. Refer to TriMatrix SOP GR-03-124 for corrective action when an internal standard fails. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for malfunctions, and corrections made as appropriate. When corrections are made, re-analyze all samples analyzed while the system was malfunctioning.

18.3 Surrogates

18.3.1 All samples must be spiked with surrogate standards. Until thirty samples of a given matrix have been analyzed, a maximum recovery window of 70-130% will be used. Once thirty samples of a given matrix have been analyzed, in-house recovery limits will be generated and used.

18.3.2 Calculate upper and lower control limits for each surrogate standard as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where:
p = average percent recovery
s = standard deviation of the percent recovery

18.3.3 Two standard deviations will be used when three standard deviations give a negative lower control limit.

18.3.4 If recovery is not within limits, TriMatrix SOP GR-03-124 outlines if or how data is qualified. If many samples are out-of-control for no apparent reason, the mass spectrometer needs to be inspected for malfunctions and corrections made as appropriate. When corrections are made, re-analyze all samples analyzed while the system was malfunctioning.

18.4 Blank Spikes (BS)

18.4.1 A blank spike (BS) and blank spike duplicate (BSD) is required with each 12-hour analytical batch or every 20 samples, whichever is more frequent and must contain all targeted analytes.

18.4.1.1 It is taken from results of the daily 5.0 ug/L CCV..

18.4.1.2 The blank spike serves as a check of method performance and to determine if the laboratory is capable of making accurate measurements. It must contain the same preservatives as the samples being analyzed.

18.4.1.3 The blank spike is run at the beginning of the 12-hour shift, immediately after the BFB tune, and is followed by the method blank (BLK).

18.4.1.4 Calculate blank spike recoveries and compare to the method control limits (±30% is the maximum allowable control window). Stop the analysis and correct any problem if recoveries are outside the control limits.

18.4.1.5 Any sample analyzed in a batch with a failing blank spike must be re-analyzed for the failing analyte(s). If this is not possible, all sample data for the failed analyte(s) must be qualified as estimated. However, for the recovery of any analyte that exceeds the upper control limit, associated sample results below the reporting limit need not be qualified.

18.4.1.6 If the BS is out-of-control, the problem must be immediately identified and corrected before further samples for that analyte can be run. Failure of any target analyte in a BS will require some sort of corrective action, depending on the failed parameter. Every effort will be made to determine the reason for the failure and appropriate actions will be taken. TriMatrix SOP GR-03-124 for corrective action details how to proceed if a BS fails.

18.4.2 At least quarterly, evaluate replicate blank spike data to determine laboratory measurements precision. Update ongoing control charts to document data quality and calculate the maximum precision limit.

18.5 Matrix Spikes and Matrix Spike Duplicates (MS/MSD) are prepared following the same procedure as that used to prepare the BS. Substitute sample matrix for the reagent water used in the BS. A MS and/or MSD are performed when requested by the client.

18.5.1 Recovery limits for all analytes are 70-130%. The maximum precision acceptance limit is 20% relative percent difference (RPD).

18.5.2 Corrective action for any precision and/or accuracy failure should be performed in accordance to the procedures specified in TriMatrix SOP GR-03-124.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before analysis of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an initial demonstration of capability (IDC) study. While a demonstration of capability is not instrument dependent, it is required on every instrument

running samples to demonstrate the ability of each instrument to generate acceptable accuracy and precision.

19.2 Prepare a blank spiking standard independent of the calibration standards containing all targeted analytes. Spike 1.00 mL into each of four, 100 mL aliquots of water. Extract and derivatize as samples, following every step in the preparative procedure.

19.3 Analyze as samples, following every step in the analytical procedure.

19.4 Input the four results to the IDC spreadsheet located on the laboratory intranet library to calculate average recovery and relative standard deviation (RSD). Percent recovery must be within blank spike acceptance limits and precision must be less or equal to 20% RSD.

19.5 If all criteria are acceptable for all targeted analytes, the IDC study is complete. The analyst and system are authorized to run samples.

19.6 If any criterion fails, locate and correct the source of the problem and repeat the study successfully for the failed analyte.

19.7 Repeated failure will confirm a general problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, correct the procedure and/or techniques used and repeat the study successfully.

19.8 Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

19.9 A demonstration of capability study must be completed annually by one of the following approaches:

19.9.1 By repeating the IDC study.

19.9.2 By using four consecutively run blank spikes from routine sample analysis if performed exclusively by the analyst.

19.9.3 By exclusively and successfully analyzing a blind performance testing sample during the course of routine sample analysis.

19.9.4 By using the last four results from an MDL study if run exclusively by the analyst. ONLY the last four results may be used.

19.10 A method detection limit study is required annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. All MSDS documents are located on the laboratory intranet library in pdf format.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Methods for the Determination of Organic Compounds in Drinking Water*, "Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry", Method 524.2, Revision 4.1, 1995

23.0 ATTACHMENTS

23.1 Target Analytes

23.2 BFB Key Ion Abundance Criteria

ATTACHMENT 23.1
TARGET ANALYTES

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard	Linear Range (ug/L)	Default Reporting Limit (ug/L)
1	Dichlorodifluoromethane	85	87, 50	1	0.50 - 40	0.50
2	Chloromethane	50	52, 49	1	0.50 - 40	0.50
3	Vinyl Chloride	62	64, 47	1	0.50 - 40	0.50
4	Bromomethane	94	96, 93	1	0.50 - 40	0.50
5	Chloroethane	64	66	1	0.50 - 40	0.50
6	Trichlorofluoromethane	101	103, 105	1	0.50 - 40	0.50
7	Dichlorofluoromethane	67	69, 47	1	0.50 - 40	0.50
8	Ethyl Ether	45	59, 73	1	0.50 - 40	0.50
9	Acrolein	55	56, 53	1	0.50 - 40	0.50
10	1,1-Dichloroethylene	96	61, 98	1	0.50 - 40	0.50
11	1,1,2-Trichloro-1,2,2-trifluoroethane	101	103, 151	1	0.50 - 40	0.50
12	Iodomethane	142	127, 141	1	0.50 - 40	0.50
13	Carbon Disulfide	76	78, 77	1	0.50 - 40	0.50
14	Acetone	43	58, 42	1	1.0 - 40	5.0
15	Methylene Chloride	49	51, 84	1	0.50 - 40	0.50
16	Acrylonitrile	52	54, 53	1	0.50 - 40	0.50
17	Trans-1,2-Dichloroethylene	96	61, 98	1	0.50 - 40	0.50
18	Methyl tert-Butyl Ether (MTBE)	73	43, 57	1	0.50 - 40	0.50
19	1,1-Dichloroethane	63	65,83	1	0.50 - 40	0.50
20	Vinyl Acetate	43	42, 44	1	0.50 - 40	0.50
21	2,2-Dichloropropane	77	41, 79	1	0.50 - 40	0.50
22	cis-1,2-Dichloroethylene	96	61, 63	1	0.50 - 40	0.50
23	Methyl Ethyl Ketone (2-Butanone)	43	72, 57	1	1.0 - 40	5.0
24	Bromochloromethane	49	130, 128	1	0.50 - 40	0.50
25	Tetrahydrofuran	71	41, 42	1	1.0 - 40	5.0
26	Chloroform	83	85, 47	1	0.50 - 40	0.50
27	1,1,1-Trichloroethane	97	99, 61	1	0.50 - 40	0.50
28	SUR:Dibromofluoromethane	113	111, 192	1		

ATTACHMENT 23.1
TARGET ANALYTES (continued)

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard	Linear Range (ug/L)	Default Reporting Limit (ug/L)
29	IS:Fluorobenzene	96	70,50	1		
30	Carbon Tetrachloride	117	119, 121	1	0.50 - 40	0.50
31	Cyclohexane	41	39, 57	1	0.50 - 40	0.50
32	1,1-Dichloropropylene	75	110, 77	1	0.50 - 40	0.50
33	Benzene	78	51, 50	1	0.50 - 40	0.50
34	1,2-Dichloroethane	62	64, 49	1	0.50 - 40	0.50
35	SUR:1,2-Dichloroethane-d4	65	67,51	1		
36	Trichloroethylene	130	95, 132	1	0.50 - 40	0.50
37	1,2-Dichloropropane	63	62, 76	1	0.50 - 40	0.50
38	Dibromomethane	93	95, 174	1	0.50 - 40	0.50
39	Bromodichloromethane	83	85, 129	1	0.50 - 40	0.50
40	Methylcyclohexane	55	83, 98	1	0.50 - 40	0.50
41	2-Chloroethyl Vinyl Ether	63	65, 106	1	1.0 - 40	5.0
42	cis-1,3-Dichloropropylene	75	77, 110	1	0.50 - 40	0.50
43	4-Methyl-2-Pentanone (MIBK)	43	58, 85	1	1.0 - 40	5.0
44	SUR:d8-Toluene	98	100,70	1		
45	Toluene	91	92, 65	1	0.50 - 40	0.50
46	Trans-1,3-Dichloropropylene	75	77, 110	1	0.50 - 40	0.50
47	1,1,2-Trichloroethane	97	83, 85	1	0.50 - 40	0.50
48	Tetrachloroethylene	166	129, 168	2	0.50 - 40	0.50
49	1,3-Dichloropropane	76	78, 41	2	0.50 - 40	0.50
50	2-Hexanone	43	58, 85	2	1.0 - 40	5.0
51	Dibromochloromethane	129	127, 131	2	0.50 - 40	0.50
52	1,2-Dibromoethane	109	107, 188	2	0.50 - 40	0.50
53	IS:d5-Chlorobenzene	82	117, 119	2		
54	Chlorobenzene	112	77, 114	2	0.50 - 40	0.50
55	1,1,1,2-Tetrachloroethane	131	133, 119	2	0.50 - 40	0.50
56	Ethylbenzene	91	106, 51	2	0.50 - 40	0.50

Approved By: [signature] 6-1-10
QA Officer

Approved By: [signature] 6-1-10
Area Supervisor

ATTACHMENT 23.1
TARGET ANALYTES (continued)

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard	Linear Range (ug/L)	Default Reporting Limit (ug/L)
57	m,p-Xylene	91	106, 51	2	1.0 - 40	1.0
58	o-Xylene	91	106, 51	2	0.50 - 40	0.50
59	Styrene	104	78, 51	2	0.50 - 40	0.50
60	Bromoform	173	175, 79	2	0.50 - 40	0.50
61	Isopropylbenzene	105	120, 79	3	0.50 - 40	0.50
62	SUR:4-Bromofluorobenzene	95	174, 176	2		
63	Bromobenzene	77	156, 158	3	0.50 - 40	0.50
64	1,1,2,2-Tetrachloroethane	83	85, 95	3	0.50 - 40	0.50
65	1,2,3-Trichloropropane	75	49, 110	3	0.50 - 40	0.50
66	n-Propylbenzene	91	120, 65	3	0.50 - 40	0.50
67	2-Chlorotoluene	126	91, 63	3	0.50 - 40	0.50
68	1,3,5-Trimethylbenzene	105	120, 77	3	0.50 - 40	0.50
69	4-Chlorotoluene	91	126, 63	3	0.50 - 40	0.50
70	tert-Butylbenzene	119	91, 134	3	0.50 - 40	0.50
71	1,2,4-Trimethylbenzene	105	120	3	0.50 - 40	0.50
72	sec-Butylbenzene	105	134, 91	3	0.50 - 40	0.50
73	1,3-Dichlorobenzene	146	111, 148	3	0.50 - 40	0.50
74	4-Isopropyltoluene	119	134, 91	3	0.50 - 40	0.50
75	IS:d4-1,4-Dichlorobenzene	152	150	3		
76	1,4-Dichlorobenzene	146	111, 148	3	0.50 - 40	0.50
77	1,2-Dichlorobenzene	146	111, 148	3	0.50 - 40	0.50
78	n-Butylbenzene	91	92, 134	3	0.50 - 40	0.50
79	1,2-Dibromo-3-Chloropropane	75	155, 157	3	0.50 - 40	0.50
80	1,2,4-Trichlorobenzene	180	182, 145	3	0.50 - 40	0.50
81	Hexachlorobutadiene	225	227, 260	3	0.50 - 40	0.50
82	Naphthalene	128	102, 51	3	0.50 - 40	0.50
83	1,2,3-Trichlorobenzene	180	182, 145	3	0.50 - 40	0.50

ATTACHMENT 23.2
BFB KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 80% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
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

TRIMATRIX LABORATORIES

STANDARD OPERATING PROCEDURE

Volatile Organic Compounds by Purge and Trap Capillary Column Gas Chromatography/Mass Spectrometry

EPA Method 624
SW-846 Method 8260B

APPROVALS:

Area Supervisor: Diane L. VanMale Date: 5-6-10

Diane L. VanMale

QA Officer: Tom C. Boocher Date: 5-6-10

Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 5/6/10

Jeff P. Glaser

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By: Diane L. VanMale

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If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure uses gas chromatography/mass spectrometry (GC/MS) for determining volatile organic compounds in a variety of matrices. It can be used to quantify most volatile organic compounds having a boiling point below 200° C. Volatile water-soluble compounds have higher quantitation limits due to poor purging efficiencies. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers and sulfides.

1.2 Nearly all matrices, including ground water, wastewater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments can be analyzed. Sample is introduced into the system using a purge and trap concentrator.

1.3 For analysis of nonaqueous matrices, refer to TriMatrix SOP GR-04-105.

Note: Low-level bulk soil analysis by method 8260B is not allowed for state of Ohio samples under Ohio VAP (Voluntary Action Program) oversight and must not be used for Ohio VAP samples unless reported by method 8260A.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 2, December 1996

2.2 *40 Code of Federal Regulations, most current edition*, Part 136, Appendix A, Method 624-Purgeables, latest revision

2.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 8260A, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 1, September, 1994

2.4 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 5030B, "Purge and Trap for Aqueous Samples", Revision 2, December, 1996

3.0 SUMMARY OF PROCEDURE

3.1 When using purge and trap technique, an inert gas is purged through a sample, at an ambient or slightly elevated temperature. Volatile components are transferred from the aqueous to vapor phase. The vapor is swept onto a sorbent column, where volatiles are trapped.

3.2 After purging, the sorbent column is heated and back flushed with inert gas to desorb onto a capillary column. The capillary column is temperature programmed to separate and elute components, which are then transferred to a mass spectrometer, via a direct connection, using an open-split interface or a capillary-direct interface with a split at the injection port.

3.3 Component analytes are detected by the mass spectrometer which is capable of qualitative and quantitative analysis. Identification is done by comparing analyte mass spectra against

calibration standard spectra. Quantitation is performed by comparing analyte ion response (using an internal standard) to a minimum five-point calibration.

4.0 PARAMETER OR COMPOUND LIST

4.1 Compounds available for analysis and referenced to method 8260B or method 624 are listed in Attachment 23.1 (Table 1) and Attachment 23.2 (Table 1A).

4.2 Other analytes may be determined providing an acceptable demonstration of capability is performed following every step in the procedure. Additionally, all described quality control must be within laboratory established control limits.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-105, *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*, latest revision

5.2 TriMatrix SOP GR-03-124, *Volatile Laboratory Corrective Actions*, latest revision

5.3 TriMatrix SOP GR-15-102, *Waste Disposal*, latest revision

5.4 TriMatrix SOP GR-07-115, *Percent Solids, Gravimetric, Dried At 103-105° C*, latest revision

5.5 TriMatrix SOP GR-10-115, *Manual Integration*, latest revision

5.6 TriMatrix SOP GR-10-123, *Element™ Data Transfer and Review*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Whether purged or injected directly onto a column, interferences naturally present in samples can elevate reporting limits and interfere with the analysis. Naturally occurring interferences can vary considerably from site to site.

6.2 Sample contamination can also raise reporting limits or give false positives. Contamination can come from a variety of sources. Improper sampling techniques can contaminate at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through septa. A trip blank prepared from reagent water and carried through sampling and handling serves as a check on such contamination. All volatile sample storage units must contain a storage blank, which must be replaced and analyzed weekly.

6.3 During analysis, contamination can come from impurities in the purge gas or from organic compounds outgassing from the plumbing ahead of the trap, if deposited by a previously analyzed high-level sample. To minimize, the non PTFE tubing, non PTFE thread sealants, and flow controllers with rubber components in the purging device, have been eliminated where possible.

6.4 Carryover can occur whenever high and low level samples are analyzed sequentially. To reduce carryover, the autosampler rinses the purging device and sample syringe with reagent water between samples. Samples with unusually high concentrations must be followed by analysis of a reagent water blank to check for carryover. If compounds present in high concentrations are present at any level in a subsequent sample, demonstrate that there is no carryover by reanalyzing the sample. If compounds are not present in the subsequent sample, reanalysis is not necessary.

6.5 The trap and other parts are also subject to contamination. Frequent bakeout and purging of the entire system is required. If contamination persists, the complete purge and trap system must be purged first with 100° C reagent water then if necessary, with methanol. If methanol is used, disconnect the trap or install a blank trap, as methanol will adversely affect the trap's performance.

6.6 For samples containing large amounts of water-soluble material, suspended solids, high boiling compounds, or high organohalide levels, it may be necessary to wash out purging devices with a detergent solution, rinse with reagent water then dry in a 105° C oven between analyses.

6.7 Methanol content in blanks, standards, and samples must be kept constant on the ion trap. Varying methanol can suppress analyte signal response, and can alter certain spectra (chloroethane spectra will not look like the reference spectra).

Note: The maximum methanol volume purged must be no more than 100 µL.

6.8 Corrective actions for unacceptable quality control are outlined in TriMatrix SOP GR-03-124.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity or carcinogenicity of reagents used has not been precisely defined. All chemicals must be treated as a potential health hazard. From this viewpoint, chemical exposure must be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel

7.3 The following analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromomethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds must be handled only in a hood, with disposable gloves, a laboratory coat and approved safety glasses.

7.4 Approved safety glasses must be worn in the laboratory except when inputting data at a computer terminal.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To achieve reporting limits, a minimum of 40 mL aqueous sample (in duplicate) needs to be collected. More sample volume will be necessary if matrix spikes are required. Collect soil, sludge and solid waste samples as specified by TriMatrix SOP GR-04-105.

8.1.1 Collect all aqueous samples and liquid waste in 40 mL borosilicate glass screw-cap VOA vials with PTFE-lined silicone septa. All sample collection vials provided by TriMatrix contain acid preservative. Gently fill sample vials such that a meniscus develops at the top rim of the vial (fill to almost overflowing). To minimize analyte loss, no air bubbles must pass through a sample as a vial is filled, or be trapped in the sample when the vial is sealed. Invert each filled and capped vial to confirm no headspace or bubbles are present. If there is headspace or bubbles larger than 5-6 mm in diameter, fill a new vial.

8.1.1.1 If vials were not supplied by TriMatrix and are not pre-preserved, fill to just overflowing, then adjust to a pH <2 by carefully adding two drops of 1:1 HCl to each vial. Seal the filled vials with a septa screw-cap lid (PTFE face must be down), then cool to 4 ±2° C.

8.1.1.2 If a sample contains residual chlorine, collect first in a 125 mL bottle, pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl the capped bottle to mix. Proceed with Section 8.1.1, then Sections 8.1.1.3 and 8.1.1.4 if applicable.

8.1.1.3 If analysis includes acrolein and/or acrylonitrile, and a project specifies it, collect additional sample in vials that do not contain preservative. Adjust to a pH of 4-5 with 1:1 HCl, then cool to 4 ±2° C.

8.1.1.4 Analysis on unpreserved or insufficiently preserved samples (where a sample has a high buffering capacity), which reference method 8260B, will not be conducted except by specific client or project request. In such cases, analysis will be conducted within 7 days, instead of 14.

Note: This is an uncommon exception to the 14-day holding time, handled on a case by case basis.

8.1.1.5 Method 624 samples may be collected with preservative as described above, or collected with no preservative. In either case, samples must be cooled to 4 ±2° C.

8.1.1.5.1 If collected without preservative, and acrolein is an analyte, sample analysis must be completed within 3 days. Acrylonitrile has a hold time of 14 days whether preserved or not. Regardless of preservation, acrolein and/or acrylonitrile results must be qualified as screening data only under method 624.

8.1.1.5.2 Samples collected for purgeable *aromatic* hydrocarbons analysis can be collected without a chemical preservative. However, the analysis must be completed within 7 days of sample collection.

8.1.1.5.3 Samples collected for purgeable *chlorinated* hydrocarbons analysis *only* can be collected without a chemical preservative. Analysis must be completed within 14 days of sample collection.

8.1.2 At least one trip blank must accompany each aqueous sample set collected from the same general site at approximately the same time, to the site and back. A trip blank is a pre-preserved 40 mL VOA vial filled at the laboratory with reagent water, sealed, and shipped to the sampling site with empty sample containers.

8.1.3 For trip blanks associated with soil, sludge and waste collection, refer to TriMatrix SOP GR-04-105

8.2 Aqueous samples and soil samples to be prepared following TriMatrix SOP GR-04-105 must be chilled to 4 ±2° C on the day of collection, and maintained at that temperature until received by the laboratory. Samples not received by the laboratory on the day of collection, must be packaged for shipment with sufficient ice to ensure a transit temperature of 4 ±2° C.

8.2.1 Once received, TriMatrix must store samples at 4 ±2° C until analysis. Aqueous, solid, and waste samples are stored separately. Each storage area must be free of organic solvent vapors.

8.2.2 With certain exceptions (noted in Sections 8.1.1.4 and 8.1.1.5), all samples must be analyzed within 14 days of collection or qualified as estimated.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Glassware and Hardware

9.1.1 Class A volumetric flasks (10 mL, 50 mL, 100 mL, 250 mL, 1000 mL)

9.1.2 Microsyringes (10 µL, 25 µL, 50 µL, 100 µL, 1000 µL)

9.1.3 Gastight luer lock syringes, 5.0 mL

9.1.4 60 or 125 mL wide mouth glass jars with PTFE-lined caps

9.1.5 Borosilicate glass vials with PTFE-lined septum caps, 20 mL and 40 mL

9.1.6 Various size PTFE-lined screw cap vials

9.1.7 Mini-inert vials, 1.0 mL for standards preparation and storage

9.1.8 Refrigerator, capable of maintaining 4 ±2° C

9.1.9 pH test strips

9.1.10 Metal spatulas

9.1.11 Analytical balance capable of reading to 0.0001 g

9.1.12 Top-loading balance capable of reading to 0.01 g

9.1.13 Pasteur pipettes (disposable) with rubber pipette bulb

9.2 Purge and trap/gas chromatograph/mass spectrometer system:

9.2.1 Concentrators

9.2.1.1 Encon (Environmental Sample Technology) concentrator conditions used:

Trap: EST Vocarb 3000
Purge: 11 minutes at 38-42 mL/minute
Dry purge: 1 minute
Desorb preheat: 245° C
Desorb: 250° C for 2.0 minutes
Bake: 10 minutes at 260° C
Valve oven: 150° C
Transfer line: 150° C

9.2.2 Autosamplers:

9.2.2.1 Archon (O.I. Analytical 4552 or Varian) – with needle sparger for heated purging of water and low-level soil samples; preheat for 1.0 minute and purge at 45° C

9.2.2.2 Centurion (Environmental Sample Technology) for water samples and MeOH preserved soil samples

9.2.3 Gas Chromatograph

9.2.3.1 Hewlett Packard Model 5890 Series II with a split/splitless injection port and a capillary direct interface. Conditions used:

Injector temperature: 200° C
Transfer line temperature: 280° C
Column temperature program:45° C for 10 minutes, then to 73° C at 7° C/minute, then to 110° C at 10°, then to 220° at 20° C/minute, hold 2.25 minutes .

9.2.3.2 Hewlett Packard Model 6890 equipped with electronic pressure control (EPC), a split/splitless injection port and a capillary direct interface. Conditions used:

Injector temperature: 200° C
Transfer line temperature: 280° C
EPC setting: Constant flow mode at 0.7 mL/minute
Split ratio: 50:1

Column temperature program: 45° C for 3 minutes, then to 200° C at 15° C/minute, hold for 1 minute

9.2.4 Columns:

9.2.4.1 30 m x 0.25 mm ID, 1.4 um film thickness, narrow bore capillary column DB-VRX (J&W Scientific). Used in HP 5890 II with capillary direct interface (Section 9.2.3.1).

9.2.4.2 20 m x 0.18 mm ID, 1.0 um film thickness, narrow bore capillary column DB-624 (J&W Scientific). Used in HP 6890 Series II with capillary direct interface (Section 9.2.3.3).

9.2.5 Mass Spectrometer (Hewlett Packard 5971A or 5973 MSD) capable of scanning from at least 35-650 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact mode, and producing a mass spectrum that meets all criteria in Attachment 23.3 (Table 2), BFB Key Ion Abundance Criteria, when 50 ng of 4-bromofluorobenzene (BFB) are purged or injected onto the analytical column.

GC/MS operating conditions:

9.2.5.1 Electron energy: 70 volts (nominal)

9.2.5.2 Mass range: 35-300 amu

9.2.5.3 Scan time: 1.5 scans/second

9.2.5.4 Source temperature: 160 - 220° C

9.2.6 Data Acquisition:

9.2.6.1 The HP MSD data acquisition system is a DOS-based HP Chemstation equipped with EnviroQuant environmental data analysis software. It is also capable of plotting EICPs and has a 120,000 compound NIST spectral library.

9.2.6.2 Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

9.2.6.3 Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Mass Spectrometer Maintenance

10.1.1 Mass Selective Detector (MSD) Maintenance Schedule:

10.1.1.1 Drain and replace rough pump oil every 6 months

10.1.1.2 Check diffusion pump oil annually and change if discolored or low

10.1.1.3 Clean ion source at least annually (may be needed more frequently as shown by detector performance)

10.1.1.4 Replace electron multiplier as needed (maximum voltage 3000)

10.1.1.5 Check calibration gas and refill as necessary

10.2 Purge and Trap Maintenance Schedule:

10.2.1 Before initial use, new traps must be conditioned for one hour at 260C.

10.2.2 Check and record the purge flow weekly

10.2.3 Empty waste bottles and fill rinse bottles daily

10.2.4 Check and record the concentrator pressures weekly

10.2.5 Check and if necessary fill internal/surrogate standard module as needed.

10.2.6 Clean the sparge tube as necessary.

11.0 CHEMICALS AND REAGENTS

11.1 Reagent water (organic free) ASTM Type II

11.2 Methanol (purge and trap grade)

11.3 Certified stock standard materials (96% pure or greater) or certified stock solutions

11.4 1:1 Hydrochloric acid by volume

11.5 Sodium bisulfate, $NaHSO_4$, ACS reagent grade or equivalent

12.0 STANDARDS PREPARATION

12.1 All laboratory standards must be recorded in the standards logbook and/or in the laboratory information management system (Element™). Information including the vendor, lot number, concentration, purity must be recorded. Each standard is given a unique ID number. All vendors and standard concentrations provided below are recommendations, and are subject to change.

12.2 Hold Time and Storage Requirements

12.2.1 Unopened ampoules of purchased standard solutions and neat materials used in stock standards preparation, may be kept for up to 12 months from the date received or as directed by the manufacturer.

12.2.2 Due to evaporative loss and reactivity, laboratory prepared stock, and nongaseous intermediate stock standards are given an expiration date of six months. An intermediate stock gas standard typically requires weekly replacement. Within expiration dates, all standards must not be kept past acceptable performance.

12.2.2.1 Continuing calibration standards must meet the calculated percent difference or drift criteria of Section 14.7 to be acceptable. This requirement applies to all analytes, both gaseous and non-gaseous. Once this criterion has been exceeded, the intermediate stock and/or the stock must be replaced unless exceedance is due to instrument performance degradation.

12.2.2.2 Dichlorodifluoromethane will generally be the first gaseous compound to fail.

12.2.2.3 Because 2-chloroethyl vinyl ether is reactive, it will typically be among the first of the nongaseous compounds to fail.

12.2.3 Store all prepared standards with minimal headspace, protected from light, in mini-inert vials at -10° C or below. Purchased standards are stored as instructed by the manufacturer. Standards must be stored separately from samples.

12.3 Stock Standard Preparation

12.3.1 Stock standards are prepared from neat materials or certified solutions. All purchased solutions must be accompanied by a Certificate of Analysis. Two separate standard sources must be obtained. This applies to both purchased standards and neat compounds. One set will be used to prepare calibration, laboratory fortified blanks (blank spikes) and matrix spikes, and the other will be used for laboratory control samples secondary calibration verifications). The two must be prepared from dissimilar or entirely separate sources. It is permissible to purchase different lots from the same vendor. However, a dissimilar lot means preparation from dissimilar chemical lots. Not prepared twice from the same lot. It is not necessary to purchase/prepare Internal Standard/Surrogate mixes from separate sources.

12.3.2 When preparing stock standards from neat material, the mass weighed out may be used without mathematical correction when the compound has a purity greater than or equal to 96%. Prepare stock standard solutions in methanol.

12.3.3 Gravimetric Method:

12.3.3.1 A 10,000 mg/L stock solution: 0.5 g of each solid analyte (neat) is transferred to a 50 mL volumetric flask partially filled with methanol which has been tared on an analytical balance. Record mass to the nearest 0.0001 g, make sure the solid is dissolved then fill to the mark with methanol. Cap and invert three times to ensure proper mixing. Discard

the contents in the flask's neck and transfer the remaining solution to a PTFE-capped vial.

12.3.4 Purchased Commercial Stock Standards:

12.3.4.1 The following standards are combined to make Stock Standard I.

- Mix A: Volatile Organic Compounds - Liquids @ 2.0 mg/mL in MeOH
- Mix B: Volatile Organic Compounds - Gases @ 2.0 mg/mL in MeOH
- Mix C: Acrolein & Acrylonitrile @ 2.0 mg/mL in MeOH
- Mix D: Ketone Mix @ 2.0 mg/mL in MeOH
- Mix E: Custom Additions to Method 8260B @ 2.0 mg/mL in MeOH
- Mix F: Dichlorofluoromethane @ 1.0 mg/mL in MeOH
- Mix G: Internal Standard and Surrogate Standard Mix at 5.0 mg/mL in MeOH.
- Mix H: BFB Standard Mix at 2.0 mg/mL in MeOH.

12.3.4.2 The following standards are combined to make Stock Standard II.

- Mix A: Appendix IX Standard @ 2.5 mg/mL in MeOH
- Ethyl Methacrylate at 1.0 mg/mL in MeOH.
- Isopropanol at 1.0 mg/mL in MeOH.
- 2-Chloro-1,3-butadiene (chloroprene) at 2.0 mg/mL in MeOH.
- N-butyl acetate at 2.0 mg/mL in MeOH.
- Methyl Methacrylate at 2.0 mg/mL in MeOH.

12.4 Intermediate Stock Standards

12.4.1 8260B Intermediate Stock Standard I

12.4.1.1 This solution is prepared in a 1.0 mL mini-inert vial. Add 650 µL of methanol to the mini-inert vial. From the mixes specified in Section 12.3.4.1, add the following volumes to produce 1.0 mL of a 100 mg/L intermediate stock standard:

50 µL Mixes A, B, C, D, and E
100 µL Mix F

12.4.2 Appendix IX Intermediate Stock Standard II

12.4.2.1 This standard is also prepared in a 1.0 mL mini-inert vial. Add 610 µL of methanol to the mini-inert vial. From the mixes specified in Section 12.3.4.2 add the following quantities to produce 1.0 mL of a 100 mg/L intermediate stock standard:

40 µL Mix A
50 µL 2-Chloro-1,3-butadiene
100 µL Isopropanol
100 µL Ethyl methacrylate
50 µL Methyl methacrylate
50 µL N-Butyl acetate

12.5 Working Standards

12.5.1 Internal Standard/Surrogate Intermediate Stock Standard III

12.5.1.1 This standard is prepared at a concentration of 200 mg/L. Fill a 25 mL volumetric flask approximately three-quarters full with methanol. Add 1.0 mL of mix G specified in Section 12.3.4.1 and dilute to volume with methanol. Cap and invert three times to ensure proper mixing. Discard the contents in the flask's neck, and transfer the remaining contents to a PTFE-capped vial. The purge and trap auto sampler adds this standard automatically to samples during the purge cycle.

12.5.2 BFB Intermediate Stock Standard IV

12.5.2.1 This standard is prepared in a 1.0 mL mini-inert vial. Add 990 µL of methanol to the mini-inert vial. Add 10 µL of mix H specified in Section 12.3.4.1 to produce 1.0 mL of a 20 mg/L intermediate stock standard. This standard is used for performing BFB tunes.

12.5.3 Initial calibration and calibration verification standards are prepared by spiking different volumes of intermediate stock standard into a 50 mL volumetric flask. These standards are prepared as needed and not stored. Calibration verification standards are typically prepared at 40 ug/L for all except Appendix IX compounds, which are prepared at 100 ug/L. Default spike volumes used in preparing the initial and continuing calibration verification standards are discussed in Section 14. Stock volumes necessary to prepare other concentrations can be calculated using the following formula:

$$V_s = \frac{C_f \times V_f}{C_s}$$

where:
V_s = Volume of stock standard to inject (µL)
C_f = Final concentration of working standard (ug/L)
V_f = Final volume of working standards (mL)
C_s = Concentration of stock standard (ug/mL)

12.6 Matrix Spike Standards

12.6.1 The second source intermediate stock standards prepared in Section 12.4 are used for matrix spiking. Matrix spikes are prepared using the formula given in Section 12.5.3. Matrix spike standards must be prepared at concentrations near the midpoint of the calibration, typically the same concentration as the continuing calibration standard. Other concentrations may be specified for certain projects.

12.6.2 The following analytes are default matrix spike compounds: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene and benzene. Additional analytes are reported if requested by clients.

12.6.2.1 Samples that must meet data quality objectives of the State of Wisconsin must be spiked with all target analytes.

12.6.2.2 Samples the must meet data quality objectives of the Department of Defence must be spiked with all target analytes.

12.6.2.3 Any client sample requiring all target analytes to be quality control spiked must be input to the laboratory information management system (Element™) to spike all target analytes.

12.7 Laboratory Fortified Blank/Blank Spike (LFB/BS) Standards

12.7.1 Primary source intermediate stock standards prepared in Section 12.4 are used for blank spikes. An LFB/BS is prepared using the calculation given in Section 12.5.3. LFB/BSs are prepared at the same concentration as the matrix spike.

12.7.2 The same analytes used in matrix spikes are used for the LFB/BS. Additional analytes are reported if requested by the client. The same analyte list must be reported for matrix spikes and the LFB/BS.

12.7.2.1 Samples that must meet data quality objectives of the State of Wisconsin must be spiked with all target analytes.

12.7.2.2 Samples the must meet data quality objectives of the Department of Defence must be spiked with all target analytes.

12.7.2.3 Any client sample requiring all target analytes to be quality control spiked must be input to the laboratory information management system (Element™) to spike all target analytes.

13.0 SAMPLE PREPARATION

13.1 Soils are prepared in accordance with TriMatrix SOP GR-04-105 with reference to SW-846 method 5035.

13.2 Waters are prepared using the Archon or Centurion Autosampler

13.2.1 Samples analyzed on these autosamplers require no preparation. The 40 mL vial is loaded directly into the autosampler.

13.2.2 If a dilution is required, the sample must be diluted into the appropriate size volumetric flask and an aliquot transferred to a 40 mL VOA vial for analysis. An ideal dilution results in an analyte concentration in the upper half of the calibration range.

13.2.3 The autosampler withdraws a five mL aliquot from the 40 mL vial, and adds the required internal standards and surrogates before transferring to the sparge vessel.

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration

14.1.1 A 7-point calibration must be run before sample analysis. The low point of the initial calibration curve must be at or below the minimum reporting limit for each analyte. The high point defines the linear range. Calibration standards must be matrix matched to samples whenever possible, and analyzed under the same operating conditions. All target compounds must be included in the initial calibration. A calibration must be analyzed only after a successful BFB analysis.

14.1.2 For the Archon or Centurion autosampler, prepare standards in 50 mL volumetric flasks using 10 times the volumes listed in Table 3 and 3A. Transfer to a 40 mL vial, then load in the autosampler. The autosampler takes a 5.0 mL aliquot and adds 1.0 µL of internal standard/surrogate mixture before purging. The Archon and Centurion are used to analyze aqueous samples, and methanol extracts after dilution into reagent water.

Note: **SODIUM BISULFATE ADDITION DEGRADES 2-CHLOROETHYL VINYL ETHER IN STANDARDS AND SAMPLES. TRIMATRIX LABORATORIES HAS DEMONSTRATED THAT ACCEPTABLE RESULTS FOR THIS COMPOUND ARE NOT ACHIEVED WITH THIS TECHNIQUE.**

14.2 Calculate Response Factors (RF) for each compound using the quantitation and internal standard ions listed in Attachments 23.6 or 23.7. Calculate RF using the following formula:

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

where:
A_x = Area of the characteristic ion for the target compound
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured
C_{is} = Concentration of the specific internal standard

14.2.1 For compounds not listed in Attachments 23.6 or 23.7, choose a major ion that is dissimilar to any potential coeluting or interfering ions, for the quantitation ion. Choose an internal standard with a retention time closest to the compound being measured.

14.2.2 Calculate average response factors as follows:

$$RF_{avg} = (RF_1 + RF_2 + RF_3 + ...RF_n)/n$$

where:
RF_{avg} = Average calibration response factor
RF_1 = Calibration response factor for standard 1
RF_2 = Calibration response factor for standard 2
RF_3 = Calibration response factor for standard 3
RF_n = Calibration response factor for standard n
N = number of calibration standards

14.3 The average RF for all calibration points must be calculated for each compound. Five System Performance Check Compounds (SPCCs) must be checked against a minimum average RF limit.

14.3.1 These SPCCs are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. These minimum acceptable average response factors are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	>0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

14.3.2 An SPCC monitors compound instability and degradation caused by contaminated lines or active sites in the system. Examples of such occurrences are:

14.3.3 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

14.3.4 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

14.3.5 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated purge-and-trap transfer lines and/or active sites in trapping materials.

14.3.6 All non-SPCC compounds must adhere to the following minimum average RF limits:

14.3.6.1 The average RF of Alcohols, 1,4-Dioxane, and Epichlorohydrin must be at least 0.001 to be acceptable.

14.3.6.2 The average RF of all other compounds must be 0.010 or higher.

14.4 Always check for carryover and memory effects (ghosting) from high concentration standards or samples when analyzing for late eluting compounds. Adequate purge chamber rinsing minimizes such effects. Newer purge-and-trap systems address this problem with a bakeout step following

trap desorb and newer traps retain less moisture. However, high concentrations can still affect subsequent runs.

14.5 Calculate Percent Relative Standard Deviation (%RSD) for all compounds. Using the initial calibration average RF, calculate percent RSD using the following formula:

$$\text{Percent RSD} = \frac{SD}{X} \times 100$$

where:

RSD = Relative standard deviation.
SD = Standard deviation of average RF for a compound.
X = Mean of the five initial response factors for a compound.

14.5.1 All compounds must have a percent RSD of ≤15%. Six compounds are used as Calibration Check Compounds (CCCs): 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethyl benzene, vinyl chloride. These compounds **must** have a percent RSD of ≤30%. If percent RSD is greater than 30 percent for any CCC, corrective action must be initiated and the entire system recalibrated.

14.5.1.1 If percent RSD of a compound is 15% or less, the calibration is assumed to be linear through the origin, and an average response factor may be used for quantitation.

14.5.1.2 If percent RSD of any compound is greater than 15%, then a regression curve of area ratio (Area analyte/Area IS) against concentration must be constructed, using first or higher order regression fit. Analysts must select a regression algorithm providing the best fit.

14.5.1.2.1 Generally this will be a first order linear regression using the following equation:

$C_i = A_i\text{-}b)/m$

Where:
C_i = Concentration in ug/mL
A_i = Area ratio ($A_{analyte}/A_{IS}$)
b = Intercept of the regression curve
m = Slope of the regression curve
i = Compound "i"

14.5.1.2.2 Second order regression must only be used for analytes with a definite quadratic response, as results produced by this algorithm may give erroneous results at concentrations near the reporting limit. A minimum of six calibration points are required to use second order calibration curves. The Enviroquant data processing software performs all regression functions, and uses coefficient of determination (r^2) to measure

calibration validity. To be considered an acceptable calibration curve, r^2 must be 0.990 or higher.

14.5.1.3 If initial calibration criteria are not achieved using all calibration points run, the lowest or highest point on the curve may be dropped, provided enough points remain for the calibration model chosen.

14.5.1.3.1 A minimum of six calibration points are required to use second order regression curves. Five date points are needed for first order.

14.5.1.3.2 The lowest calibration point must be at the analyte reporting limit. The low point can not be dropped without elevating reporting limits.

14.5.1.3.3 Dropping the highest point shortens the calibration range, which could lead to a greater number of sample dilutions.

14.6 Initial calibration verification must be performed immediately after the initial calibration by running a second-source calibration verification (SCV) standard at 40 ug/L.

14.6.1 The SCV must be run after each initial calibration.

14.6.2 The SCV must include all targeted analytes.

14.6.3 Acceptance criteria for all analytes are ±25% of expected value unless otherwise specified in the laboratory information management system (Element™).

14.6.4 If any SCV analyte fails, locate and correct the problem (up to and including remaking the SCV solution) then repeat the SCV analysis successfully.

14.6.5 If the second SCV attempt fails, review the initial calibration solutions and remake or obtain a new calibration stock then repeat the SCV successfully.

14.6.5 Sample analysis may not begin until an acceptable initial calibration and SCV have been run successfully.

14.7 Every 12 hours a 40 ug/L continuing calibration verification (CCV) containing each compound quantified must be run after the BFB (Section 15.3). If a calibration has just been run, then the 40 ug/L standard from the calibration is used. Analysis of the CCV verifies instrument sensitivity and confirms acceptability of the initial calibration curve. A CCV is verified the same as an initial calibration, by checking SPCCs and CCCs. The CCV must be analyzed and quantified against a regression fit or average RF under conditions identical to the initial calibration.

14.7.1 System Performance Check Compounds (SPCC): This is the same check applied during initial calibration. SPCC compound response factors must be as listed in Section 14.3.1 (Section 14.2 for calculating response factors) In addition, non-SPCC compounds must meet the initial calibration criteria (Section 14.3.6) If minimum response factors are not met, the system must be evaluated and corrective action taken before sample analysis begins. Possible problems include standards

degradation, injection port contamination and/or buildup in the first six inches of the analytical column and active sites in the column or chromatographic system.

14.7.2 Calibration Check Compounds (CCCs): After SPCCs are met, CCCs are used to check initial calibration validity. Calculate percent drift or percent difference using the following equations:

Percent Drift $= [(C_c - C_1)/C_1]*100$

where:
C_1 = CCC concentration
C_c = Measured concentration using selected average RF or regression.

Percent Difference $= [(RF_{ccc} - RF_{avg})/ RF_{avg}]*100$

where:
RF_{avg} = Average Response Factor from Initial Calibration
RF_{ccc} = Response Factor from Continuing Calibration

14.7.3 If CCC percent drift or difference does not exceed ±20%, the initial calibration continues to be valid. If acceptance is not met, then corrective action must be taken. An acceptable CCC **MUST** be run before sample analysis can begin. If CCC compounds are not target analytes, then all target analytes must be used to meet the ±20% control limits.

14.7.4 For non-CCC compounds in the CCV, the following criteria must be used.

14.7.4.1 All percent differences or drifts must not exceed ±25%, with the following exceptions:

14.7.4.2 Alcohols, ketones, 2-Methylnaphthalene, 2-Chloroethyl vinyl ether, 1,4-Dioxane, Vinyl acetate, and Iodomethane must not exceed ±40%.

14.7.4.3 Also, if non-CCC compound responses are high and out of control, then non-detect sample results for those compounds may still be reported since there is no question of analyte sensitivity for that day.

14.7.5 It is not permitted to choose a quantitation technique dissimilar to that used during initial calibration to achieve CCC criteria. For example: An analyte initial calibration %RSD was within ±15% drift, and average RF is used for quantitation. The last CCC is not acceptable for percent drift. To attain a passing CCC, it is not permitted to switch to a regression curve in an attempt to pass the CCC, even if the regression curve passes coefficient of determination criteria.

14.7.6 Internal standard responses and CCV retention times must be evaluated during or immediately after data acquisition.

14.7.6.1 If the retention time for an internal standard changes by more than 30 seconds compared to the 40 ug/L initial calibration, the system must be inspected for malfunctions. Corrections must be made as needed.

14.7.6.2 If the area for an internal standard changes by a factor of two (-50% to +100%) compared to the 40 ug/L initial calibration standard, the system must be inspected for malfunctions. Corrections must be made as needed.

14.7.6.3 When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

15.0 ANALYTICAL PROCEDURE

15.1 Before initial use, a Vocarb 3000 trap must be conditioned for at least one hour by baking at 260° C and purging with helium. If other trapping materials are substituted for the Vocarb 3000, follow the manufacturer's conditioning recommendations. After periods of inactivity, GC columns may be run through the temperature program, or ramped to the final temperature and held for 15 minutes.

15.2 Set up the autosampler, purge-and-trap, and GC/MS as instructed in Section 9.0. Sample prep and purging conditions are specific to each matrix. The procedure for soil samples is described in TriMatrix SOP GR-04-105.

15.3 BFB Tuning

15.3.1 At the beginning of every 12-hour shift 4-Bromofluorobenzene (BFB) must be used to tune the mass spectrometer. Analyze the BFB and compare the spectra obtained to the criteria given in Attachment 23.3. No other analysis may begin prior to running an acceptable BFB tune.

15.3.2 The standard prepared in Section 12.5.2.1 contains 20 mg/L of 4-Bromofluorobenzene (BFB).

15.3.2.1 For the Archon autosampler, spike 25 µL into a 50 mL volumetric flask containing reagent water. Transfer the contents to a 40 mL sample vial.

15.3.2.2 The result will be to inject 50 ng of BFB into the GC/MS system. All subsequent analysis must commence within 12 hours of the BFB injection. BFB must be analyzed using the same acquisition parameters as standards and samples.

15.3.3 The following evaluation sequence must be used to determine BFB tune performance.

15.3.3.1 All BFB tunes must be initially evaluated using the CLP BFB tuning procedure, which takes the scan average at the peak apex and two scans immediately before and after the apex, followed by a background subtraction.

15.3.3.2 If the above does not generate an acceptable BFB tune, then take a scan average of the entire peak, followed by a background subtraction.

15.3.3.3 If the above two options do not yield an acceptable BFB tune, then evaluate with a single scan at the apex.

15.3.3.4 If none of the above give an acceptable BFB tune, then corrective action is required. The first step is to reinject and reanalyze the same BFB standard. Next, prepare a new BFB working solution, then inject and analyze. The last step is to optimize the tuning acquisition parameters by tweaking manually or by performing a new autotune. Once acquisition parameters are optimized, restart the tuning sequence.

15.4 Initial/Continuing Calibration

15.4.1 After a successful BFB analysis, an initial curve or continuing calibration verification must be analyzed (Section 14.0). Initial and continuing calibrations must match sample methanol and/or sodium bisulfate concentrations wherever possible, and be run under the same analytical conditions as samples. Follow initial or continuing calibration with the quality control described in Section 18.0.

15.5 Sample Analysis

15.5.1 For aqueous samples analyzed using the Archon or Centurion autosampler, the 40 mL sample collection vial is placed directly into the autosampler without opening. The autosampler removes 5.0 mL and transfers it to the purge vessel automatically. Internal standards and surrogate are added by the autosampler as well. The purge sequence will start automatically, using the operating conditions in Section 9.0. At the end of the purge cycle, analytes remain on the adsorbent trap, and are ready for desorption to the gas chromatograph.

15.5.2 Low concentration analysis for bulk and sodium bisulfate preserved soils (approximate concentration range 0.010 - 1.0 mg/kg):

15.5.2.1 If a sample is preserved in sodium bisulfate, remove the vial from storage, and let warm to room temperature. Agitate the vial gently so contents move freely and stirring will be effective. For bulk soils prepared in the laboratory, 5.0 mL of reagent water and a PTFE stir bar must be added during preparation. Place the vials in the autosampler.

15.5.2.2 The autosampler adds 10 mL of organic-free reagent water, internal standards, and surrogates before initiating the purge sequence. Other volumes may be used but **it is imperative that all samples, blanks and calibration standards have exactly the same added volume of organic-free reagent water**.

15.5.2.3 Before purging, vials are heated to 45° C and held for 1.0 minute. Purge follows the operating conditions provided in Section 9.0. Heat and stir using the magnetic stir bar, for the entire purge cycle. After purging, analytes remain on the adsorbent trap, and are ready for desorption to the gas chromatograph.

15.5.2.4 This technique must be used only by client request.

15.5.4 The following is for extraction of high concentrations (greater than 1 mg/kg), from solid and oily waste:

15.5.4.1 Remove sample methanol extracts from storage and let warm to room temperature. Using a microsyringe, transfer an appropriate extract volume to organic-free reagent water in a 50 mL volumetric flask. Minimally a 1:50 dilution is done.

15.5.4.2 Transfer the diluted extract to a 40 mL vial and place in the autosampler. The autosampler will add 10 mL of organic-free reagent water if using the Archon autosampler and internal standards/surrogates before purging.

15.5.4.3 Report soil results on a dry weight basis. Report waste results on a wet weight basis.

15.5 Sample Desorption

15.5.1 After the 11 minute purge, the system will automatically advance to the desorb mode. The trap will preheat to 245° C without desorption gas flow. When the trap reaches this setpoint, it will desorb at 20 mL/minute for two minutes. Desorbing initiates the gas chromatograph oven temperature program, and data acquisition begins.

15.6 After desorbing, the trap is reconditioned by baking out at 260° C (or the temperature recommended by the trap packing material manufacturer). After 10 minutes, the trap heater is turned off and purge flow through the trap is halted. When the trap is cool, the next analysis can begin.

15.7 If any compound response exceeds the calibration range, prepare a sample dilution. An ideal dilution will result in analyte response at midrange in the calibration. However, a dilution with a response in the upper 60% of the range is acceptable.

15.7.1 For aqueous sample dilutions, prepare a sample dilution from an aliquot of the second (duplicate) 40 mL vial or from unused sample saved under zero headspace from the first vial. A dilution is prepared in a volumetric flask (50 mL or larger). The minimum volume that can be diluted is 1.0 mL. The total volume purged must equal the volume used for calibration standards (5.0, 10 or 25 mL). Organic-free reagent water must be used for all dilutions. Matrix-matching must be done by including the same methanol volume to dilutions as used in calibration standards. Pour the contents into a 40 mL vial then load into the autosampler, which will automatically add internal standards/surrogates.

15.7.2 For bulk soil dilutions, prepare a 1.0 g sample instead of 5.0 g if the expected concentration is within the calibration range. If a larger dilution is needed, refer to TriMatrix SOP GR-04-105.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Qualitative Analysis:

16.1.1 An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard. Standard spectra are obtained from calibration standards.

16.1.1.1 Two criteria must be satisfied to verify analyte identification:

16.1.1.1.1 Elution at the same GC relative retention time (RRT) as the standard.

16.1.1.1.2 Positive correlation of the sample analyte mass spectrum with the standard.

16.1.1.2 The sample component RRT must compare within ±0.06 RRT units of the daily continuing calibration standard. If coelution interferes with accurate component RRT assessment from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles, for ions unique to the compound.

16.1.1.3 All ions present in standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%), are automatically checked by the software to be present in the sample spectrum. Relative ion intensities must agree within plus or minus 20%, between standard and sample spectra.

Example: For an ion having a relative intensity of 50 percent in standard spectra, corresponding sample ions must be between 30 and 70 percent.

16.1.2 For sample compounds not associated with a calibration standard, a library search can be made for tentative identification. Only after visual comparison of sample spectra with the nearest library searches will an analyst assign a tentative identification. When a tentatively identified compound (TIC) cannot be identified by name, a generic description will be given to help identify functional groups. These TICs will have names such as:

Name	CAS #
Unknown Alcohol	xx-xx-xx
Unknown Freon	xx-xxx-xxxx
Unknown Ketone	xx-xxx-xx
Unknown Acid	xxx-xx-xxxx
Unknown Hydrocarbon	xxx-xxx
Unknown Glycol Ethers	xxx-xx-x
Unknown Substituted Benzenes	Xxxxx
Hydrocarbons (sub. Benzenes)	xxxxx-x-x
Hydrocarbons, Total	Xxxxxx

Guidelines for making a tentative identification are:

16.1.2.1 Relative intensities of major ions in the library spectrum (ions >10% of the most abundant ion) must be present in a sample spectrum.

16.1.2.2 Relative intensities of major ions must agree within ±20%. As an example: For an ion having a relative intensity of 50 percent in library spectra, corresponding sample ions must be between 30 and 70 percent.

16.1.2.3 Library spectra molecular ions must be present in a sample spectrum.

16.1.2.4 Sample spectra ions not present in the library spectrum must be reviewed for the presence of background contamination or coeluting compounds.

16.1.2.5 Library spectra ions not in sample spectra must be reviewed for possible subtraction from the sample spectrum, because of background contamination or coeluting peaks. Data system library reduction can sometimes create such discrepancies. When TIC searches are performed, the criteria for determining whether or not a peak is a TIC are:

16.1.2.5.1 The peak in question must be >10% of the nearest internal standard.

16.1.2.5.2 The top 10 potential TICs must be reviewed, unless otherwise required. The match quality must be 70 percent (corresponding to a fit of 700 on the Ion Trap) or higher, to report a positive identification in the absence of interfering peaks. If there are interfering peaks, analyst discretion must be employed when reporting positive matches.

16.1.2.5.3 Concentrations <1 ug/L or <0.01 mg/kg (assuming a nominal sample extraction volume or mass, and instrument response) will not be reported as a TIC unless otherwise specified for the project, and deemed achievable following supervisor data review.

16.1.2.6 Concentrations obtained must be reported indicating the values are estimated, and are flagged with an E. Calibrated analytes not part of a client's target analyte list will also be reported when TIC's are requested. They are to be given a Q flag (quantitated) if the concentration is within the calibration range or CE flag (curve estimated) if outside the range. If no valid identification can be made, the compound must be reported as an unknown aromatic, hydrocarbon or other class, if classification is possible.

16.2 Quantitative Analysis

16.2.1 Quantitation is performed using the same technique used during initial calibration (average response factor or regression). Use analyte area and internal standard ions as specified in Attachment 23.1 (Table 1A) and Attachment 23.2 (Table 1A), and the equations below.

16.2.1.1 Aqueous Samples

$$\frac{A_x \times C_{is}}{A_{is} \times RF} \times DF = ug/L$$

where:
A_x = Area of the characteristic ion for the target compound
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard in ug/L

RF = Average Response Factor
DF = Dilution factor

16.2.1.2 Low Level Soil Samples

$$\frac{mg}{kg} = \frac{A_x \times C_{is}}{A_{is} \times RF} \times \frac{5\ mL}{W \times \%S} \times \frac{L}{1000\ mL} \times \frac{mg}{1000\ ug} \times \frac{1000\ g}{kg}$$

where additionally:
W = Wet weight of sample (g)
%S = Percent solids in decimal form (example: 0.90, not 90%). Used to calculate dry weight results for soils and sludges. Wastes are calculated on a wet weight basis only. Refer to TriMatrix SOP GR-07-115.

16.2.1.3 High Level Soil Samples

$$\frac{mg}{kg} = \frac{A_x \times C_{is}}{A_{is} \times RF} \times \frac{DF}{\%S} \times \frac{V}{W} \times \frac{L}{1000\ mL} \times \frac{mg}{1000\ ug} \times \frac{1000\ g}{kg}$$

where additionally:
V = Volume of solvent added to sample during purging or dilution (mL)
W = Wet weight of sample purged or diluted (g)

16.2.1.4 Equations given above will also be used to quantitate TICs, substituting total ion areas for A_x and A_{is}. The internal standard chosen must be the one closest in elution time to an unknown peak, provided it is free from most interferences. C_{is} remains the same and RF will be a default of 1.0.

16.3 All manual integration must be performed with strict adherence to TriMatrix SOP GR-10-115.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running sample sets are responsible for correctly filling in, handing in and filing associated paperwork. It is essential to perform these tasks to provide defensible data and client reporting.

17.2 Reporting to the laboratory information system must be done in accordance with TriMatrix SOP GR-10-123.

17.3 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in and archived correctly. The time each analyst has sample possession must be accounted for on the form.

17.3 All laboratory hardcopy (including CoC forms) must be archived appropriately and correctly.

17.3.1 All run and maintenance logbooks must be filled in completely and correctly. Corrections must be made with a lineout, not a writeover and must be dated and initialed. Blank lines must be Z'd out, initialed and dated.

17.3.2 All tunes, calibrations and continuing calibration verification runs must be placed in the correct archival folder.

17.3.3 All LIMS reports must be kept and placed in the correct archival folder.

17.4 The following are required for data packages requiring CLP-like deliverables:

17.4.1 Copies of all tunes including the EICP that has BFB ion peaks and ion abundance results and requirements, including mass listings, and associated Form Vs

17.4.2 Copies of all associated curves, including raw data quantitation reports, chromatograms, and associated Form VIs

17.4.3 Copies of all associated continuing calibration verifications, including raw data quantitation reports, chromatograms, and associated Form VIIs

17.4.4 Copies of all sample raw data, including all applicable quality control samples, quantitation reports, including triple plots for positive sample results, chromatograms and all associated TIC reports when requested

17.4.5 Method Blank Summary Form IVs

17.4.6 Internal standard recovery Form VIIIs

17.4.7 Copies of all applicable supporting information including: internal chain of custody forms, run and standard logbooks, method detection limits and extraction summaries

Note: For most projects, Forms I, II and III will be included with the deliverables package.

17.5 Rounding and significant figure adjustment is only performed on the final reported result by Element™.

18.0 QUALITY ASSURANCE

18.1 Quality control requirements include the following previously covered requirements and the quality control that follows:

18.1.1 BFB (Section 15.3)

18.1.2 Initial Calibration (Section 14.1)

18.1.3 Initial Calibration Vericiation (Section 14.6)

18.1.3 Continuing Calibration (Section 14.7)

18.2 Method Preparation Blanks (BLK)

18.2.1 After the BFB and initial calibration or continuing calibration have been run, a purged blank is required before sample analysis showing the analytical system to be free of interference and contamination.

18.2.2 The blank concentration must be at or below the maximum acceptance limit listed in the laboratory information management system. However methylene chloride and acetone may be up to five times the reporting limit unless further restricted by specific data quality objectives.

18.2.3 At a minimum, a BLK is run every 12-hour shift. The BLK must be run more frequently if carryover is suspected from a high concentration sample analysis or if laboratory contamination is in question. The BLK must be carried through all steps of sample preparation and analysis, including the addition of internal standards/surrogates spiking.

18.2.4 The BLK must be prepared in the same matrix as samples:

18.2.4.1 For methanol extractions, the BLK must be prepared as for a methanol extraction using a 1:50 ratio of methanol.

18.2.4.2 If samples are prepared with sodium bisulfate preservative, the BLK must also be prepared with sodium bisulfate.

18.2.4.3 For low-level soil analyses, the BLK must be prepared in a clean solid matrix.

18.3 Internal Standards

18.3.1 Internal standard responses and retention times in all runs following continuing calibration verification must be evaluated during or immediately after data acquisition. The retention time for internal standards must be within ±30 seconds from the current 12-hour continuing calibration standard. The quantitation ion area for all internal standards must stay within a factor of two (-50% to +100%) from the current 12-hour continuing calibration verification.

18.3.2 If at any time an internal standard fails the -50% to +100% area criteria, the ability to accurately quantitate analyte is reduced. Every effort must be made to prevent an internal standard failure, including sample dilution and reanalysis. Refer to TriMatrix SOP GR-03-124, for when an internal standard fails. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for malfunctions and appropriate maintenance performed. After maintenance has resolved the problem, samples run while the instrument was malfunctioning must be reanalyzed.

18.4 Surrogates

18.4.1 All samples and quality control must be spiked with surrogates. Until thirty samples of a given matrix have been analyzed, default recovery limits of 70 - 130% will be used. Once thirty samples of a given matrix have been analyzed, in-house recovery limits will be generated. At a minimum, surrogate acceptance limits must be updated annually

on a matrix by matrix basis. Calculate surrogate recovery for each analysis. If recovery or precision is not within acceptable limits, consult TriMatrix SOP GR-03-124 to determine when and how data is qualified.

18.4.1.1 High recovery may be due to a co-eluting matrix interference from the sample. Examine the chromatogram for evidence of co-elution. No corrective action is required in this instance.

18.4.1.2 Low recovery may be due to poor purge efficiency. This should be verified by re-purging the sample if hold times permit.

18.4.1.3 If surrogate recovery in a purge blank (BLK) is below the lower control limit, only samples with failing surrogate recoveries will require re-purging. If BLK surrogate recovery is above the upper control limit, no corrective action is required as long as sample surrogate recoveries are acceptable.

18.4.1.4 If surrogate recovery fails in the MS/MSD, re-analysis is only required if LFB/BS spike recovery also fails. If the LFB/BS is still out-of-control after re-analysis, all associated samples must be re-analyzed.

18.4.2 Calculate upper and lower control limits for each surrogate. This must be done as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where
p = average percent recovery
s = standard deviation of the average

18.4.3 Two standard deviations will be used when three give a negative lower control limit.

18.4.4 If recovery fails, refer to TriMatrix SOP GR-03-124. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for malfunctions and appropriate maintenance performed. After maintenance has resolved the problem, samples run while the instrument was malfunctioning must be reanalyzed.

18.5 Laboratory Fortified Blank/Blank Spike (LFB/BS)

18.5.1 An LFB/BS is required with each 12-hour shift or with each batch of up to 20 samples, whichever is more frequent. The LFB/BS serves as a check of methanol extraction/purging efficiency should matrix spike recoveries not be within quality control limits.

18.5.1.1 When possible, the daily CCV doubles as the blank spike.

18.5.1.2 The LFB/BS is run at the beginning of the 12-hour shift following analysis of the BLK.

18.5.1.3 Recoveries must be calculated and compared to LIMS control limits. Analysis must be stopped and the problem corrected if recoveries are

outside quality control limits. Samples analyzed in a batch with a failing LFB/BS must be reanalyzed for failed analytes. If this is not possible due to holding time or lack of sample, data for the failed analytes must be qualified as estimated.

18.5.1.4 An LFB/BS may be from the same source as the calibration or from the second-source used to verify the intial calibration.

18.5.2 At a minimum, the LFB/BS must contain the following analytes:

18.5.2.1 1,1-Dichloroethylene

18.5.2.2 Trichloroethylene

18.5.2.3 Chlorobenzene

18.5.2.4 Benzene

18.5.2.5 Toluene

Note: **As noted previously, where project specifications require, all target analytes must be in the matrix spike, matrix spike duplicate and LFB/BS.**

18.5.3 Until at least twenty blank spikes have been analyzed in a given matrix, recovery will be validated against default recovery limits of 70 - 130%. The blank spike must be prepared in the same matrix as samples:

18.5.3.1 For methanol extractions, the blank spike must be prepared as for a methanol extraction using a ratio of 1:50 of methanol.

18.5.3.2 If samples are prepared with sodium bisulfate preservative, the blank spike must also be prepared with sodium bisulfate.

18.5.3.3 For low-level soil analyses, the blank spike must be prepared in a clean solid matrix.

18.5.4 If an LFB/BS is out-of-control, the problem must be immediately identified and corrected before samples can be run.

18.5.4.1 The LFB/BS verifies that out-of-control compounds in an MS or MSD are the result of matrix interference rather than extraction or purging error.

18.5.4.2 Failure of any targeted analyte in an LFB/BS requires corrective action depending upon the failed parameter. Every effort must be made to determine the reason for failure (spike, extraction or purge error). Appropriate corrective action must then be taken.

18.5.4.3 Take corrective action in accordance with TriMatrix SOP GR-03-124.

18.5.4.4 Samples may not be reported without an acceptable LFB/BS.

18.6 Matrix Spikes (MS, MSD)

18.6.1 To assess extraction and purge efficiency from specific matrices, extract a Matrix Spike (MS/SPK) and Matrix Spike Duplicate (MSD/SPK) at least once every 20 samples prepared for each matrix. If less than 20 samples are analyzed in a month, at least one matrix spike and one matrix spike duplicate is required. Generally, matrix spikes are analyzed at the end of the 12-hour shift. Running matrix spikes at the end of the shift helps document the instrument is still functioning correctly.

18.6.2 Until at least twenty matrix spike/matrix spike duplicates have been analyzed, recovery will be validated against default recovery limits of 70 - 130%. The maximum default precision acceptance limit is 20% relative percent difference (RPD).

18.6.3 Once twenty MS/MSD sets of a given matrix (water, soil or waste) have been analyzed, statistical acceptance limits will be calculated by the laboratory information management system and listed there.

18.6.4 If MS/MSD recovery or duplication is not within acceptable limits, take corrective action in accordance with TriMatrix SOP GR-03-124.

18.7 This procedure is written primarily in reference to SW-846 8260B, however the following modifications are allowed when used for EPA method 624 sample analyses:

18.7.1 The 12-hour shift is replaced by a 24-hour shift. BFB and continuing calibration verifications are still required but only run once in 24 hours instead of every 12.

18.7.2 A 3-point calibration may be used in place of the 7-point.

18.7.3 SPCCs and CCCs are not used. Instead response factors for every compound listed on the EPA 624 list must have ≤35% RSD for the calibration to be valid.

18.7.4 The continuing calibration verification does not use SPCCs and CCCs. The RF for every compound in the 40 ug/L continuing calibration verification is compared with the corresponding calibration acceptance criteria found in Attachment 23.8 (Table 5). If parameter responses fall within the designated ranges, analysis of samples can begin. If any individual RF falls outside the range, a new continuing calibration verification or a new calibration must be run.

18.7.5 There are no criteria for internal standard areas or retention times in method 624. However, method 8260B criteria will be followed.

18.7.6 Every targeted analyte must be spiked in the MS/MSD, SCV and LFB/BS. Not just the limited list in Section 18.5.2.

18.7.7 Acrolein and Acrylonitrile may only be screened by GC/MS. All positive results must be qualified in the report as estimated.

18.7.8 Refer to Section 8.1.1.5 for sample collection requirements unique to method 624.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis to demonstrate acceptable accuracy and precision.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Spike four aliquots of organic-free reagent water so analyte concentrations are in the lower half of the calibration range. Process the four spikes following every step outlined in the procedure, including quality control.

19.1.1.2 Input all four results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must be within the LFB/BS acceptability limits listed in the laboratory information management system. Relative standard deviation must be ≤20%.

19.1.1.3 If any criterion in the study fails, locate and correct the source of the problem and repeat the study successfully.

19.1.1.4 Repeated failure, will confirm a general problem with the procedure and/or techniques used. If this occurs, locate the problem and correct the procedure and/or techniques used then repeat the study successfully.

19.1.1.5 Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

19.1.1.6 Copies of successful demonstration of capability studies and raw data must be submitted to the Quality Assurance department.

19.1.2 A Continuing Demonstration of Capability (CDC) must be performed annually by all analysts running samples by any of the following approaches:

19.1.2.1 By using the last four of seven results obtained from a method detection limit study if run exclusively by the analyst. ONLY the last four results may be used.

19.1.2.2 BY repeating the IDC study.

19.1.2.3 By using four consecutively run blank spike results obtained during the course of routine analysis and if run exclusively by the analyst.

19.1.2.4 By running an acceptable blind performance testing sample during the course of routine sample analysis.

19.1.2.5 Copies of successful demonstration of capability studies and raw data must be submitted to the Quality Assurance department.

19.2 A Method Detection Limit (MDL) Study must be performed annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 2, December 1996

22.2 *40 Code of Federal Regulations, most current edition*, Part 136, Appendix A, Method 624-Purgeables, latest revision

22.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 8260A, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 1, September, 1994

22.4 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 5030B, "Purge and Trap for Aqueous Samples", Revision 2, December, 1996

23.0 ATTACHMENTS

23.1 Attachment 23.1, Table 1, Compound List, CAS #, and Routine Reporting Limits, for Standard I

23.2 Attachment 23.2, Table 1A, Compound List, CAS #, and Routine Reporting Limits, For Standard II

23.3 Attachment 23.3, Table 2, BFB Key Ion Abundance Criteria

23.4 Attachment 23.4, Table 3, Standard 8260B Volumes Required For Curve

23.5 Attachment 23.5, Table 3A, Appendix IX Volumes Required For Curve

23.6 Attachment 23.6, Table 4, Elution Order, Quantitation and Characteristic Ions, Internal Standards, and Surrogates for Standard I

23.7 Attachment 23.7, Table 4A, Elution Order, Quantitation and Characteristic Ions, for Standard II

23.8 Attachment 23.8, Table 5, Continuing Calibration QC Acceptance Criteria, Method 624

23.9 Attachment 23.9, Example Chromatogram, DB-624

23.10 Attachment 23.10, Example Chromatogram, DB-VRX

Attachment 23.1
Table 1
Compound List, CAS Number and Routine Reporting Limits for Standard I

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting Limit		
				Aqueous ug/L	Low Level Soil, mg/kg	High Level Soil, mg/kg
D	Acetone	67-64-1	100	5	0.1	0.75
C	Acrolein	107-02-8	100	5	0.01	0.05
C	Acrylonitrile	107-13-1	100	1	0.01	0.05
A	Benzene	71-43-2	100	1	0.01	0.05
A	Bromobenzene	108-86-1	100	1	0.01	0.05
A	Bromochloromethane	74-97-5	100	1	0.01	0.05
A	Bromodichloromethane	75-27-4	100	1	0.01	0.05
A	Bromoform	75-25-2	100	1	0.01	0.05
B	Bromomethane	74-83-9	100	1	0.01	0.05
A	n-Butylbenzene	104-51-8	100	1	0.01	0.05
A	sec-Butylbenzene	135-98-8	100	1	0.01	0.05
A	tert-Butylbenzene	98-06-6	100	1	0.01	0.05
D	Carbon disulfide	75-15-0	100	1	0.1	0.5
A	Carbon tetrachloride	56-23-5	100	1	0.01	0.05
A	Chlorobenzene	108-90-7	100	1	0.01	0.05
B	Chloroethane	75-00-3	100	1	0.01	0.05
D	2-Chloroethyl vinyl ether	110-75-8	100	10	0.1	0.5
A	Chloroform	67-66-3	100	1	0.01	0.05
B	Chloromethane	74-87-3	100	1	0.01	0.05
A	2-Chlorotoluene	95-49-8	100	1	0.01	0.05
A	4-Chlorotoluene	106-43-4	100	1	0.01	0.05
E	Cyclohexane	110-82-7	100	10	0.01	0.25
A	Dibromochloromethane	124-48-1	100	1	0.01	0.05
A	1,2-Dibromo-3-chloropropane	96-12-8	100	1	0.01	0.05
A	1,2-Dibromoethane	106-93-4	100	1	0.01	0.05
A	Dibromomethane	74-95-3	100	1	0.01	0.05
A	1,2-Dichlorobenzene	95-50-1	100	1	0.01	0.05
A	1,3-Dichlorobenzene	541-73-1	100	1	0.01	0.05
A	1,4-Dichlorobenzene	106-46-7	100	1	0.01	0.05
E	trans-1,4-Dichloro-2-butene	110-57-6	100	1	0.05	0.25
B	Dichlorodifluoromethane	75-71-8	100	1	0.01	0.05
A	1,1-Dichloroethane	75-34-3	100	1	0.01	0.05
A	1,2-Dichloroethane	107-06-2	100	1	0.01	0.05
A	1,1-Dichloroethylene	75-35-4	100	1	0.01	0.05
A	cis-1,2-Dichloroethylene	156-59-2	100	1	0.01	0.05
A	trans-1,2-Dichloroethylene	156-60-5	100	1	0.01	0.05
F	Dichlorofluoromethane	73-43-4	100	1	0.01	0.05
A	1,2-Dichloropropane	78-87-5	100	1	0.01	0.05
A	1,3-Dichloropropane	142-28-9	100	1	0.01	0.05
A	2,2-Dichloropropane	594-20-7	100	1	0.01	0.05
A	1,1-Dichloropropylene	563-58-6	100	1	0.01	0.05
A	cis-1,3-Dichloropropylene	10061-01-5	100	1	0.01	0.05
A	trans-1,3-Dichloropropylene	10061-02-6	100	1	0.01	0.05
A	Ethylbenzene	100-41-4	100	1	0.01	0.05
E	Ethyl ether	60-29-7	100	5	0.1	0.5
E	Heptane	142-82-5	100	10	0.1	0.5

Attachment 23.1
Table 1
Compound List, CAS Number and Routine Reporting Limits for Standard I (continued)

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting Limit		
				Aqueous ug/L	Low Level Soil, mg/kg	High Level Soil, mg/kg
A	Hexachlorobutadiene	87-68-3	100	1	0.01	0.05
E	Hexachloroethane	67-72-1	100	1	0.01	0.05
D	2-Hexanone	591-78-6	100	5	0.1	2.5
D	Iodomethane	74-88-4	100	1	0.01	0.05
E	Isopropanol	67-63-0	100	25	0.1	2.5
A	Isopropylbenzene	88-82-8	100	1	0.01	0.05
A	p-Isopropyltoluene	99-87-6	100	1	0.01	0.05
E	Methyl tert-butyl ether (MTBE)	1634-04-4	100	1	0.1	2.5
E	Methyl Acetate	79-20-9	100	10	0.01	0.25
E	Methylcyclohexane	108-87-2	100	10	0.01	0.25
A	Methylene chloride	75-09-2	100	1	0.01	0.25
D	Methyl ethyl ketone (2-Butanone)	78-93-3	100	5	0.1	0.75
E	2-Methylnaphthalene	91-57-6	100	5	0.05	0.25
D	4-Methyl-2-pentanone (MIBK)	108-10-1	100	5	0.1	2.5
A	Naphthalene	91-20-3	100	5	0.01	0.5
A	n-Propylbenzene	103-65-1	100	1	0.01	0.05
A	Styrene	100-42-5	100	1	0.01	0.05
A	Tetrachloroethylene	127-18-4	100	1	0.01	0.05
A	1,1,1,2-Tetrachloroethane	630-20-6	100	1	0.01	0.05
A	1,1,2,2-Tetrachloroethane	79-34-5	100	1	0.01	0.05
A	Toluene	108-88-3	100	1	0.01	0.05
A	1,2,3-Trichlorobenzene	87-61-6	100	1	0.01	0.05
A	1,2,4-Trichlorobenzene	120-82-1	100	1	0.01	0.05
A	1,1,1-Trichloroethane	71-55-6	100	1	0.01	0.05
A	1,1,2-Trichloroethane	79-00-5	100	1	0.01	0.05
A	Trichloroethylene	79-01-6	100	1	0.01	0.05
B	Trichlorofluoromethane	75-69-4	100	1	0.01	0.05
A	1,2,3-Trichloropropane	96-18-4	100	1	0.01	0.05
E	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	100	1	0.01	0.05
A	1,2,4-Trimethylbenzene	95-63-6	100	1	0.01	0.05
A	1,3,5-Trimethylbenzene	108-67-8	100	1	0.01	0.05
D	Vinyl acetate	108-05-4	100	5	0.01	0.05
B	Vinyl chloride	75-01-4	100	1	0.01	0.05
A	m,p-Xylene	106-42-3	200	1	0.02	0.1
A	o-Xylene	95-47-6	100	1	0.01	0.05
A	Xylene, total	1330-20-7	300	1	0.03	0.15

*Mix A: Volatile Organic Compounds - Liquids @ 2.0 mg/mL in MeOH (Accustandard M502A-R-10X)

*Mix B: Volatile Organic Compounds - Gases @ 2.0 mg/mL in MeOH (Accustandard M502B-10X)

*Mix C: Acrolein & Acrylonitrile at 2.0 mg/mL in MeOH (Accustandard S-948)

*Mix D: Ketone Mix @ 2.0 mg/mL in MeOH (Accustandard M-8260-ADD-10X)

*Mix E: Custom Additions to Method 8260B @ 2.0 mg/mL in MeOH (Accustandard S-3439-R1)

*Mix F: Dichlorofluoromethane @ 1.0 mg/mL in MeOH (Absolute #70904)

Attachment 23.2
Table 1A
Compound List, CAS#, And Routine Reporting Limits For Standard II

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting		
				Aqueous ug/L	Low Level Soil mg/kg	High Level Soil mg/kg
A	Allyl chloride	107-05-1	250	5	0.05	0.25
A	2-Butanol	15892-23-6	250	50	0.5	2.5
A	n-Butanol	71-36-3	250	50	0.5	2.5
A	t-Butanol	75-65-0	250	50	0.5	2.5
Neat	n-Butyl acetate	123-86-4	250	10	0.02	0.5
B	2-Chloro-1,3-butadiene (Chloroprene)	126-99-8	250	5	0.05	0.25
Neat	1-Chlorohexane	544-10-5	250	1	0.01	0.05
E	Cyclohexane	110-82-7	100	10	0.01	0.25
A	Cyclohexanone	108-94-1	250	50	0.5	2.5
A	2,3-Dichloro-1-propene	78-88-6	250	5	0.05	0.25
A	1,4-Dioxane	123-91-1	250	25	0.1	2.5
C	Epichlorohydrin	106-89-8	250	25	0.05	1.3
A	Ethanol	64-17-5	250	50	0.5	2.5
A	Ethyl acetate	141-78-6	250	10	0.5	2.5
Neat	Ethyl methacrylate	97-63-2	250	5	0.05	0.25
A	Hexachloroethane	67-72-1	250	5	0.05	0.25
A	Hexane	110-54-3	250	10	0.1	0.5
A	Isobutanol	78-83-1	250	50	0.5	2.5
A	Isobutyl acetate	110-19-0	250	5	0.1	0.5
Neat	Isopropanol	67-63-0	250	25	0.05	2.5
A	Isopropyl ether	108-20-3	250	5	0.01	0.05
A	Methacrylonitrile	126-98-7	250	5	0.5	2.5
E	Methylcyclohexane	108-87-2	100	10	0.01	0.25
Neat	Methyl methacrylate	80-62-6	250	5	0.05	0.25
A	2-Nitropropane	79-46-9	250	5	0.1	0.5
A	n-Propanol	71-23-8	250	50	0.5	2.5
A	Propionitrile	107-12-0	250	5	0.5	2.5
A	Tetrahydrofuran	109-99-9	250	5	0.1	0.5

*Mix A: Appendix IX Standard @ 2.5 mg/mL in MeOH (Accustandard S-3651 Volatile Custom Solution)
*Mix B: 2-Chloro-1,3-butadiene (Chloroprene) @ 2.0 mg/mL in MeOH (Accustandard App-9-048-R1-20X)
*Mix B: Epichlorohydrin @ 1.0 mg/mL in MeOH (Accustandard 70377)

Attachment 23.3
Table 2
BFB Key Ion Abundance Criteria

Table 2
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
96	5 to 9% of mass 95
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

Attachment 23.4
Table 3
Standard 8260B Volumes Required For Curve

Concentration of Working Standard (ug/L)	Volume (µL) of Standard 12.4.1.1	Additional Volume (µL) of Methanol Required
1.0	0.5	999.5
5.0	2.5	997.5
10	5.0	995
20	10	990
40	20	980
100	50	950
200	100	900

Attachment 23.5
Table 3A
Appendix IX Volumes Required For Curve

Concentration of Working Standard (ug/L)	Volume (µL) of Standard 12.4.2.1	Additional Volume (µL) of Methanol Required
5.0	1.0	999
25	5.0	995
50	10	990
100	20	980
250	50	950
500	100	900
1000	20*	980

*Note: when preparing the 1000 ug/L Appendix IX calibration standard, use the stock standard listed in 12.3.4.2.

Attachment 23.6
Table 4
Elution Order, Quantitation And Characteristic Ions, Internal Standards, And Surrogates For Standard I

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
1	Dichlodifluoromethane	85	87, 50	1
2	Chloromethane	50	52, 49	1
3	Vinyl Chloride	62	64, 47	1
4	Bromomethane	94	96, 93	1
5	Chloroethane	64	66	1
6	Trichlorofluoromethane	101	103, 105	1
7	Dichlorofluoromethane	67	69, 47	1
8	Ethyl Ether	45	59, 73	1
9	Acrolein	55	56, 53	1
10	1,1-Dichloroethylene	96	61, 98	1
11	1,1,2-Trichloro-1,2,2-trifluoroethane	101	103, 151	1
12	Iodomethane	142	127, 141	1
13	Carbon Disulfide	76	78, 77	1
14	Acetone	43	58, 42	1
17	Methylene Chloride	49	51, 84	1
18	Acrylonitrile	52	54, 53	1
19	Trans-1,2-Dichloroethylene	96	61, 98	1
20	Methyl tert-Butyl Ether (MTBE)	73	43, 57	1
21	1,1-Dichloroethane	63	65,83	1
22	Vinyl Acetate	43	42, 44	1
23	2,2-Dichloropropane	77	41, 79	1
24	cis-1,2-Dichloroethylene	96	61, 63	1
25	Methyl Ethyl Ketone (2-Butanone)	43	72, 57	1
26	Bromochloromethane	49	130, 128	1
27	Tetrahydrofuran	71	41, 42	1
28	Chloroform	83	85, 47	1
29	1,1,1-Trichloroethane	97	99, 61	1
30	SUR:Dibromofluoromethane	113	111, 192	1
31	IS:Fluorobenzene	96	70,50	1
32	Carbon Tetrachloride	117	119, 121	1
33	Cyclohexane	41	39, 57	1
34	1,1-Dichloropropylene	75	110, 77	1
35	Benzene	78	51, 50	1
36	1,2-Dichloroethane	62	64, 49	1
37	1,2-Dichloroethane-d4	65	67,51	1
38	Trichloroethylene	130	95, 132	1
39	1,2-Dichloropropane	63	62, 76	1
40	Dibromomethane	93	95, 174	1
41	Bromodichloromethane	83	85, 129	1

Attachment 23.6
Table 4
Elution Order, Quantitation And Characteristic Ions, Internal Standards, And Surrogates For Standard I (Cont.)

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
44	cis-1,3-Dichloropropylene	75	77, 110	1
45	4-Methyl-2-Pentanone (MIBK)	43	58, 85	1
46	SUR:d8-Toluene	98	100,70	1
47	Toluene	91	92, 65	1
48	Trans-1,3-Dichloropropylene	75	77, 110	1
49	1,1,2-Trichloroethane	97	83, 85	1
50	Tetrachloroethylene	166	129, 168	2
51	1,3-Dichloropropane	76	78, 41	2
52	2-Hexanone	43	58, 85	2
53	Dibromochloromethane	129	127, 131	2
54	1,2-Dibromoethane	109	107, 188	2
55	IS:d5-Chlorobenzene	82	117, 119	2
56	Chlorobenzene	112	77, 114	2
57	1,1,1,2-Tetrachloroethane	131	133, 119	2
58	Ethylbenzene	91	106, 51	2
59	m,p-Xylene	91	106, 51	2
60	o-Xylene	91	106, 51	2
61	Styrene	104	78, 51	2
62	Bromoform	173	175, 79	2
63	Isopropylbenzene	105	120, 79	3
64	SUR:4-Bromofluorobenzene	95	174, 176	2
65	Bromobenzene	77	156, 158	3
66	1,1,2,2-Tetrachloroethane	83	85, 95	3
67	1,2,3-Trichloropropane	75	49, 110	3
68	n-Propylbenzene	91	120, 65	3
69	2-Chlorotoluene	126	91, 63	3
70	1,3,5-Trimethylbenzene	105	120, 77	3
71	4-Chlorotoluene	91	126, 63	3
72	tert-Butylbenzene	119	91, 134	3
73	sec-Butylbenzene	105	134, 91	3
74	1,3-Dichlorobenzene	146	111, 148	3
75	p-Isopropyltoluene	119	134, 91	3
76	IS:d4-1,4-Dichlorobenzene	152	150	3
77	1,4-Dichlorobenzene	146	111, 148	3
78	1,2-Dichlorobenzene	146	111, 148	3
79	n-Butylbenzene	91	92, 134	3
80	1,2-Dibromo-3-Chloropropane	75	155, 157	3
81	1,2,4-Trichlorobenzene	180	182, 145	3
82	Hexachlorobutadiene	225	227, 260	3
83	Naphthalene	128	102, 51	3
84	1,2,3-Trichlorobenzene	180	182, 145	3

Attachment 23.7
Table 4A
Elution Order, Quantitation And Characteristic Ions, For Standard II

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
1	Ethanol	45	43	1
2	Acetonitrile	41	40, 54	1
3	Allyl Chloride	76	39, 41	1
4	Isopropanol	45	59, 43	1
5	t-Butanol	59	41, 43	1
6	Hexane	41	56, 57	1
7	Isopropylether	45	69, 86	1
8	n-Propanol	42	59, 41	1
9	Methylcyclopentane	56	41, 69	1
10	Propionitrile	54	52, 56	1
11	Ethyl Acetate	43	61, 45	1
12	Methacrylonitrile	67	41, 52	1
13	2-Butanol	45	59, 57	1
14	Sur: Dibromofluoromethane	113	111, 79	1
15	Cyclohexane	41	39, 57	1
16	IS: Fluorobenzene	96	70,50	1
17	Isobutanol	41	43, 42	1
18	n-Butanol	41	39, 56	1
19	1,2-Dichloroethane-d4	65	67,51	1
20	2,3-Dichloro-1-propylene	75	77, 110	1
21	Methyl Methacrylate	41	69, 100	1
22	1,4-Dioxane	88	57, 43	1
23	2-Nitropropane	41	43, 46	2
24	Epichlorohydrin	49	57, 62	2
25	Hexachloroethane	117	119, 201	3
26	Ethyl methacrylate	41	99, 69	2
27	Sur: d-8-Toluene	98	100, 70	2
28	n-Butyl acetate	43	56, 57	2
29	IS: d5-Chlorobenzene	82	117, 119	2
30	Cyclohexanone	55	42, 98	2
31	Sur: 4-Bromofluorobenzene	95	174, 176	2
32	trans-1,4-Dichloro-2-butylene	53	75, 89	3
33	IS: d4-1,4-Dichlorobenzene	152	150	3

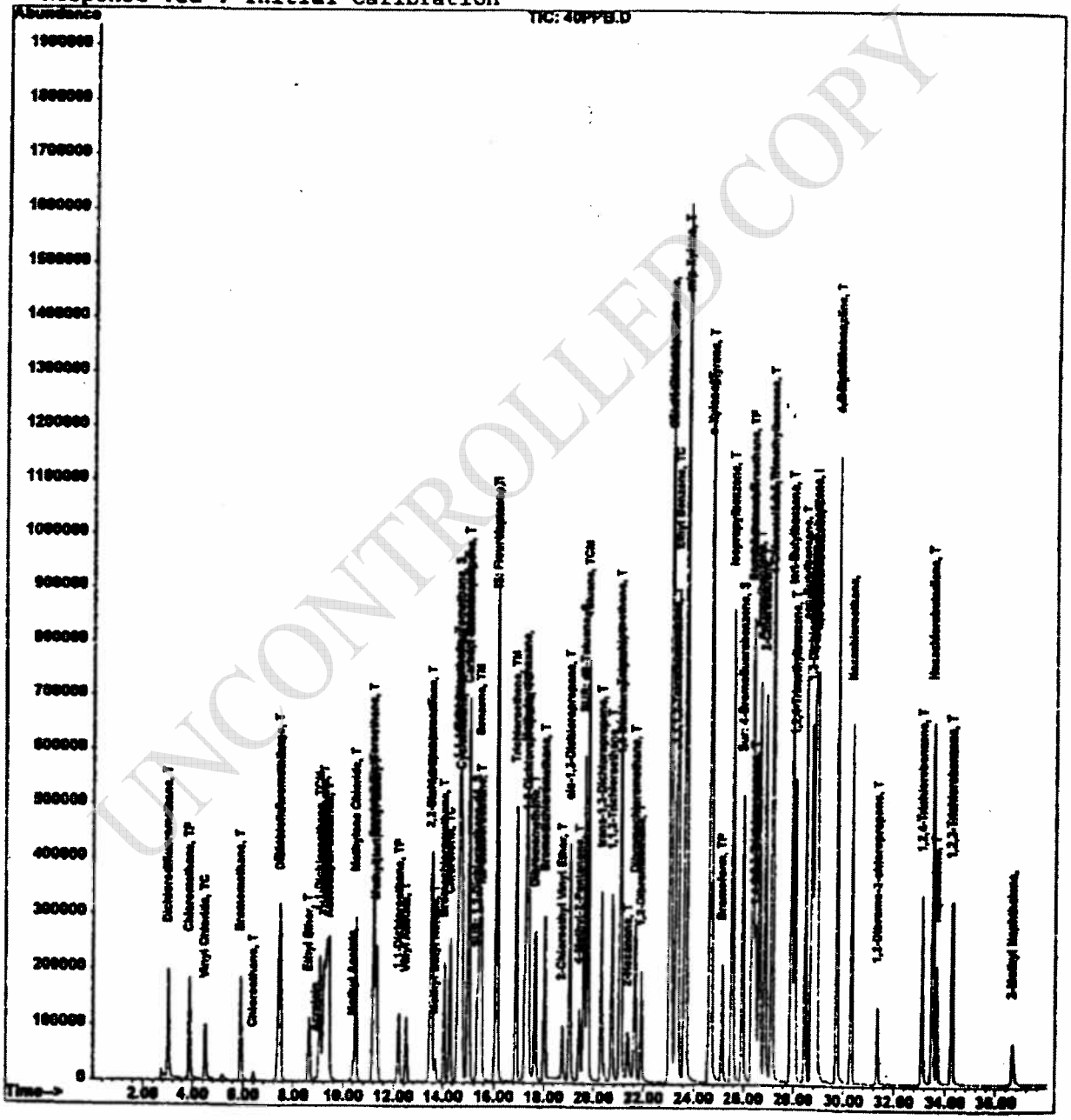
Attachment 23.8
Table 5
Continuing Calibration QC Acceptance Criteria
Method 624

Parameter	Range for Q (ug/L)
Benzene	25.6-54.4
Bromodichloromethane	26.2-53.8
Bromoform	28.4-51.6
Bromomethane	5.6-74.4
Carbon tetrachloride	29.2-50.8
Chlorobenzene	26.4-53.6
Chloroethane	15.2-64.8
2-Chloroethylvinyl ether	D-89.6
Chloroform	27.0-53.0
Chloromethane	D-81.6
Dibromochloromethane	27.0-53.0
1,2-Dichlorobenzene	25.2-54.8
1,3-Dichlorobenzene	29.2-50.8
1,4-Dichlorobenzene	25.2-54.8
1,1-Dichloroethane	29.0-51.0
1,2-Dichloroethane	27.2-52.8
1,1-Dichloroethylene	20.2-59.8
trans-1,2-Dichloroethylene	27.8-52.2
1,2-Dichloropropane	13.6-66.4
cis-1,3-Dichloropropylene	9.6-70.4
trans-1,3-Dichloropropylene	20.0-60.0
Ethyl benzene	23.6-56.4
Methylene chloride	24.2-55.8
1,1,2,2-Tetrachloroethane	24.2-55.8
Tetrachloroethylene	29.4-50.6
Toluene	29.8-50.2
1,1,1-Trichloroethane	30.0-50.0
1,1,2-Trichloroethane	28.4-51.6
Trichloroethylene	26.6-53.4
Trichlorofluoromethane	19.2-60.8
Vinyl chloride	1.6-78.4-

D=Detected result must be greater than zero.

Attachment 23.9
Example Chromatogram, DB-624

```
Method        : C:\Sat132\QUANT\8260b49.m (RTE Integrator)
Title         : Saturn 132
Last Update   : Thu May 09 11:09:55 2002
Response via  : Initial Calibration
```



Attachment 23.10
Example Chromatogram, DB-VRX

```
Method        : C:\HPCHEM\2\METHODS\8260WT23.M (RTE Integrator)
Title         : EPA METHOD 624/8240
Last Update   : Thu Jun 06 15:21:31 2002
Response via  : Initial Calibration
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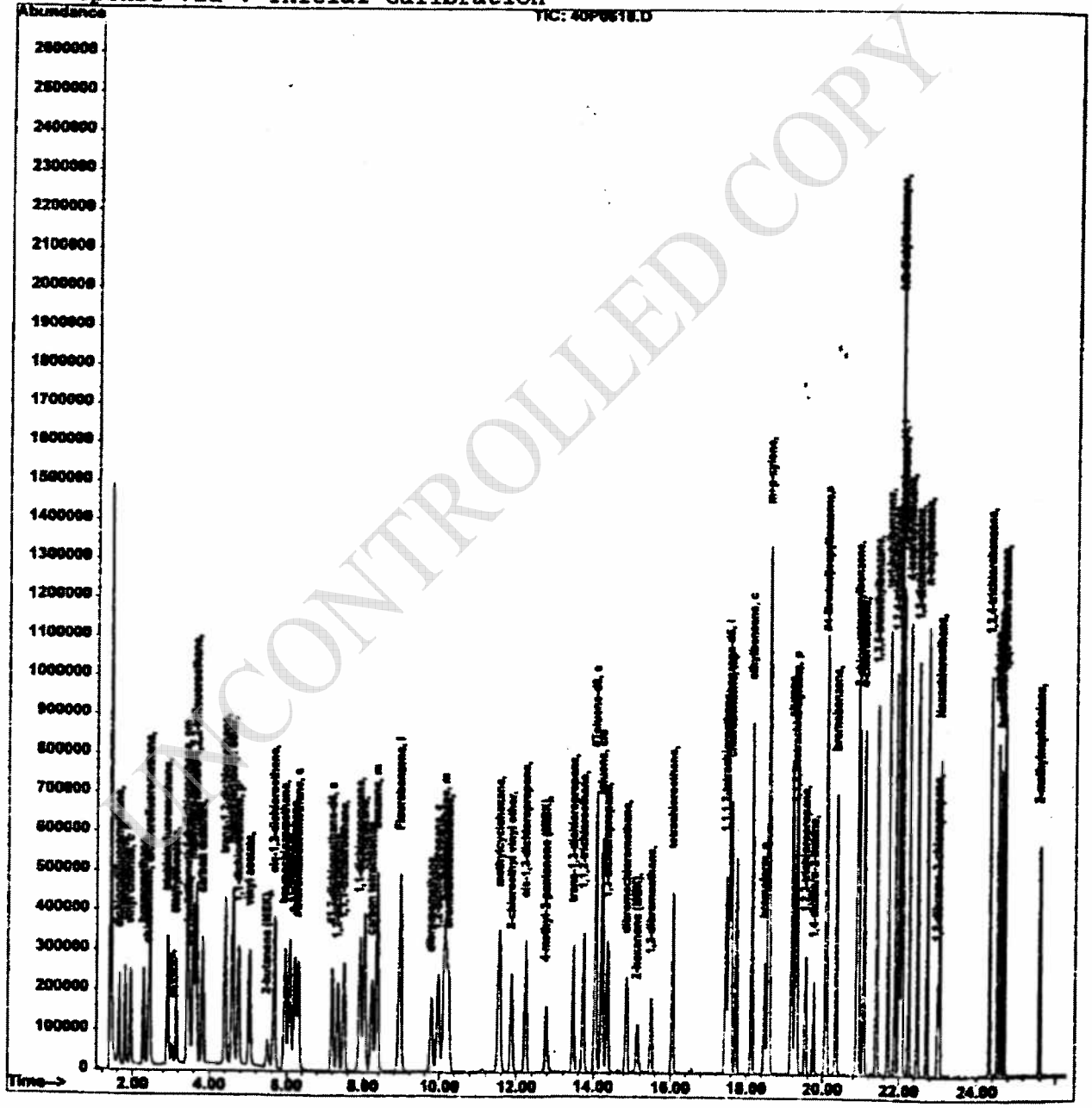

Approved By: QA 5-6-10 **QA Officer**

Approved By: DLV 5-6-10 **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Total, Bicarbonate, Carbonate and Hydroxide Alkalinity by Titration

EPA Method 310.1
Standard Methods 2320 B

APPROVALS:

Area Supervisor: Heather L. Brady Date: 1-31-09
Heather L. Brady

QA Officer: Date: 1-29-09
Tom C. Boocher

Operations Manager: Date: 2/4/09
Jeff P. Glaser

Procedure Number: GR-06-101
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By: Iryna N. Ruptash
Total Number of Pages: 19

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-19-10		4-19-11

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to water, wastewater and aqueous waste.

1.2 The procedure is suitable for all concentration ranges of alkalinity. However, appropriate aliquots must be used to avoid a titration volume greater than 50 mL.

1.3 The reporting limit is 2 mg/L.

2.0 PRINCIPAL METHOD REFERENCES

2.1 *Methods for Chemical Analysis of Water and Wastes,* March, 1983, "Alkalinity", Method 310.1 (Titrimetric, pH 4.5), Issued 1971, Editorial revision 1978

2.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998,* Alkalinity, 2320 B. "Titration Method"

3.0 SUMMARY OF PROCEDURE

3.1 Alkalinity is the acid-neutralizing capacity of a water solution. It is the sum of all titratable bases. The measured value will vary significantly with the pH end-point used. Alkalinity is a measure of an aggregate property of water and can be interpreted in terms of specific substances only when the chemical composition of the sample is known.

3.2 Alkalinity of a typical water sample is assumed to be attributable to one or a combination of hydroxide, carbonate and bicarbonate.

3.3 Sample is titrated with dilute acid to two endpoints; pH = 8.3 and pH = 4.5. From the titration, the proportions of each form of alkalinity can be determined. Alkalinity is reported as mg $CaCO_3$/L.

4.0 PARAMETER OR COMPOUND LIST

4.1 Total Alkalinity (as $CaCO_3$)

4.2 Bicarbonate Alkalinity (as $CaCO_3$)

4.3 Carbonate Alkalinity (as $CaCO_3$)

4.4 Hydroxide Alkalinity (as $CaCO_3$)

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Correction Actions*, latest revision

5.3 TriMatrix SOP GR-16-108, *Glassware Cleaning for Wet Chemistry and Metals*, latest revision

5.4 TriMatrix SOP GR-10-104, *Chain-of-Custody*, latest revision

5.5 TriMatrix SOP GR-07-100, *Potentiometric pH*, latest revision

5.6 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soaps, oily matter, suspended solids, or precipitates can coat the glass pH electrode and cause a sluggish response. The electrode must be kept clean.

6.2 Since this test is meant to determine all forms of alkalinity present in the sample as originally received, samples must not be filtered, concentrated or altered in any way.

6.3 Groundwater samples containing sediment or soil that interferes with the endpoint may be centrifuged to remove solids. This is done only after manager approval.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratories, Laboratory Safety Manual and Chemical Hygiene Plan.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are to be collected in inert plastic bottles and stored at 4 ±2° C until time of analysis.

8.2 Clean samples must be homogenized by shaking just prior to removing a sample aliquot.

8.3 Samples with sediment (groundwaters) must not be shaken. Decant an aliquot for analysis, being careful not to disturb the sediment.

Note: If a sample requires suspended solids analysis, perform the suspended solid analysis prior to decanting for alkalinity.

8.4 Analysis must be performed within 14 days of sample collection.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Mettler, model DL-12 titrator

9.2 Volumetric pipets

9.3 Graduated cylinders

9.4 Sample cups, disposable

9.5 Volumetric flasks

9.6 10 mL micro buret

9.7 Orion pH meter

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 When not in use, close the fill hole and store the pH electrode in a pH 4 or 7 buffer solution. Never store the electrode in laboratory reagent water, as this may lead to junction clogging and slow response.

10.2 A dirty electrode will also contribute to sluggish response. A dirty electrode is indicated by beads of water forming on the surface of the electrode when rinsed with laboratory reagent water. Clean inorganic residues from the electrode with EDTA, ammonia, or acids. Clean organic deposits such as grease and similar films with acetone or methanol.

10.3 The buret cylinder, piston, stopcock, and tubing must be cleaned monthly following the instructions provided in the operating manual. Additional maintenance items are specified in the instrument operating manual.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM Type II, MilliQ system

11.2 Hydrochloric acid (HCl), concentrated

11.3 Hydrochloric acid (HCl) 1.0N

11.3.1 Add approximately 500 mL of reagent water to a 1000 mL volumetric flask. Add 83 mL of concentrated hydrochloric acid, swirl to mix, and dilute to volume with reagent water.

11.4 Hydrochloric Acid (HCl) 0.1N

11.4.1 Add approximately 500 mL of reagent water to a 1000 mL volumetric flask. Pipet in 100 mL of 1.0 HCl, and dilute to volume with reagent water.

11.5 Hydrochloric acid (HCl) 0.02N

11.5.1 Add approximately 500 mL of reagent water to a 1000 mL volumetric flask. Pipet in 20.0 mL of 1.0N HCl, and dilute to volume with reagent water.

11.6 Sodium carbonate (Na_2CO_3)

11.7 Standardizing solution, 0.050N sodium carbonate - available commercially, or prepared as in 11.7.1.

11.7.1 Weigh 2.65 ±0.2 g Na_2CO_3 (dried at 250° C for 4 hours and cooled in desiccator) into a 1 L volumetric flask, dilute to volume with reagent water. Prepare this solution weekly.

11.8 Sodium bicarbonate ($NaHCO_3$)

11.9 All reagent preparations must be recorded with a unique identification number (Attachment 23.1 and/or 23.2).

12.0 STANDARDS PREPARATION

12.1 Dry the Na_2CO_3, $CaCO_3$, and $NaHCO_3$ at 250° C for 4 hours and cool in desiccator prior to preparing standards.

12.2 Carbonate stock: 1887 mg/L as $CaCO_3$

12.2.1 Weigh 2.00 g sodium carbonate (Na_2CO_3) into a 1 L volumetric flask, and dilute to volume with reagent water. Prepare new stock every 6 months.

12.3 Bicarbonate stock: 1190 mg/L as $CaCO_3$

12.3.1 Weigh 2.00 g sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask, and dilute to volume with reagent water. Prepare new stock every 6 months.

12.4 Hydroxide stock: 1000 mg/L as $CaCO_3$

12.4.1 Add approximately 500 mL of reagent water to a 1 L volumetric flask. Add 2.0 mL of the 0.01N sodium hydroxide solution, and dilute to volume with reagent water. Prepare new stock every 6 months.

12.5 All standard preparations must be recorded with a unique identification number (Attachment 23.1 and/or 23.2).

13.0 SAMPLE PREPARATION

13.1 Samples must be warmed to room temperature 25 ±5° C prior to analysis.

13.2 If groundwater samples have soil or sediment that interferes with the endpoint, centrifuging to remove soil and sediment may be performed if approved. An analysis narrative must be recorded indicating if centrifuging was performed prior to analysis.

14.0 CALIBRATION PROCEDURES

14.1 Calibration of electrode - to select buffers

14.1.1 Touch[1]-[ELEC CALIB] for buffer 4 and 7.

14.1.2 Touch[2]-[ELEC CALIB] for buffer 7 and 10.

14.1.3 Touch[3]-[ELEC CALIB] for buffer 4 and 10.

14.1.4 Choose one calibration range only. Usually the 4 and 10 buffer are selected.

14.2 To calibrate:

14.2.1 Display blinks with "Buffer A".

14.2.2 Place electrode in first buffer and touch [START].

14.2.3 Display blinks with "Buffer B".

14.2.4 Place electrode in second buffer and touch [START].

14.2.5 Calibration values are stored automatically.

14.3 Analyze a pH SCV and pH buffer ICV at this time to confirm the pH probe calibration.

15.0 ANALYTICAL PROCEDURE

15.1 Instrument set up

15.1.1 Turn on the DL12 titrator using button on upper left corner.

15.1.2 Push [Reset] button

15.1.3 Prime the buret as follows:

15.1.3.1 Place titrant line into the titrant being used.

15.1.3.2 Place an empty cup on the titrator to collect the titrant.

15.1.3.3 Push the "fill buret" button to initiate priming.

15.1.3.4 Repeat the buret filling three times.

15.1.3.5 After priming, rinse probe with laboratory reagent water.

15.1.4 To enter the titration control parameters

15.1.4.1 Type endpoint desired then touch [Endpoint]. Enter volume in mL to predose, then touch [Predose].

15.1.4.2 To change the speed of the titration, enter [1] slow or [2] normal and press [SPEED].

15.1.4.3 Use normal speed for normal titrations and slow speed for sharp/sudden endpoints.

15.2 Calibrate instrument following Section 14.0.

15.3 Standardization of HCl titrant:

15.3.1 Pipet 15 mL of 0.05N sodium carbonate solution into sample cup with about 60 mL reagent water. Titrate with 0.02N HCl down to pH of about 5. Lift out electrodes, rinse into the same beaker, and boil gently for 3-5 minutes under a watch glass. Cool to room temperature, rinse watch glass into beaker, finish titrating to pH of 4.5.

Calculate the normality of the acid with the following formula:

$$\text{Normality} = \frac{A \times B}{53.00 \times C}$$

where:

A = g Na_2CO_3 weighed into 1 L flask
B = mL Na_2CO_3 solution taken for titration, and
C = mL acid used

15.3.2 Standardize the 0.1N HCl with 40 mL of the 0.05N sodium carbonate solution according to the same procedure described in section 15.3.1.

15.4 Sample addition

15.4.1 Add 50 mL sample to a sample cup. Attach the sample cup to the titrator. Titrant delivery tube and pH electrode must be below the sample surface in the cup.

Note: If alkalinity concentration is less than 100 mg/L and greater than 20 mg/L, use a 100 mL sample volume and use the DL-12 slow speed for titration.

Note: For concentrations greater than 1000 mg/L use 0.1N HCl.

Note: For alkalinity concentrations less than 20 mg/L use 100 to 200 mL of sample titrate with the 0.02N HCl using a 10 mL micro-buret and pH meter. Add 0.05 mL at a time until the pH reaches 4.3 to 4.7. Record the volume and pH. Carefully add additional titrant to reduce the pH exactly 0.3 pH units and again record volume.

15.5 Measuring sample pH

15.5.1 Place electrode in sample-touch [pH MEAS].

15.5.2 The DL12 stirrer starts automatically.

15.5.3 Touch [RESET] to return to standby.

15.5.4 Record initial pH of the sample.

15.6 Titrating the sample

15.6.1 Touch [START]-"SAMPLE SIZE" blinking-enter sample size.

15.6.2 Touch [START]-"IDENT" blinking-enter ID if needed.

15.6.3 Touch [START]-"BUSY" blinking.

15.6.4 Touch [START] to begin titration.

15.6.5 For manual titration-hold [MAN TITR] down.

15.6.6 Touch [RESET] to abort titration or return to standby.

15.7 To analyze a sample

15.7.1 Titrate the sample using 0.02N HCl from initial pH to 8.3; record mL of titrant used as V_1 (if initial pH < 8.3, $V_1 = 0$).

15.7.2 Titrate from pH 8.3 to 4.5; record mL of titrant as V_2 (if initial pH < 4.5, $V_2 = 0$).

15.7.3 Input all data to the alkalinity spreadsheet located on the laboratory intranet library to determine all forms of alkalinity and calculate results.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Calculations of alkalinity using the spreadsheet available on the laboratory intranet library are as follows:

RESULT OF TITRATION	HYDROXIDE (OH^-)	CARBONATE (CO_3^{-2})	BICARBONATE (HCO_3^-)	TOTAL ALKALINITY AS $CaCO_3$
$V_1 = 0$	0	0	V_2	V_2
$V_1 < V_2$	0	V_1	$V_2 - V_1$	$V_1 + V_2$
$V_1 = V_2$	0	V_1	0	$V_1 + V_2$
$V_1 > V_2$	$V_1 - V_2$	V_2	0	$V_1 + V_2$
$V_2 = 0$	V_1	0	0	V_1

16.2 Discussion of alkalinity relationships

16.2.1 $V_1 = 0$

16.2.1.1 For pH < 8.3, no OH^- or CO_3^{-2} exists, and all alkalinity is assumed to be HCO_3^-.

16.2.2 $V_1 < V_2$

16.2.2.1 Some CO_3^{-2} is present. Bicarbonate titration (V_2) also titrates the CO_3^{-2}, which was protonated during V_1 part of titration, so V_1 must be subtracted from bicarbonate, V_2, prior to calculation for bicarbonate alkalinity.

16.2.3 $V_1 = V_2$

16.2.3.1 All alkalinity is CO_3^{-2}. The titration converts the CO_3^{-2} to HCO_3^- in the V_1 step, then titrates this HCO_3^- in the V_2 step. Therefore, V_2 is not used in the calculation.

16.2.4 $V_1 > V_2$

16.2.4.1 All alkalinity is OH^- and CO_3^{-2}. The V_1 step converts OH^- to water and CO_3^{-2} to bicarbonate. The bicarbonate formed from CO_3^{-2} titrates in the V_2 step, so V_2 is used for CO_3^{-2} calculation. OH^- is calculated from V_1 minus the part attributable to CO_3^{-2} (V_2).

16.2.5 $V_2 = 0$

16.2.5.1 All alkalinity is due to OH^- when a high pH sample (pH>8.3) is titrated to 8.3 but immediately drops to pH < 4.5 ($V_2 = 0$). The absence of weak bases such as CO_3^{-2} and HCO_3^- results in a lack of buffering ability in the sample, and pH fluctuates rapidly.

16.3 After the forms of alkalinity present in a sample have been determined, the values for volume of titrant used are put into the formulas below to calculate the parameter desired.

16.4 Calculations

PARAMETER	FORMULA
Alkalinity, Total (as $CaCO_3$)	$\frac{(V_2 + V_1) \times 50{,}000 \times N}{S}$
Bicarbonate Alkalinity (as $CaCO_3$)	$\frac{(V_2 - V_1) \times 50{,}000 \times N}{S}$
Carbonate Alkalinity (as $CaCO_3$)	$\frac{Lowest\ V\ Value \times 100{,}000 \times N}{S}$
OH Alkalinity (as $CaCO_3$)	$\frac{(V_1 - V_2) \times 50{,}000 \times N}{S}$

where

V_1 = Volume in mL of titrant needed to lower sample pH to 8.3
V_2 = Volume in mL of titrant needed to lower sample pH from 8.3 to 4.5
S = Volume of sample titrated in mL
N = Normality of titrant

16.5 Calculation for alkalinity concentrations less than 20 mg/L using the 10 mL buret:

$$\text{Alkalinity, Total mg } CaCO_3/L \quad \frac{(2B - C) \times N \times 50{,}000}{\text{mL Sample}}$$

Where:
B = mL titrant to first recorded pH
C = Total mL titrant to reach pH 0.3 unit lower
N = Normality of acid

17.0 DATA REPORTING AND DELIVERABLES

17.1 The analyst processing samples is responsible for data quality and for correctly filling in the proper documentation. This is required for quality control and to provide the client with defensible data.

17.2 The following must be reported with each batch analyzed:

- Standard log numbers of stock standards used
- Normality of titrant used
- mL of titrant and sample volume for each analysis
- Reported concentration in units of mg/L as $CaCO_3$
- Original sample pH

17.3 The instrument run logbook must be filled in for each batch analyzed with the following information:

- date analyzed
- analyst's initials
- method name and number
- calibration standards used
- client name and sample numbers analyzed

17.4 All logbooks must be filled in completely and correctly. All corrections are to be made in indelible ink. **Corrections are to be made with a single lineout, which is then dated and initialed. Write-overs are not acceptable.** The erroneous result must remain legible. The new result is placed near the incorrect result. Blank lines in the logbook must be Z'd out.

17.5 If internal chain-of-custody is required it is very important that the COC form be filled in correctly.

18.0 QUALITY ASSURANCE

18.1 Method and matrix QC must be analyzed with each batch.

18.1.1 Method QC for aqueous samples consists of an Instrument Blank (BLK), Second-Source Calibration Verification (SCV), Initial Calibration Verification (ICV), Continuing Calibration Verifications (CCV), and a non-reported pH SCV used to confirm the probe calibration.

18.1.1.1 A BLK and a SCV are analyzed with every daily batch.

18.1.1.1.1 The BLK is an aliquot of reagent water analyzed like a sample. The results must be less that the reporting limit for the analysis.

18.1.1.1.2 The SCV is an aliquot of the appropriate stock standard prepared in section 12.0 analyzed like a sample.

18.1.1.1.2.1 To prepare a SCV, pipet 10 mL of appropriate standard into 50 mL of reagent water and titrate like a sample. Calculate the true value of the SCV using the following equation:

$$\frac{A \times 10}{50} = \text{mg / L } CaCO_3$$

where
A= Concentration of the stock standard

18.1.1.1.2.2 All SCV standard preparations must be recorded in the standard logbook with their own unique ID numbers. All SCV standards must be labeled with the following information:

- initials of preparer
- date of preparation
- concentration
- name of parameter
- log number as it appears in the standard logbook

18.1.1.1.2.3 The acceptance limits for the SCV are in the Laboratory Information Management System (Element™).

18.1.1.2 The ICV is an aliquot of a pH buffer (4, 7, or 10) analyzed immediately after the curve. The acceptance limit for the ICV is ± 0.05 pH units from the true value.

18.1.1.3 CCVs are aliquots of pH buffers (4, 7, or 10) that are analyzed after every ten samples and at the end of every batch. The acceptance range for CCVs is ± 0.05

pH units from the true value (included in the ten samples are the SCV, and the Duplicate (DUP)).

18.1.1.4 The non-reported pH SCV is an aliquot of a purchased pH standard (usually a 7.40 pH Beckman standard, or equivalent) that is analyzed on the pH probe immediately after calibration of the probe to confirm the calibration. The acceptable limits for this SCV are ± 0.05 pH units from the true value.

18.1.2 Matrix QC consists of a DUP, and a matrix spike (SPK).

18.1.2.1 The DUP is a replicate sample analysis. The maximum relative percent difference is 20%.

18.1.2.2 To prepare a SPK, pipet 10 mL of appropriate standard into 50 mL of a sample. Calculate the spike concentration using the following equation:

$$\frac{A \times 10}{50} = mg/L\ CaCO_3$$

where
A = concentration of the stock standard

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Spike four samples with the SCV prepared in section 18.1.1.1.2.1. Alternatively, the last four of the seven samples used in the MDL study may be used in the IDC. ONLY the last four of the seven samples may be used. Process the four samples following the procedures outlined in the SOP. Calculate the average percent recoveries and the standard deviations of the 4 recoveries. Average percent recovery must be within current SCV acceptance limits. The maximum acceptable relative standard deviation (RSD) is 20%.

19.1.1.2 If either criterion is not met, locate and correct the source of the problem and repeat the test. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test. Samples may not be analyzed by any analyst or on any instrument until the analyst certification has been successfully completed. Copies of successful IDC/Method Validation spreadsheets and raw data must be given to the Quality Assurance Department.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A CDC must be performed annually by all analysts running samples by this SOP.

19.1.2.2 A CDC may be accomplished by repeating the IDC procedure using specific CDC samples, by processing four consecutive SCVs following the IDC procedure, or by submitting an acceptable PT sample on the appropriate paperwork for inclusion in the analyst's training file.

19.2 A Method Detection Limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve use of chemicals where applicable

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Methods for Chemical Analysis of Water and Wastes,* March, 1983, "Alkalinity", Method 310.1 (Titrimetric, pH 4.5), Issued 1971, Editorial revision 1978

22.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998,* Alkalinity, 2320 B. "Titration Method"

23.0 ATTACHMENTS

23.1 Analytical Standards Record Example

23.2 Standards Logbook Example

23.3 Analysis Sequence Report Example

23.4 Instrument Logbook Example

23.5 Preparation Batch Report Example

Attachment 23.1
Analytical Standards Record Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
6110408

Description	Alk.Carb LCS 377 mg/l	Expires	May-13-07
Standard Type	Calibration	Prepared	Nov-13-06
Solvent	Solvent Lot #	Prepared By	Irina N. Ruptash
Final Volume (mls)	50	Department	Expired
Vials	1	Last Edit	Jun-04-07 10:32 by CCC

50ml DI H2O + 10ml of 1887 mg l sodium carb

Analyte	CAS Number	Concentration	Units
Alkalinity, Carbonate		378	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
6110407	Alkalinity Carbonate 1887 mg/l	Nov-13-06	Irina N. Ruptash	May-13-07	Jun-04-07 10:32 by CCC	10

Reviewed By _____ Date _____

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Approved By: _____ 1-29-09
QA Officer

Approved By: Heather L Brady
Area Supervisor

Attachment 23.2
Standards Logbook Example

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By	Checked Against Standard #
1	IN1.21.1	orthophos	IN1.8.8			1000ppm	10 mL	H_2O	100 mL	100 ppm	SAT	11/3/00	11/4/02		
2	IN1.21.2	↓	IN1.8.9			↓	↓	↓	↓	↓	↓	↓	↓		
3	IN1.21.3	Chloride	764J.37			1000ppm	40mL	H_2O	100mL	400ppm	SAT	11/1/00	12/31/01		
4	IN1.21.4					1000ppm	15mL		100mL	150ppm					
5	IN1.21.5		8/0			1000ppm	25mL		250mL	100ppm					
6	IN1.21.6		↓			1000ppm	6mL		100mL	60ppm					
7	IN1.21.7	SAT	764J37			1000ppm	2.0mL		100mL	20ppm					
8	IN1.21.8		IN1.21.5			100ppm	10mL	SAT	100mL	10ppm	SAT	BC	BJ		
9	IN1.21.9		↓			100ppm	5mL		100mL	5.0ppm					
10	IN1.21.10		BJ			100ppm	2mL		100mL	2.0ppm					
11	IN1.21.11	Chloride	IN1.21.5			100ppm	2mL	H_2O	200mL	1.0ppm	SAT	11/2/00	12/31/01		
12	IN1.21.12	TOC	IN1.18.12			10ppm	1mL	H_2O	100mL	1.0ppm	JMB	11/6/00	11/6/01		
13	[illegible]	Sulfite				1.5758g	1000mL	H_2O	1000mL	1000ppm	JMB	10/30/00	10/30/01		
14	IN1.21.14	Sulfate	225854		3/1/01	1000mg/L	20mL	H_2O	100mL	200mg/L	MS	11/6/00	12/31/01 11/6/01		
15	IN1.21.15	↓	↓			↓	15mL	↓	100mL	150		↓	↓		
16	IN1.21.16						20mL		200mL	100					
17	IN1.21.17	↓	↓			↓	16mL	↓	200mL	80		↓			
18	IN1.21.18	Sulfate	225854			1000mg/L	5mL	H_2O	100mL	50		11/6/00	12/31/01 11/6/01		

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Approved By: 1-29-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.3
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. **ANALYSIS SEQUENCE** 7010814 Page 1 of 1 Printed: 1/8/2007 11:43:13AM

Inorganic - Wet Chemistry, Water, Jan-08-07

Instrument = 187, Calibration = UNASSIGNED

Sequence Analyses:
Alkalinity, Carbonate 310.1 0.02N HCl 25.942ml N = 0.0192737

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID* pH	*Client / QC Type*	*Extraction Comments* V_1 V_2 Conc.
7010814-ICV1	QC		A607484	6.99	INITIAL CAL CHECK	6.79
0700195-BLK1	QC			4.23	BLANK	<2.0
0700195-BS1	QC			10.53	LCS	9.6258 9.4757 366
7010814-SCV1	QC		6110408	10.53	SECONDARY CAL CHECK	9.6358 9.4757
0701035-01	Alkalinity, Carbonate 310.1	A		7.37	[redacted]	11.161 — <2.0
0701035-02	Alkalinity, Carbonate 310.1	A		7.25	[redacted]	8.984 — <2.0
0701035-03	Alkalinity, Carbonate 310.1	A		6.52	[redacted]	38.835 — <2.0
0701035-04	Alkalinity, Carbonate 310.1	A	5ml	7.34	[redacted]	25.8493 — <2.0
0701035-05	Alkalinity, Carbonate 310.1	C		6.99	[redacted]	35.942 — <2.0
0700195-DUP1	QC			6.87	DUPLICATE	36.914 — <2.0
7010814-CCV1	QC		A607484	7.02	CALIBRATION CHECK	7.02
0701035-06	Alkalinity, Carbonate 310.1	A		7.38	[redacted]	9.9651 — <2.0
0701035-07	Alkalinity, Carbonate 310.1	A		6.43	[redacted]	38.011 — <2.0
0701035-08	Alkalinity, Carbonate 310.1	A	10ml	7.68	[redacted]	45.924 — <2.0
0701035-09	Alkalinity, Carbonate 310.1	A		7.04	[redacted]	30.187 — <2.0
0701035-10	Alkalinity, Carbonate 310.1	A	5ml	8.13	[redacted]	29.3294 — <2.0
7010814-CCV2	QC		A607484	7.01	CALIBRATION CHECK	7.01

Comments: | Analyst Initials:

seq_TriMatrix.rpt

Approved By: [signature] 1-29-09 **QA Department**

Approved By: Heather L Brady **Operations Manager**

Attachment 23.4
Instrument Logbook Example

TriMatrix Laboratories, Inc.

Instrument Number 187 Run Logbook

Date	Analyst	Method Name or Number	Concentration of Calibration Standards Used							Units	Client Names and Sample Numbers
			No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7		
6/8/01	CEM	ALK	[illegible]	No. 1 4	No 2 7						[redacted]
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				

file: instlogbook.xls page: 1 of 50 revision: 1.0

Approved By: QD 1-29-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.5
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0700195 Page 1 of 1

Printed: 7/26/2007 6:27:43PM

Inorganic - Wet Chemistry, Water, General Inorganic Prep

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0701035	Alkalinity, Carbonate 310.1				

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0700195-BLK1		Jan-06-07 11:40	INR	100	100					BLANK	
0700195-DUP1		Jan-06-07 11:40	INR	50	50		0701035-05			DUPLICATE	
0700195-BS1		Jan-06-07 11:40	INR	50	50			6110407	10000	LCS	
0701035-01	A	Jan-06-07 11:40	INR	50	50					[redacted]	
0701035-02	A	Jan-06-07 11:40	INR	50	50					[redacted]	
0701035-03	A	Jan-06-07 11:40	INR	50	50					[redacted]	
0701035-04	A	Jan-06-07 11:40	INR	5	5					[redacted]	
0701035-05	C	Jan-06-07 11:40	INR	50	50					[redacted]	
0701035-06	A	Jan-06-07 11:40	INR	50	50					[redacted]	
0701035-07	A	Jan-06-07 11:40	INR	50	50					[redacted]	
0701035-08	A	Jan-06-07 11:40	INR	10	10					[redacted]	
0701035-09	A	Jan-06-07 11:40	INR	50	50					[redacted]	
0701035-10	A	Jan-06-07 11:40	INR	5	5					[redacted]	

Comments:	Analyst Initials

bch_TriMatrix.rpt

Approved By: [signature] 1-29-08 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Chloride (Konelab Ferricyanide)

EPA Method 325.2
Standard Methods 4500-Cl⁻ E
SW-846 Method 9251

APPROVALS:

Area Supervisor: Heather L. Brady Date: 1-27-09
Heather L. Brady

QA Officer: Tom C. Boocher Date: 1-26-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 1/30/09
Jeff P. Glaser

Procedure Number: GR-05-123
Revision Number: 0.1

Date Initiated: 7/27/04
Effective Date: 2/24/09

Date Revised: 1/24/09
Pages Revised: All

By: Jodi L. Blouw
Total Number of Pages: 16

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-19-10	Tom Boocher	4-19-11

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the analysis of chloride in drinking, surface, saline and wastewaters. Analysis can also be extended to water-extracted solid and waste samples which can then be reported as "soluble" or "exchangeable" chloride.

1.2 Samples ranging from 1 mg/L to 100 mg/L can effectively be analyzed. This range can be extended by sample dilution.

1.3 The reporting limit is 1.0 mg/L.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Methods for Chemical Analysis of Water and Wastes,* March, 1983, "Chloride (Colorimetric, Automated Ferricyanide AAII)", Method 325.2, Issued 1978

2.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998,* 4500-Cl^- Chloride, E, Automated Ferricyanide Method

2.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September, 1986, Method 9251, Chloride (Colorimetric, Automated Ferricyanide AAII)

Note: A Konelab discrete analyzer is used in place of the Technicon autoanalyzer.

3.0 SUMMARY OF PROCEDURE

3.1 Thiocyanate ion (SCN) is liberated from mercuric thiocyanate through sequestration of mercury by chloride ion to form un-ionized mercuric chloride.

3.2 In the presence of ferric ion, the liberated SCN forms highly colored ferric thiocyanate in a concentration proportional to the original chloride concentration.

3.3 Ferric thiocyanate absorbs strongly at 480 nm although the calibration curve is non-linear.

4.0 PARAMETER OR COMPOUND LIST

4.1 Chloride

5.0 REFERENCED SOPs

5.1 TriMatrix Laboratories SOP GR-16-117, "*Extraction of Soluble Inorganic Analytes from Soil*", latest revision

5.2 TriMatrix Laboratories SOP GR-05-129, "*Konelab Aqua 20 Operation*", latest revision

5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.4 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.5 TriMatrix SOP GR-16-103, Glassware Cleaning and Preparation for the Wet Chemistry and Metals Laboratories, latest revision

5.6 TriMatrix Laboratories SOP GR-10-104, *Chain-of-Custody (COC)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 No significant interferences exist except for turbidity or color from the sample matrix. Filtration using 0.45 µm syringe filters or dilution may be necessary.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all health and safety instructions as outlined in the TriMatrix Laboratories Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Approved safety glasses must be worn in the laboratory. Disposable gloves must be worn when handling reagents, samples and when performing the analysis. Refer to the material safety data sheet (MSDS) for any unfamiliar chemical or reagent. MSDS information is maintained on the intranet library.

7.3 Mercuric thiocyanate may only be handled inside a hood. Mercury is a very toxic metal. Dispose of all mercury waste in the appropriate mercury waste container.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are to be collected in inert plastic bottles and stored at 4 ±2° C until time of analysis. Chemical preservation is not required. Analysis must be within 28 days from the date of collection.

8.2 Do not homogenize samples containing sediment before removing an aliquot unless otherwise specified. Turbidity interferes with the colorimetric measurement. Sediment is not a significant source of chloride ion. However, homogenization and filtration may be necessary to preserve the sample's integrity for other analyses.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Konelab Aqua 20 Spectrophotometer, wavelength of 480 nm

9.2 Standard cups, 2 mL

9.3 Volumetric flasks, class A

9.4 Macropipettes and micropipettes, verified accurate the the dispensing volume

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Refer to TriMatrix SOP GR-05-129 for a preventive maintenance schedule and listing.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM type II, MilliQ system

11.2 Stock Mercuric Thiocyanate Solution. In a 1 L volumetric flask dissolve 4.17 g mercuric thiocyanate ($Hg(SCN)_2$) into about 500 mL methanol. Dilute to the mark with methanol and invert three times. Store at room temperature in a plastic container. Replace after 6 months. Caution: Mercury is a very toxic metal. Wear gloves and handle only in the fume hood.

11.3 Stock Ferric Nitrate Reagent (0.5M): In a 1 L volumetric dissolve 202 g ferric nitrate ($Fe(NO_3)_3 \cdot 9H_2O$) in approximately 800 mL laboratory reagent water. Add 25 mL concentrated nitric acid and dilute to the mark with reagent water. Invert three times to mix. Store at room temperature in a glass amber container. Replace after 6 months.

11.4 Combined Color Reagent. In a 500 mL volumetric flask mix 75 mL stock mercuric thiocyanate solution with 75 mL stock ferric nitrate reagent and dilute to the mark with reagent water. Invert three times to mix. Vacuum filter through a 0.45 micrometer membrane filter. Store at room temperature in a glass amber container. Replace after 1 month.

11.5 When disposing of the reagents containing mercury, pour them into a waste container labeled "mercury waste".

12.0 STANDARDS PREPARATION

12.1 Primary Stock Standard 10,000 mg Cl^-/L

12.1.1 Dissolve 16.49 g sodium chloride (NaCl) (previously dried overnight in an oven at 140° C and stored in a desiccator) in a 1 L volumetric flask partially filled with reagent water. Dilute to the mark with reagent water. This standard is used for preparing the matrix spikes discussed in section 18.1.2.1. 1 mL of this solution = 10 mg of Cl^-. Store in a plastic container at room temperature. Replace after 6 months.

12.2 Secondary Stock Standard 1000 mg Cl^-/L

12.2.1 Dissolve 1.649 g sodium chloride (previously dried overnight in an oven at 140° C and stored in a desiccator) in a 1 L volumetric flask partially filled with reagent water. Dilute to the mark with reagent water. 1 mL of this solution = 1 mg of Cl^-. Store in a plastic container at room temperature. Replace after 6 months.

12.3 Working Standards

Concentration of Standard used, mg/L	Volume of Standard, mL	Final Volume, mL	Final Standard Concentration, mg/L
10,000	5	100	500 (autodilute check)
1000	25	100	250 (autodilute check)
1000	20	200	100
1000	15	200	75
100	40	100	40
100	20	100	20
100	10	100	10
100	5	100	5
10	10	100	1.0

Store in a plastic container at room temperature. Replace working standards after one month.

12.4 All standards must be labeled with the following information:

12.4.1 Initials of preparer

12.4.2 Date of preparation

12.4.3 Concentration with units

12.4.4 Parameter name

12.4.5 Standards logbook number as it appears in the standards logbook and/or the Element™ number

12.4.6 Diluted volume with units

12.5 All standards must be recorded in the appropriate standards logbook and/or in Element™ with a unique ID number. Refer to the standards logbook example in Attachment 23.1.

13.0 SAMPLE PREPARATION

13.1 Filter samples as needed using a 0.45 µm syringe filter to remove turbidity and excess color. Dilute if necessary, to remove color interference.

14.0 CALIBRATION PROCEDURES

14.1 A description of the calibration is included in Section 15.0. It is performed using the seven standards prepared in Section 12.3 plus a reagent water blank. The standard range is 1.0 mg/L to 100 mg/L.

14.2 The tray protocol for the batch will include an initial blank and calibration verification as well as continuing blank and calibration verifications every ten samples. These are sampled from the reagent water blank and standards used for the calibration curve.

Acceptable limits are below:

ICB/CCB less than the reporting limit of 1.0 mg/L
ICV/CCV 85-115%

14.3 Auto dilute verifications are analyzed as part of each batch to verify the diluter is working properly. A 1000 mg/L standard is run at a dilution of 1:20. A 500 mg/L standard is run at a dilution of 1:10. A 250 mg/L standard is run at a dilution of 1:5 and a 100 mg/L standard is run at a dilution of 1:2. Acceptable limits are 90-110%.

15.0 ANALYTICAL PROCEDURE

15.1 Refer to TriMatrix SOP GR-05-129 for detailed instructions on operating the Konelab instrument.

15.2 Fill sample cups with appropriate standards and samples. Place in the Konelab. For color development, the instrument performs the following:

15.2.1 Measures 100 uL chloride color reagent into the cuvette.

15.2.2 Performs a background reading.

15.2.3 Measures 20 uL of sample into the cuvette.

15.2.4 Incubates the sample for 4 minutes.

15.2.5 Measures absorbance at 480 nm.

15.3 Perform standby and shutdown of the Konelab following completion of the analysis batch.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The instrument regresses all measured absorbance data against the calibration and prints out concentration.

16.2 The instrument will automatically perform dilutions as required, calculate the dilution then report the final concentration.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts processing samples are responsible for data quality and for correctly filling in all documentation. This is required for quality control and to provide the client with a fully defensible report.

17.2 The following must be attached to the laboratory benchsheet with each analytical batch completed:

17.2.1 The calibration hardcopy (refer to the calibration hardcopy example in Attachment 23.2)

17.2.2 Pretreatment sheets, if necessary

17.2.3 The sample/quality control report

17.3 The instrument run logbook must be completed for each analytical batch and must include the following information (refer to Attachment 23.3 for an instrument logbook example):

17.3.1 Date analyzed

17.3.2 Analyst's initials

17.3.3 Method reference and number

17.3.4 Calibration standards used

17.3.5 Client name and sample numbers analyzed

17.4 All logbooks must be filled in completely and accurately. Corrections are to be made in indelible blue or black ink. **Corrections are to be made with a single lineout, which is then dated and initialed. Write-overs are not acceptable.** The erroneous result must remain legible. The new result is to be placed near the incorrect result. Blank lines in logbooks must be Z'd out.

17.5 The following data must be recorded on the benchsheet for each batch turned in (refer to Attachment 23.4 for an preparation batch report example and Attachment 23.5 for a data review report example):

17.5.1 ICV, ICB, LCS, MPB, sample concentration, CCV, CCB, SPK, MSD, or DUP

17.5.2 Instrument number, owner, date run, supervisor, stock standard number, wavelength, and cell path

17.5.3 Standard values, observed standard values, working standard numbers

17.5.4 Reagent numbers

17.6 If internal chain-of-custody is required it is very important that the COC for be filled in completely and accurately in adherence with TriMatrix SOP GR-10-104.

18.0 QUALITY ASSURANCE

18.1 Method and matrix QC must be analyzed with each batch.

18.1.1 Method QC for aqueous samples consists of an Instrument Blank (BLK), Laboratory Control Sample (LCS), Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verifications (CCV), Continuing Calibration Blanks (CCB), and a Detection Limit Confirmation Standard (CRL). Solid and waste samples additionally require a pre-treated Method Preparation Blank (MPB) and Laboratory Fortified Blank (LFB).

Requirements for the MPB and LFB are discussed in the pre-treatment SOP, TriMatrix SOP GR-16-117.

18.1.1.1 A BLK and a LCS are analyzed with every daily batch.

18.1.1.1.1 The BLK is an aliquot of reagent water analyzed like a sample. The results must be less that the reporting limit for the analysis.

18.1.1.1.2 The LCS is an aliquot of an Analytical Products Group (APG), or equivalent, purchased standard used to validate the curve. To prepare a second source LCS, follow the instructions supplied with the vial.

18.1.1.1.3 All LCS standard preparations must be recorded in the standard logbook with their own unique ID numbers. All LCS standards must be labeled with the following information:

- initials of preparer
- date of preparation
- concentration
- name of parameter
- log number as it appears in the standard logbook

18.1.1.1.4 The acceptance limits for the LCS are listed in Element™.

18.1.1.2 A MPB and LFB are also analyzed with solid and waste samples. Refer to TriMatrix SOP GR-16-117 for the pre-treatment. The BLK and LCS batching and recording requirements stated above also apply to the MPB and LFB.

18.1.1.3 The ICV is an aliquot of a midrange standard analyzed immediately after the curve. The acceptance limits for the ICV are 85-115% of the calculated value.

18.1.1.4 The ICB is an aliquot of reagent water analyzed immediately after the ICV. The result must be less than the reporting limit for the analysis.

18.1.1.5 The CCVs are aliquots of midrange standards analyzed after every ten samples and at the end of every batch. Acceptance limits for the CCV is 85-115% of the calculated value (Include in the ten-sample count the LCS, BLK, SPK, and DUP).

18.1.1.6 The CCB is an aliquot of laboratory reagent water analyzed after every ten samples (after the CCV) and at the end of each analytical batch. Results must be less than the reporting limit.

18.1.1.7 The CRL is an aliquot of the lowest concentration calibration standard analyzed like a sample. Acceptance limits are 80-120% of the calculated value.

18.1.2 Matrix QC consists of a Matrix Spike (MS) and MSD Matrix Spike Duplicate (MSD)

18.1.2.1 The MS/MSD are prepared by taking 10 mL of sample and spiking with 50 uL of the 10000 mg/L standard. This results in a concentration of 50 mg/L.

18.1.2.2 Acceptance limits for the MS/MSD are the laboratory control limits listed in Element™.

18.1.3 Matrix QC must be performed on prior to the soil, sludge and waste sample extraction described in TriMatrix SOP GR-16-117.

18.2 If any quality control parameter is out of established control limits, corrective action must be initiated that includes the following:

18.2.1 Follow every step in TriMatrix SOP GR-10-106 for correcting the out-of-control event.

18.2.2 Once corrective action has been taken, re-analyze all samples associated with the out-of-control event unless otherwise specified.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. While IDCs are not instrument dependent, one is required on each instrument used in sample analysis to demonstrate the instrument's ability to generate acceptable accuracy and precision. A Continuing Demonstration of Capability (CDC) study is required annually

19.1.1 Initial Demonstration of Capability

19.1.1.1 Spike four samples with the LCS prepared in Section 18.1.1.1.2 so the resulting concentration is in the lower half of the calibration range. Process the four samples following every step outlined in the procedure. Calculate average percent recovery and relative standard deviation using the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must be between the current LCS acceptance limits in Element™. Percent relative standard deviation must be ≤20%.

19.1.1.2 Alternatively, the last four of seven results obtained in an MDL study if run exclusively by the analyst may be used as the IDC. ONLY the last four results may be used.

19.1.1.3 If either IDC criterion is not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure and/or techniques used. If this occurs, locate and correct the procedure and/or techniques used then repeat the study.

19.1.1.4 Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed. Copies of successful IDC spreadsheets and raw data must be given to the Quality Assurance department for training documentation.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A CDC must be performed annually by all analysts running samples.

19.1.2.2 A CDC may be accomplished by repeating the IDC study, by processing four consecutive LCS results obtained during the course of routine sample analysis or by submitting an acceptable PT sample on the appropriate paperwork for inclusion in the analyst's training file.

19.2 Method Detection Limit Studies

19.2.1 A Method Detection Limit (MDL) study must be performed annually on every instrument using this procedure. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. Actual reporting limits are derived from the MDL study. The minimum possible non-estimated reporting limit is equal to the concentration spiked in the MDL study, provided the MDL passes. Any reporting limit actually achieved in any given sample matrix will vary depending on instrument sensitivity, matrix effects and dilutions.

19.2.2 The procedure followed for a MDL study is based on the method given in the Code of Federal Regulations, part 136, Appendix B, latest revision.

19.2.3 Seven replicate analyses are performed using laboratory reagent water spiked at the estimated minimum reportable concentration.

19.2.4 The standard deviation of the concentration found using all seven results is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

19.2.5 If the concentration spiked is between the calculated MDL and ≤5 times the calculated MDL, and there are no zero percent recoveries in the data set, the MDL is acceptable. If not, the MDL study must be repeated. If a study needs repeated at a different concentration, the entire data set needs repeated. If a study does not pass due to poor reproducibility on one result, only that one result needs repeated. However, only one result can be rejected from the data set before repeating the entire study. All seven analyses do not need analyzed in the same analytical batch.

19.2.7 If at any time the reporting limit is above a client's or state's desired reporting limit, the calculated MDL value may be used as a reporting limit, provided results are narrated. Narration must state that the reporting limit is based on the calculated MDL value and that chloride, when spiked at that level, is not observed.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. MSDSs are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Methods for Chemical Analysis of Water and Wastes,* March, 1983, "Chloride (Colorimetric, Automated Ferricyanide AAII)", Method 325.2, Issued 1978

22.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998,* 4500-Cl⁻ Chloride, E, Automated Ferricyanide Method

22.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September, 1986, Method 9251, Chloride (Colorimetric, Automated Ferricyanide AAII)

Note: A Konelab discrete analyzer is used in place of the Technicon autoanalyzer.

23.0 ATTACHMENTS

23.1 Standards Logbook Example

23.2 Calibration Hardcopy Example

23.3 Instrument Logbook Example

23.4 Preparation Batch Report Example

23.5 Data Review Report Example

Attachment 23.1
Standards Logbook Example

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires
1	IN19.34-1	av. CN-	IN19 33.17			1.0 mg/L	5 mL	IWR7917	100mL	0.05 mg/L	LMB	11-21-06	11-21-06
2	IN19.34-2		IN19 33.18			0.20	10			0.02			
3	IN19.34-3		IN19 33.18			0.20	5			0.01			
4	IN19.34-4		IN19.34-1			0.05	10			0.005			
5	IN19.34-5	av. CN	IN19.34-2			0.02 mg/L	10mL	IWR7917	100mL	0.002 mg/L	LMB	11-21-06	11-21-06
6	IN19.34-6	Conductivity LCS	KCl	Fisher 042829	10/15/09	dry chem.	1.4912g	di H_2O	2L	1412 µmhos/cm	BMH	11/20/06	5/21/07
7	IN19.34-7	av. CN LCS	NaCN aldrich	10411528HV	7/7		0.485g	IWR7917	100mL	1000 mg/L	LMB	11-21-06	2-21-07
8	IN19.34-8	Residue	IN.9.28-7		5/17/07	1000ppm	200mL	dilH2O	1000mL	200ppm	MBH	11/28/06	5/17/07
9	IN19.34-9	av. CN- LCS	IN19.34.7			1000 mg/L	10mL	IWR7917	100mL	100 mg/L LCS	LMB	11-22-06	11-22-06
10	IN19.34-10	LCS	IN19.34.9			100	10			10			
11	IN19.34-11		IN19.24.17			10	10			1.0			
12	IN19.34-12		IN19.34.11			1.0	20			0.2			
13	IN19.34-13		IN19.34.11			1.0	5			0.05			
14	IN19.34-14		IN19.34.12			0.2	10			0.02			
15	IN19.34-15		IN.19.34.13			0.2	5			0.01			
16	IN19.34-16		IN19.34.14			0.05	10			0.005			
17	IN19.34-17	av. CN	IN19.34.15			0.02 mg/L	10mL	IWR7917	100mL	0.002 mg/L	LMB	11-22-06	11-22-06
18	IN19.34-18	TOC	IN19.29.17			10 ppm	5mL	di H2O	100mL	0.5ppm	GBH	11-22-06	11-22-06

file: Standard Logbook Auto xls
page: 34 of 100
revision: 2.0

Approved By: _____ 1-26-09
QA Officer

Approved By: Heather L Brady
Area Supervisor

Attachment 23.2
Calibration Hardcopy Example

Calibration results AquaKem 6.5 Page: 1

Laboratory Instrument no. 298
Analyzer User

30.11.2006 08:10

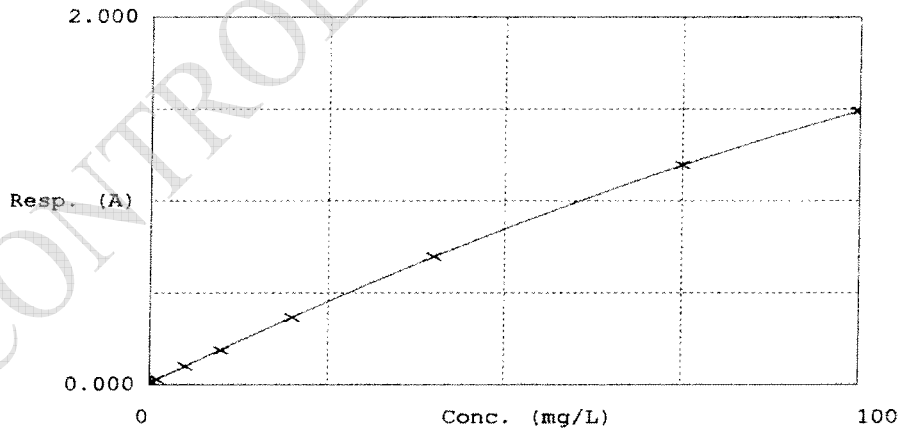
Test Chloride

Accepted 30.11.2006 08:06

Resp. = A * Conc. ^ 2 + B * Conc. + C
A = 0
B = 0.019
C = 0.009

Coeff. of det. 0.999971

Errors



	Calibrator	Response	Calc. con.	Conc.	Errors
1	Cl-0	0.013	0.168	0.000	
2	Cl-1	0.031	1.142	1.000	
3	Cl-5	0.101	4.967	5.000	
4	Cl-10	0.187	9.676	10.000	
5	Cl-20	0.366	19.901	20.000	
6	Cl-40	0.697	40.075	40.000	
7	Cl-75	1.196	75.214	75.000	
8	Cl-100	1.490	99.853	100.000	

Attachment 23.3
Instrument Logbook Example

TriMatrix Laboratories, Inc.

Instrument Number 298 Run Logbook

Date	Analyst	Method Name or Number	Concentration of Calibration Standards Used							Units	Client Names and Sample Numbers
			No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7		
10/5/06	VAS	Cl	0/1	5	10	20	40	75	100	mg/L	(Reid county) Barr Smeltzer city of Lowell
			Calibration Requirements		≥0.995		Calibration Results		0.999907		N Jackson Avard St Mary Cement
10/5/06	VAS	SO4	0/5	10	15	20	25	30	35	mg/L	Cole Terra Lincoln St Mary Cement Avard
			Calibration Requirements		≥0.995		Calibration Results		0.998977		
10/18/06	VAS	SO4 low	0	1	2	5	10	15		mg/L	N Jackson
			Calibration Requirements		≥0.995		Calibration Results		0.999[illegible]		
10/24/06	VAS	Cl	0/1	5	10	20	40	75	100	mg/L	06102222 06102224 06102228 06102255, 267, 270, 306, 320, 321
			Calibration Requirements		≥0.995		Calibration Results		0.999929		
10/26/06	VAS	SO4	0/5	10	15	20	25	30	35	mg/L	06102222, ~~06~~320, 321
			Calibration Requirements		≥0.995		Calibration Results		0.999109		
10/26/06	VAS	SO4 low	0	1	2	5	10	15		mg/L	
			Calibration Requirements		≥0.995		Calibration Results		0.999021		
10/27/06	VAS	phenol h	0	.02	~~.05~~ .10	.5	1	2		mg/L	06102796606
			Calibration Requirements		≥0.995		Calibration Results		0.99976		

Approved By: [signature] 1-26-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.4
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0614129 Page 1 of 1

Printed: 12/4/2006 4:44:08PM

Inorganic - Wet Chemistry, Waste Water, General Inorganic Prep

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0611390	Chloride 325.2	0611419	Chloride 325.2	0611434	Chloride 325.2
0611436	Chloride 325.2	0611467	Chloride 325.2		

Lab Number	*Contain*	*Prepared*	*By*	*Initial (mL)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0614129-BLK1		Nov-30-06 07:44	VAS	10	10					BLANK	
0614129-BS1		Nov-30-06 07:44	VAS	10	10			A607228		LCS	
0614129-MS1		Nov-30-06 07:44	VAS	10	10		0611419-01	A607496	50	MATRIX SPIKE	
0614129-MSD1		Nov-30-06 07:44	VAS	10	10		0611419-01	A607496	50	MATRIX SPIKE DUP	
0611390-01	B	Nov-30-06 07:44	VAS	10	10					[redacted]	
0611419-01	A	Nov-30-06 07:44	VAS	10	10					[redacted]	
0611419-02	A	Nov-30-06 07:44	VAS	10	10					[redacted]	
0611434-01	A	Nov-30-06 07:44	VAS	10	10					[redacted]	
0611436-01	A	Nov-30-06 07:44	VAS	10	10					[redacted]	
0611467-01	A	Nov-30-06 07:44	VAS	10	10					[redacted]	

Comments	Analyst Initials

bch_TriMatrix.rpt

Approved By: QA 1-26-09 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

Attachment 23.5
Data Review Report Example

TriMatrix Laboratories, Inc

Data Review Report -- Wet Chem Lab
Sequence = 6120131 Page 1 of 1

on 12/4/2006 at 4:47

SampleID	Analysis	IResult	Dila	FResult	FMRL	Qualifier	Recovery	RPD	Analyzed	Analyst	Batch
6120131-CAL1	Chloride 325.2	0.168	1	0.168					11/30/2006 8:05	VAS	6120131
6120131-CAL2	Chloride 325.2	1.142	1	1.14			114		11/30/2006 8:05	VAS	6120131
6120131-CAL3	Chloride 325.2	4.967	1	4.97			99		11/30/2006 8:05	VAS	6120131
6120131-CAL4	Chloride 325.2	9.676	1	9.68			97		11/30/2006 8:05	VAS	6120131
6120131-CAL5	Chloride 325.2	19.901	1	19.9			100		11/30/2006 8:06	VAS	6120131
6120131-CAL6	Chloride 325.2	40.075	1	40.1			100		11/30/2006 8:06	VAS	6120131
6120131-CAL7	Chloride 325.2	75.214	1	75.2			100		11/30/2006 8:06	VAS	6120131
6120131-CAL8	Chloride 325.2	99.853	1	99.9			100		11/30/2006 8:06	VAS	6120131
6120131-ICV1	Chloride 325.2	39.627	1	39.6			99		11/30/2006 8:23	VAS	6120131
6120131-ICB1	Chloride 325.2	0.37	1	0.370					11/30/2006 8:23	VAS	6120131
0614129-BLK1	Chloride 325.2	0.373	1	0.373	1.0				11/30/2006 8:23	VAS	0614129
0614129-BS1	Chloride 325.2	51.59	1	51.6			102		11/30/2006 8:23	VAS	0614129
6120131-SCV1	Chloride 325.2	51.59	1	51.6			102		11/30/2006 8:23	VAS	6120131
6120131-CCV1	Chloride 325.2	39.538	1	39.5			99		11/30/2006 8:24	VAS	6120131
6120131-CCB1	Chloride 325.2	0.322	1	0.322					11/30/2006 8:24	VAS	6120131
6120131-CRL1	Chloride 325.2	0.743	1	0.743			74		11/30/2006 8:44	VAS	6120131
6120131-CCV2	Chloride 325.2	39.938	1	39.9			100		11/30/2006 8:47	VAS	6120131
6120131-CCB2	Chloride 325.2	0.283	1	0.283					11/30/2006 8:47	VAS	6120131
0611390-01	Chloride 325.2	69.323	1	69	1.0				11/30/2006 2:17	VAS	0614129
0611434-01	Chloride 325.2	78.535	1	79	1.0				11/30/2006 2:17	VAS	0614129
0611467-01	Chloride 325.2	77.543	1	78	1.0				11/30/2006 2:17	VAS	0614129
6120131-CCV3	Chloride 325.2	43.09	1	43.1			108		11/30/2006 2:17	VAS	6120131
6120131-CCB3	Chloride 325.2	0.61	1	0.610					11/30/2006 2:17	VAS	6120131
0611419-01	Chloride 325.2	1,258.521	20	1300	20	MS006			11/30/2006 2:31	VAS	0614129
0614129-MS1	Chloride 325.2	1,307.389	20	1310	20		98		11/30/2006 2:31	VAS	0614129
0614129-MSD1	Chloride 325.2	1,337.521	20	1340	20		168-CH	2	11/30/2006 2:31	VAS	0614129
0611419-02	Chloride 325.2	1,268.438	20	1300	20				11/30/2006 2:37	VAS	0614129
0611436-01	Chloride 325.2	197.067	2	200	2.0				11/30/2006 2:37	VAS	0614129
6120131-CCV4	Chloride 325.2	41.333	1	41.3			103		11/30/2006 2:46	VAS	6120131
6120131-CCB4	Chloride 325.2	0.124	1	0.124					11/30/2006 2:48	VAS	6120131

rev_TM_WetChem.rpt

Approved By: _____ 1-26-08 **QA Officer**

Approved By: Heather L Brady **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Ferrous Iron

Standard Methods 3500-Fe B

APPROVALS:

Area Supervisor: Heather L. Brady Date: 1-31-09
Heather L. Brady

QA Officer: Date: 1-26-09
Tom C. Boocher

Operations Manager: Date: 2/4/09
Jeff P. Glaser

Procedure Number: GR-05-113
Revision Number: 2.2

Date Initiated: 11/28/95
Effective Date: 2/24/09

Date Revised: 1/24/09
Pages Revised: All

By: Jodi L. Blouw
Total Number of Pages: 14

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-19-10		4-19-11

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to water and wastewater samples.

1.2 The nominal reporting limit is 0.02 mg/L

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Standard Methods for the Examination of Water and Wastewater,* 20th Edition, 1998, Part 3500-Fe B, ", Iron, Phenanthroline Method"

3.0 SUMMARY OF PROCEDURE

3.1 Dissolved ferrous iron reacts with phenanthroline to form an orange-colored complex.

3.2 The intensity of color is linearly proportional to the concentration of ferrous iron.

3.3 Absorbances are read on a UV/VIS spectrophotometer at 510 nm.

4.0 PARAMETER OR COMPOUND LIST

4.1 Ferrous Iron

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interfering substances include strong oxidizing agents, cyanide, nitrite, and phosphates (polyphosphates more so than orthophosphate), chromium, zinc in concentrations exceeding 10 times that of iron, cobalt and copper in excess of 5 mg/L, and nickel in excess of 2 mg/L.

6.2 Bismuth, cadmium, mercury, molybdate, and silver precipitate phenanthroline.

6.3 In the presence of interfering metal ions, use a larger excess of phenanthroline to replace that complexed by the interfering metals. This indication of interfering ions must be noted.

6.2 Colored and turbid samples are a positive interference because they absorb light. Eliminate this interference by zeroing the spectrophotometer on a sample that has been prepared without the color reagent.

7.0 SAFETY PRECAUTIONS

7.1 Analyst must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Preserve the sample by adding 2 mL of concentrated HCl per 100 mL sample to each sample container. Fill the sample bottle directly from the sampling source and immediately stopper.

8.2 Store samples at 4° C until analysis. Samples must be analyzed as soon as possible.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Shimadzu UV160U-UV/VIS spectrophotometer - to be operated using the following conditions:

wavelength: 510 nm
cell length: 10 mm

9.2 10 mm glass cuvet

9.3 plastic disposable sample cups

9.4 volumetric flasks

9.5 volumetric pipets

9.6 10 mL oxford pipet and tips

9.7 100-1000 uL adjustable Eppendorf pipette with tips

9.8 Mettler analytical balance (readable to 0.1 mg)

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Phenanthroline solution
0.5 g phenanthroline: 500 mL reagent water

- add 10 drops concentrated HCl to aid dissolution
- this reagent must be made fresh daily
- 20 mL are required per sample, vary the amount prepared based on number of samples

11.2 Ammonium acetate buffer - dissolve 250 g ammonium acetate in 150 mL reagent water and add 700 mL glacial acetic acid. (See Attachment 24.4 for an example Reagent Logbook page.)

11.3 Reagent water, ASTM Type II, Milli-Q

11.4 Ferrous ammonium sulfate - Fe $(NH_4)_2(SO_4)_2$ - $6H_2O$

11.5 Concentrated sulfuric acid - H_2SO_4

11.6 Concentrated hydrochloric acid - HCl

11.7 Potassium permanganate, $KMnO_4$

11.8 Potassium permanganate solution, 0.1N. Dissolve 0.316 g $KMnO_4$ in reagent water and dilute to 100 mL in a volumetric flask.

12.0 STANDARDS PREPARATION

12.1 Primary stock - 400 mg/L

12.1.1 In a 500 mL volumetric flask, combine 10 mL of concentrated sulfuric acid with 50 mL reagent water.

12.1.2 Accurately weigh 1.404 g of ferrous ammonium sulfate and add to flask. Swirl to dissolve, add 0.1N potassium permanganate drop wise until a faint pink color persists.

12.1.3 Dilute to 500 mL with reagent water.

12.2 Secondary Stock - 2.0 mg/L

12.2.1 Dilute 1.0 mL of primary stock to 200 mL with reagent water.

12.3 Working Standards (Refer to Attachment 24.3 for an example Standards Log example):

Concentration mg/L	**Dilution (Diluted with reagent water)**
0.50	25 mL of 2.0 mg/L per 100 mL
0.20	10 mL of 2.0 mg/L per 100 mL
0.10	5.0 mL of 2.0 mg/L per 100 mL
0.04	20 mL of 0.2 mg/L per 100 mL
0.02	20 mL of 0.1 mg/L per 100 mL

12.4 All standards must be labeled with the following information:

- initials of preparer
- date of preparation
- concentration

- name of parameter
- standard log number as it appears in the standard logbook (Refer to Attachment 24.3 for an example Standards log)
- amount of each compound contained and diluted volume

13.0 SAMPLE PREPARATION

13.1 Pour 50 mL of sample into sample cup. If samples were not acidified upon collection, add 1.0 mL of concentrated hydrochloric acid to sample cup. Add 20 mL phenanthroline solution and 10 mL ammonium acetate buffer. Add 20 mL reagent water and mix.

13.2 Solutions containing ferrous iron should form an orange colored complex rapidly. Absorbances must be measured within 5-10 minutes.

14.0 CALIBRATION PROCEDURES

14.1 Set the UV/VIS spectrophotometer to read absorbance at 510 nm.

14.2 Set zero using 0 mg/L standard in a 10 mm cuvet.

14.3 Run all standards in increasing order, and obtain a calibration curve based on the best-fit straight line through the standard points.

15.0 ANALYTICAL PROCEDURE

15.1 Measure absorbances for each sample and read concentration directly from instrument.

15.2 Samples that give a higher absorbance than the highest standard must be diluted and carried through the color development procedure once again.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The spectrophotometer regresses all sample data against the calibration curves and prints out concentration. No manual calculations are done by the analyst except accounting for any dilutions.

16.2 Basic rules apply for significant figure reporting: report 3 figures over 100 mg/L, report 2 figures below 100 mg/L, and report only 1 place to the right of the decimal for <10 mg/L.

17.0 DATA REPORTING AND DELIVERABLES

17.1 The following must be attached to the laboratory benchsheet for each batch turned in:

- A listing of standard concentrations, standard log numbers, and absorbances
- A copy of the calibration curve(s)

- A copy of the spectrophotometer raw data printout(s) (See Attachment 24.2 for an example raw data printout.)

17.2 The instrument logbook must be filled out for each batch turned in, with the following information: (See Attachment 24.5 for an example Instrument Logbook page.)

- date analyzed
- analysts initials
- method name and number
- calibration standards used
- client name and sample numbers analyzed

17.3 Refer to Attachment 24.1 for an example Preparation Batch Report.

18.0 QUALITY ASSURANCE

18.1 Method and Matrix QC must be analyzed with each batch.

18.1.1 Method QC consists of a Blank (BLK), Laboratory Control Sample (LCS), Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), and Continuing Calibration Blanks (CCBs). A Detection Limit Standard (CRDL) is also analyzed with each run.

18.1.1.1 The BLK is an aliquot of reagent water that is analyzed as a sample. The result must be less than the reporting limit.

18.1.1.2 The LCS is an aliquot of a stock standard prepared from a ferrous ammonium sulfate source separate than that used in the quantitation standard. See 12.1 and 12.2 to prepare this standard. Prepare the working concentration, 0.2 mg/L as in section 12.3. Acceptance limits are found on the laboratory benchsheet.

18.1.1.3 The ICV is an aliquot of a midrange standard that is analyzed immediately after the standard curve. The acceptance level is 90-110% of the true value.

18.1.1.4 The ICB is an aliquot of reagent water analyzed after the ICV. The result must be less than the reporting limit.

18.1.1.5 CCV standards are aliquots of the midrange standard analyzed after every ten samples and at the end of a batch. Acceptance levels are 90-110% of the true value.

18.1.1.6 The CCBs are aliquots of reagent water analyzed after the CCV standards. The result must be less than the reporting limit.

18.1.1.7 The CRDL is an aliquot of the 0.02 mg/L standard used for the curve and analyzed as a sample.

18.1.2 Matrix QC consists of a spike (SPK) and a sample duplicate (DUP).

18.1.2.1 The SPK is a sample aliquot spiked with a known quantity, normally 0.20 mg/L, of a primary stock standard. Acceptance limits are on the laboratory benchsheet.

18.1.2.2 The DUP is a second aliquot of sample analyzed in the same way as the sample. Acceptance limits for the % difference are listed on the benchsheet.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before the analysis of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one-time analyst certification. While analyst certification is not instrument dependent, this certification is required on every instrument that will be running samples to demonstrate instrument ability to generate acceptable accuracy and precision.

19.2 Prepare a quality control check sample spiking standard at a level that will give concentrations at 0.2 mg/L.

19.3 Analyze four check samples following the SOP.

19.4 Calculate average recovery (x) in mg/L, and standard deviation (s) of the average in mg/L, for ferrous iron using the four results.

19.4.1 For each analyte (x) must be in the range 70-130% and (s) must be less than or equal to 20. If (s) and (x) meet the acceptance criteria, analyst certification is good. The analyst and system are authorized to run samples once acceptance is obtained.

19.4.2 When a demonstration of capability fails at least one acceptance criteria, the analyst must proceed according to 19.5.

19.5 Locate and correct the source of the problem and repeat the test. Repeated failure will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test beginning with section 19.1. Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability has been successfully completed. Copies of successful demonstration spreadsheets and raw data must be given to the Quality Assurance department.

20.0 Method Detection Limit Studies

20.1 A Method Detection Limit (MDL) study must be performed annually. MDL studies must be performed on each instrument used for ferrous iron quantitation.

20.1.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero.

20.1.2 The quantitation limit is derived from the MDL study and is equal to the amount spiked for the MDL study, provided the MDL passes.

20.1.3 Quantitation limits actually achieved in any given analysis will vary depending on instrument sensitivity, matrix effects, and dilutions.

20.2 The procedure followed for MDL studies is based on the method given in the Code of Federal Regulations, Part 136, Appendix B, and latest revision. All quality control procedures during the study must be followed.

20.3 Seven replicate analyses are performed using reagent water spiked with ferrous iron, at the estimated minimum reportable limit. The last four replicates can also be used for an analyst demonstration of capability, if run exclusively by the analyst.

20.4 A blank measurement is required to calculate the measured level of an analyte and seven separate blanks must be run, one after each of the seven MDL analyses. The average blank measurement is subtracted from all seven MDL runs.

20.5 The standard deviation of the average (blank-subtracted) of the seven runs is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

20.6 If the amount spiked is ≥ the calculated MDL and ≤ 5 times the calculated MDL and there are no 0 percent recoveries, the MDL result is acceptable. If not, the MDL must be re-run. If the study needs re-run at a different concentration, the entire set of seven needs re-run. If the study does not pass due to poor reproducibility on one of the replicates, only that replicate needs re-analyzed. Only one replicate can be rejected however. Not all seven replicates need run during the same shift.

20.7 If the reporting limit is above a client's or state's desired reporting limit, the MDL value may be used for as a quantitation limit, if the report is narrated. State that the reporting limit is a calculated method detection limit and when ferrous iron is spiked at that level, it is not observed.

21.0 POLLUTION PREVENTION

21.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

21.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

21.3 Conserve use of chemicals where applicable.

21.4 Comply with all environmental laws associated with chemicals in the laboratory.

22.0 WASTE MANAGEMENT

22.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

22.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

22.3 Follow all instructions in TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, for laboratory waste disposal requirements.

23.0 REFERENCES

23.1 *Standard Methods for the Examination of Water and Wastewater,* 20th Edition, 1998, Part 3500-Fe B, "Iron, Phenanthroline Method"

24.0 ATTACHMENTS

24.1 Preparation Batch Report Example

24.2 Raw Data Example

24.3 Standards Log Example

24.4 Reagent Logbook Example

24.5 Instrument Logbook Example

Attachment 24.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0814760 Page 1 of 1

Printed: 1/26/2009 4:41:01PM

Inorganic - Wet Chemistry, Water, Method-Specific Preparation

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0812358	Fe, Ferrous 3500-Fe B				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (mL)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client QC Type*	*Extraction Comments*
0814760-BLK1		Dec-18-08 09:00	CLD	25	25					BLANK	
0814760-BS1		Dec-18-08 09:00	CLD	25	25			8120219	20	LCS	
0814760-MS1		Dec-18-08 09:00	CLD	25	25		0812358-03	8120219	2000	MATRIX SPIKE	
0814760-MSD1		Dec-18-08 09:00	CLD	25	25		0812358-03	8120219	2000	MATRIX SPIKE DUP	
0812358-01	E	Dec-18-08 09:00	CLD	25	25					[redacted]	
0812358-02	E	Dec-18-08 09:00	CLD	25	25					[redacted]	
0812358-03	E	Dec-18-08 09:00	CLD	25	25					[redacted]	

Approved By: _____ 1-26-9 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

Attachment 24.2
Raw Data Example

Fe2+ std
1.5 L3

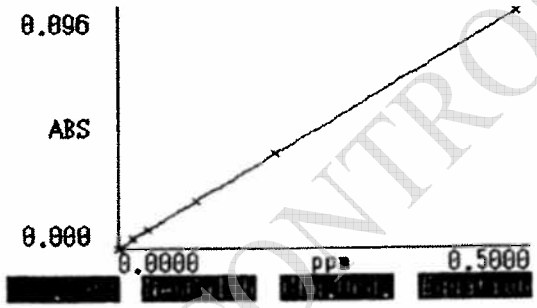
·08/Jan/03 06:40:40

510.0nm 0.096A

No.	Conc.	ABS	No.	ABS
1	0.0000	0.000		
2	0.0200	0.004		
3	0.0400	0.007		
4	0.1000	0.019		
5	0.2000	0.039		
6	0.5000	0.096		

08/Jan/03 06:41:00

510.0nm -0.038A



08/Jan/03 06:41:14

ABS = K3C³+ K2C²+ K1C+ K0

K3 = 0.0000
K2 = 0.0000
K1 = 0.1925
K0 = -0.0000
r² = 1.0000

No.	ABS	ppm
1	0.039	0.2049 ICV
2	0.001	0.0052 ICB
3	0.001	0.0058 BLK
4	0.039	0.2043 LCS
5	0.005	0.0274 CRDL (mdl)
6	0.005	~~0.0242 mdl2~~
7	0.005	~~0.0256 mdl3~~
8	0.005	~~0.0261 mdl4~~
9	0.005	~~0.0255 mdl5~~
10	0.005	0.0242 mdl6
11	0.005	~~0.0236 mdl7~~
12	0.014	0.0743 323372
13	0.040	0.2081 ccv1
14	0.001	0.0065 ccb1
15	0.014	0.0743 323372 dup
16	0.046	0.2405 323372 spk
17	0.040	0.2081 ccv2
18	0.001	0.0065 ccb2

Approved By: 1-26-09 **QA Officer**

Approved By: Heather L Brady **Area Supervisor**

Attachment 24.3
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
8120219

Description:	Fe+2 400mg/L	Expires:	Jun-04-09
Standard Type:	Analyte Spike	Prepared:	Dec-04-08
Solvent:	Solvent Lot #	Prepared By:	Heather L. Brady
Final Volume (mls):	500	Department:	Inorganic - Wet Chemistry
Vials:	1	Last Edit:	Dec-04-08 14:13 by HLB

1.404g Ferrous Ammonium Sulfate 6H2O + 10mls H2SO4into 500mls DI

Analyte	CAS Number	Concentration	Units
Iron, Ferrous	15438-31-0	400	mg/L
Ferrous Iron		400	mg/L

Approved By: [signature] 1-26-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 24.4
Reagent Logbook Example

TriMatrix Laboratories, Inc.

Reagent Preparation Logbook

Reagent Name: Ammonium Oxalate Buffer **Stock Reagent Number:** *INR2.55*

Prep Instructions: 260grams Ammonium Oxalate, 150mls diNater, 700mls Acetic Acid (glacial)

Concentrations:

Working Reagent Number	Date Made	Expiration Date	Made By	Reagent(s)/Lot Number(s) Used	Solvent(s)/Lot Number(s) Used
INR2.55-1	5-9-01	5-9-03	NLB	Ammonium Oxalate 402716108	Acetic Acid 619110
INR2.55-2	8-8-02	8-8-03	NLB	Amm. Acetate ↓	↓
INR2.55-3	10-1-02	10-1-03	NLB	017044	
INR2.55-4	12-3-02	12-3-03	NLB	Ammonium Oxalate 017044	Acetic Acid 420104
INR2.55-5					
INR2.55-6					
INR2.55-7					
INR2.55-8					
INR2.55-9					
INR2.55-10					
INR2.55-11					
INR2.55-12					
INR2.55-13					
INR2.55-14					
INR2.55-15					
INR2.55-16					
INR2.55-17					
INR2.55-18					

Approved By: [signature] 1-26-09 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

Attachment 24.5
Instrument Logbook Example

TriMatrix Laboratories, Inc.

Instrument Number 120 Run Logbook

Date	Analyst	Method Name or Number	Concentration of Calibration Standards Used							Units	Client Names and Sample Numbers
			No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7		
10.31.2	SMJ	COD	0	5	10	20	30	40			[redacted] 318831-2, 319 [redacted] 318944-5, 318947-62 [redacted] 319104
			Calibration Requirements	≥0.995			Calibration Results	0.998			
10-31-02	NLB	Cr^{+6}	0	0.001	0.0035	0.005	0.010	0.035		mg/L	[redacted] (1)
			Calibration Requirements	≥0.995			Calibration Results	0.9999			
10-31-02	NLB	Fe^{+2}	0	0.02	0.04	0.1	0.2	0.5		mg/L	[redacted] 319269-319270
			Calibration Requirements	≥0.995			Calibration Results	0.9997			
10.31.2	SMJ	COD	0	50	100	500	1000	2000			[redacted] 318728
			Calibration Requirements	≥0.995			Calibration Results	0.999			
11-2-02	NLB	Cr^{+6}	0	0.001	0.0035	0.005	0.010	0.035		mg/L	[redacted] 319479, 319483, 319484
			Calibration Requirements	≥0.995			Calibration Results	0.9998			
11-2-02	NLB	NO_2	0	0.01	0.05	0.1	0.25	0.5		mg/L	[redacted] (5)
			Calibration Requirements	≥0.995			Calibration Results	0.9998			
11-2-02	NLB	Fe^{+2}	0	0.02	0.04	0.1	0.2	0.5		mg/L	[redacted] 319414 - 319415
			Calibration Requirements	≥0.995			Calibration Results	0.9991			

file: instlogbook page: 4 of 50 [redacted] revision: 1.0

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Dissolved Methane, Ethane and Ethene in Water by Headspace Equilibrium and Gas Chromatography

Newell SOP RSK-175

APPROVALS:

Area Supervisor: Janet M. Kudirka Date: 1/23/09
Janet M. Kudirka

QA Officer: Tom C. Boocher Date: 1-22-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 1/23/09
Jeff P. Glaser

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Total Number of Pages: 21

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
5-5-10		5-5-11

1.0 SCOPE AND APPLICATION

1.1 This analysis is applicable to the determination of dissolved gases in non-saline groundwater by headspace equilibrium, at parts-per-million levels. Measurement of dissolved gases in water is used to monitor active bioremediation and natural attenuation.

1.2 This procedure is restricted to use by or under the supervision of analysts experienced in headspace and gas chromatography analysis. A demonstration of capability study must be performed by all analysts, before processing samples.

2.0 PRINCIPLE METHOD REFERENCES

2.1 Newell, B.S., RSKSOP-175, *Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC headspace Equilibration Technique*, Revision 0, August 11, 1994

2.1.1 A modification to this procedure is that injection volumes used are 200 µL instead of 300 µL.

2.2 USEPA, Region 1, *Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, and Ethene*, Revision 1, 02/21/02

3.0 SUMMARY OF PROCEDURE

3.1 Water samples are preserved and collected in 40 mL VOA vials with PTFE-faced septum-lined caps, without headspace. Headspace is introduced at the laboratory by replacing a specified sample volume with ultra-high purity helium.

3.2 Water and headspace equilibration is performed by shaking each vial, for at least 5 minutes. A measured headspace volume is then removed and injected onto a gas chromatograph column. Target gases are separated on the column and detected by flame ionization (FID), for quantitation against a standard calibration.

3.3 Target gas concentration is calculated using Henry's Law constants (at the sample temperature), the quantitated headspace concentration, vial volume, and the sample temperature. The concentration of gas in the liquid is proportional to the partial pressure of the gas above the liquid.

4.0 PARAMETER OR COMPOUND LIST

Analyte	Linear Range (ug/L)	Reporting Limit (ug/L)	CAS Number	Molecular Weight (g/mole)
Methane	0-50	5.0	74-82-8	16
Ethene	0-50	5.0	74-85-1	28
Ethane	0-50	5.0	74-84-0	30

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

5.2 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.3 TriMatrix SOP GR-03-101, *Semi-Volatiles Laboratory Quality Control Corrective Actions*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through vial septa, during shipment and storage. A field blank must be prepared from analyte-free reagent water and carried through all sampling and handling steps, to verify contamination by diffusion has not occurred.

6.2 Carryover can occur whenever low-level samples are sequentially analyzed after a high-level sample. To minimize carryover, sample syringes must be rinsed between gas chromatograph injections with helium gas. When a highly concentrated sample is analyzed, it must be followed by analysis of a syringe blank.

6.3 Before processing samples, the analyst must demonstrate an interference-free analytical system by analysis of an organic gas-free water blank. The blank analysis must be performed each day samples are analyzed. If interferences are detected at or above the reporting limit, the problem must be resolved before sample analysis begins.

6.4 Methane is a very common contaminant and occurs naturally in the atmosphere. Automobile exhaust also contains high levels of target gases. Care must be exercised to prevent contamination during transport and the field blank used to monitor contamination levels.

6.5 Moisture can interfere with low-level analysis. If a problem, moisture interference can be minimized by injecting through a calcium sulfate moisture trap.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Collect samples in duplicate pre-preserved 40 mL VOA vials.

8.1.1 Use hydrochloric acid (HCl) as the preservative.

8.1.2 Fill each vial by gently pouring down the inside of the glass without agitation, to just before overflowing then cap with the PTFE septum facing inward. Do not let the vial overflow.

8.1.3 Do not fill in a way that air bubbles pass through the sample. If headspace or air bubbles are trapped in the sample after a vial is sealed, repeat the sampling with a fresh vial.

8.2 Store collected samples at 4 ±2° C until analysis. Sample storage must be free of organic vapors.

8.3 Analyze samples within 14 days of collection. Samples not analyzed within this holding time must be narrated and qualified as estimated.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Glassware and Materials

9.1.1 Gas sampling bulbs for working standards, glass, with PTFE stoppers and a septum interface, 250 mL

Note: Verify the gas sampling bulb volume annually. Volume must be within ±1 mL of 250 mL. If not, replace the bulb or use based on the actual volume measured.

9.1.2 Gas-tight syringe, with side-port needle, glass, certified accurate, 500 µL

9.1.3 Screw-cap VOA vials, 40 mL, with PTFE-faced septa

9.1.4 Gas-tight syringe, with side-port needle, glass, certified accurate, 5.0 mL

9.1.5 Gas standards at 10,000 µL/L (ppmv)

9.1.6 Helium, ultra-high purity or equivalent

9.1.7 Mechanical shaker, Burrell wrist-action shaker, model 75

9.1.8 Three-finger clamps

9.1.9 Ring stand

9.2 Instrumentation

9.2.1 Turbochrome data acquisition software, version 4.1, PE Nelson

9.2.2 Varian 3400 gas chromatograph, equipped with an FID detector

9.2.2.1 Injector Temperature: 250° C

9.2.2.2 Detector Temperature: 280° C

9.2.2.3 Column Temperature: 45° C Isothermal

9.2.2.4 Hold Time: 3.0 min

9.2.3 Packed Column: Supelco 50/80 Porapak P, 12' x 1/8"

9.2.4 Injection Volumes: 2.0 µL to 500 µL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Change GC injection port septa as needed.

10.2 Run helium blanks to clean out the column if poor chromatography or interferences appear. If running blanks does not resolve the problem, corrective action is required up to and including column replacement, before sample analysis can begin.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water (gas-free)

11.2 Gas standard cylinders containing methane, ethene and ethane at 1% (10,000 µL/L), purchased commercially and certified to be NIST traceable

12.0 STANDARDS PREPARATION

12.1 Stock Standard

12.1.1 The 10,000 µL/L gas mixture is withdrawn directly from the purchased cylinder as follows:

12.1.1.1 Turn on the gas so a steady stream of bubbles is emerging from cylinder and into a bubbler which is a tubing line connected to a T-connector with the tubing end in a beaker of water. The other end of the T-connector contains a septum port for syringe extraction of the gas.

12.1.1.2 When the gas flow is steady, insert a syringe in the septum port and withdraw the appropriate gas volume for injection into the 250 mL gas sampling bulb.

12.2 Working Standards

12.2.1 Label a 250 mL gas sampling bulb as 200 µL/L and fill it with helium. Quickly rotate the stopcock several times to relieve excess pressure. With a gas-tight syringe, draw out 5.0 mL of helium. Purge the helium in the syringe to the atmosphere. With the same syringe, withdraw 5.0 mL of 10,000 µL/L standard (Section 12.1.1) and inject into the 200 µL/L bulb.

12.2.2 Different volumes of the 200 µL/L standard are injected to develop the calibration based on a 200 µL injection, as follows:

Volume Injected (µL)	Concentration (µL/L)	Concentration (P_{gas})
500	500	0.0005
100	100	0.0001
50	50	0.00005
20	20	0.00002
10	10	0.00001
4.0	4.0	0.000004

13.0 SAMPLE PREPARATION

13.1 Remove samples from the refrigerator and bring to room temperature before continuing.

13.2 To generate sample vial headspace, invert a sample vial in a three-finger clamp attached to a ring stand. Insert a needle attached to a Luer-Lok syringe through the septum. Connect another needle to the two-stage regulator of helium with a length of PTFE tubing. Insert the helium needle into the vial and inject helium at 5 mL/min or less. The helium will force sample into the syringe. Remove approximately 10% of the vial volume. When the appropriate volume is removed, immediately pull all needles from the vial.

Note: Purge the PTFE tubing with helium before injecting a sample and do not turn the gas off until all samples are prepared.

13.3 Shake the sample vial for at least 5 minutes to equilibrate headspace and liquid phases. After shaking, extract 200 µL of headspace with a 500 µL gas-tight syringe. If an immediate injection can not be made, vials must be kept inverted until the headspace can be analyzed.

13.4 Insert the syringe needle into the septum far enough so the needle port is fully in the headspace before extracting. As soon as the headspace is extracted, inject into a gas chromatograph for analysis.

14.0 CALIBRATION PROCEDURES

14.1 Analyze a helium blank before calibrating the GC to determine if the system is clean. If contamination is observed, correct the problem before calibrating.

14.2 Construct an initial six-point calibration curve by injecting each standard volume (Section 12.2.2). Inject sequentially from the lowest to highest concentration. The lowest standard must have a signal to noise ration greater than 5. Plot peak area against concentration as a decimal fraction relating to partial pressure (For example: 10 µL/L is 0.00001 as a decimal fraction). A linear regression coefficient of 0.995 or higher

must be achieved. Use only linear regression to calibrate with. Refer to Attachment 21.3 for a methane calibration example.

14.2 Continuing Calibration

After initial calibration, analyze, calibration verification standards (ICV/CCV). An ICV/CCV is a 100 µL/L standard (0.0001 as a decimal fraction), which must be analyzed initially then at a frequency of once every 10 samples. ICV/CCV recovery must fall within 85% to 115%. If a target gas falls outside these acceptance limits, repeat the CCV analysis. If recovery is still unacceptable, correct the problem and rerun all samples analyzed since the last acceptable CCV.

15.0 ANALYTICAL PROCEDURE

15.1 After GC calibration, prepare samples by introducing headspace. After preparing, analyze immediately or keep vials inverted until the injection can be made. Use a 500 µL gas-tight syringe to inject 200 µL of headspace, as described in Section 13.0.

15.2 If a peak response exceeds the calibration range, inject a lesser volume of headspace. If less than 2 µL is required to put response within the calibration range, prepare a headspace dilution by extracting headspace from the sample and injecting into a gas sampling tube filled with helium.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Dissolved gas calculations involve several steps, as follows:

16.1.1 From sample analysis, an area count is obtained. Using area count and the standard curve, determine sample vial partial gas pressure.

NOTE: To develop the calibration regression, plot area count on the x-axis against the gas standard decimal fraction concentration on the y-axis (For example: 10 µL/L is 0.00001 on the curve). The decimal fraction concentrations are unitless.

NOTE: In these calculations total pressure is assumed equal to one atmosphere, as follows:

$$\frac{P_{gas}}{P_{total}} = P_{gas}$$

The linear regression conforms to partial pressure based on the assumption, as follows:

$$P_{gas} = m(\text{area count}) + b$$

Where
p_{gas} = Partial pressure of the target gas (decimal fraction concentration)
m = Slope of the calibration line

b = Y-intercept of the calibration line

16.1.2 Calculate target gas concentration in the sample vial headspace phase, as follows:

$$\text{Concentration in headspace (mg/L)} = \frac{\text{MW g} \mid \text{mole} \mid 273\text{ K} \mid 1000\text{ mg} \mid mL_{headspace} \mid P_{gas}}{\text{Mole} \mid 22.4\text{ L} \mid T\text{ K} \mid \text{g} \mid mL_{water} \mid}$$

Where:
MW = Molecular weight of the target gas
T = Sample temperature, in Kelvins
$mL_{headspace}$ = Sample vial headspace volume, in mL
mL_{water} = Sample vial water volume, in mL (calculate by subtracting headspace volume from total vial volume)

16.1.3 Calculate target gas concentration in the sample vial liquid phase, as follows:

$$\text{Concentration in Water Phase (mg/L)} = \frac{P_{gas} \mid 55.5\text{ mole} \mid \text{MW g} \mid 1000\text{ mg}}{H_{gas\ @\ T} \mid \text{L} \mid \text{mole} \mid \text{g}}$$

Where:
Hgas @ T = Henry's law constant for target gas at the sample temperature

16.1.4 Calculate total target gas concentration, as follows:

TC = Concentration in headspace phase (mg/L) + Concentration in water phase (mg/L)

Where:
TC = Concentration of dissolved target gas in the sample, in mg/L

16.2 Example Calculation

16.2.1 Methane is used in the following example calculation. Parameters for the example are as follows:

Headspace Area Count =	978264
Method Blank Area Count =	2766
Henry's Law Constant =	4.13E+04 @ 25° C
Sample Temperature =	25° C (298.15 K)
Vial Volume =	60 mL
Headspace Volume =	6.0 mL
Molecular Weight =	16 g/mole

16.2.2 Let the calibration curve be as follows:

P_{gas} = 1.814E-09(area count) – 6.716E-06

P_{gas} = 1.814E-09(978264 - 2766) – 6.716E-06

P_{gas} = 0.0018

16.2.3 Calculate target gas concentration in the sample vial headspace phase, as follows:

$$\text{Headspace Concentration (mg/L)} = \frac{16\text{ g}}{\text{mole}} \left| \frac{\text{mole}}{22.4\text{ L}} \right| \frac{273\text{ K}}{298\text{ K}} \left| \frac{1000\text{ mg}}{\text{g}} \right| \frac{6\text{ mL}}{(60\text{-}6)\text{ mL}} \left| 0.0018 \right.$$

Headspace Concentration (mg/L) = 0.131 mg/L

16.2.4 Calculate target gas concentration in the sample vial liquid phase, as follows:

$$\text{Concentration in Water Phase (mg/L)} = \frac{0.0018}{4.13\text{E+}04} \left| \frac{55.5\text{ mole}}{\text{L}} \right| \frac{16\text{ g}}{\text{mole}} \left| \frac{1000\text{ mg}}{\text{g}} \right.$$

Concentration in Water Phase (mg/L) = 0.039 mg/L

16.2.5 Calculate total target gas concentration, as follows:

TC = Concentration in headspace phase (mg/L) + Concentration in water phase (mg/L)

TC = (0.131 + 0.39) mg/L = 0.170 mg/L methane

16.3 A spreadsheet is available on the library drive of the laboratory intranet which performs all relevant calculations upon entering the area count. Refer to Attachment 21.1 for an example spreadsheet calculation.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analyst running samples are responsible for data quality and for filling in documentation correctly. It is important to document analysis by correctly filling in, handing in and filing paperwork. This is required for quality control and to provide clients with defensible data.

17.2 LIMS Reporting

17.2.1 When an analyst finishes running a sample batch, data must be input to LIMS (Element™). Data must be entered completely to ensure that results are reported correctly and data is associated with

the right quality control batch. Dilution factors need added, so reporting limits are raised accordingly.

17.2.2 A Method Preparation Blank (BLK) must be run for each quality control batch, for each 24-hour shift. It is important to remember that a Blank Spike (BS) cannot be entered without also entering the associated BLK.

17.2.3 If internal chain-of-custody (CoC) is required, it is important that the CoC form be filled in and archived correctly.

17.2.4 All data hardcopy (including CoC forms) must be archived appropriately.

17.3 Laboratory Required Paperwork

17.3.1 All run, maintenance and standards logbooks (if used) must be filled in completely and accurately. Corrections must be made with a lineout, not a writeover. Blank lines in run logbooks must be Z'd out, dated and initialed. Refer to Attachment 21.7 for an instrument run logbook example.

17.3.2 All initial calibration, ICV and CCV runs must be archived in the correct binder or hardcopy box.

17.3.3 All hardcopy documentation and raw data must be archived. Give these data to Data Management who must record the date, time and contents handed in. Sample and quality control benchsheets are returned to the proper folder after data review and approval.

17.4 Rounding and significant figures is to be performed only on final quantitated results by Element™.

18.0 QUALITY ASSURANCE

18.1 Continuing Calibration Verifications (CCV)

18.1.1 A CCV consists of a 100 µL/L standard, which must be analyzed at a frequency of one per 10 samples. CCV recoveries must fall within 85% to 115% for acceptance. If a CCV analyte recovery is outside the acceptance range, analyze another CCV to confirm. If results are still unacceptable, locate and correct the problem before processing further samples. All samples processed since the last acceptable CCV must be re-analyzed or narrated as estimated.

18.2 Method Preparation Blanks (BLK)

18.2.1 For each 24-hour analysis period, analyze a method preparation blank (BLK) to monitor for background contamination. The BLK is a 40 mL vial, filled with laboratory reagent water, prepared and analyzed as a sample. All target gas levels in the BLK must be less than the reporting limit. If contamination is found, locate the problem and correct before further samples are processed. All samples with concentrations above the reporting limit and processed since the last acceptable BLK, need re-analyzed or narrated as estimated.

18.3 Duplicates (DUP)

18.3.1 For every twenty samples at least one sample duplicate must be analyzed. Relative percent difference (RPD) must be less than 20%. If RPD is greater than 20%, repeat the sample analysis to confirm. If RPD remains greater than 20%, locate and correct any system problem before further samples are analyzed. If the problem is not matrix specific, narrate samples associated with the unacceptable DUP as estimated.

18.4 Verify each new calibration with a second-source calibration verification standard (SCV). Compare SCV recovery to LIMS control limits for acceptance. The SCV must be acceptable to begin sample analysis. If not acceptable, locate and correct the problem then repeat the SCV successfully. Prepare new standards and re-calibrate when no instrument malfunction is indicated.

18.5 Take corrective action for quality control problems in accordance with TriMatrix SOP GR-03-101.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATIONS

19.1 Before analysis of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a demonstration of capability study. While demonstrations of capability are not instrument-dependent, one is required on every instrument running samples, to demonstrate the instrument's ability to generate acceptable accuracy and precision.

19.2 Prepare four gas-free SCV spikes in reagent water for the demonstration of capability study, as follows:

19.2.1 Create headspace in the sample vial, as outlined in Section 13.2. Keeping the vial inverted, inject an aliquot (100 µL) of the highest standard or secondary stock cylinder containing methane, ethane and ethene. Continue the preparation including every step in the process and analyze exactly like a field sample.

19.2.2 Calculate the SCV concentration for each gas as follows:

$$\text{Spike amount (ug)} = \text{Gas Density (ug/µL)} * \text{Gas Volume (µL)}$$

$$\text{Spike amount (ug)} = \frac{\text{MW g} \mid \text{mole} \mid 273\ \text{K} \mid \text{STD µL} \mid V_{inj}\ \text{mL} \mid \text{L}}{\text{mole} \mid 22.4\ \text{L} \mid T\ \text{K} \mid \text{L} \mid \mid 1000\ \text{mL}}$$

Where:
STD = Concentration of the spiking standard, in µL/L (ppmv)
Vinj = Injection volume of the spiking standard, in mL
T = Temperature during the process, in K
MW = Molecular weight of the target gas, in g/mole

$$\text{SCV Concentration (mg/L)} = \frac{\text{Spike amount (ug)}}{V_{water}\ \text{mL}}$$

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Where:
V_{water} = Water phase volume, in mL

19.3 Analyze the four spikes following every step in the procedure.

19.4 Input to the IDC spreadsheet to calculate average recovery and relative standard deviation for each gas.

19.5 Results must be within the quality control limits associated with the SCV in Element™. If results are acceptable, the demonstration of capability study passes. The analyst and instrument are authorized to process samples.

19.6 If any gas fails, locate and correct the problem then repeat the study for the failed gas. Repeated failure indicates a general problem with the procedure and/or techniques used. If this occurs, locate problem, correct the procedure and/or techniques used then repeat the demonstration of capability study successfully. Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

19.7 A demonstration of capability study is required for each analyst annually.

19.8 A method detection limit (MDL) study in accordance with TriMatrix SOP GR-10-125 is also required annually.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written disposal instructions for that particular material.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material Safety Data Sheets are located on the laboratory intranet library.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal.

20.0 REFERENCES

20.1 Newell, B.S., RSKSOP-175, *Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC headspace Equilibration Technique*, Revision 0, August 11, 1994

2.1.1 A modification to this procedure is that injection volumes used are 200 µL instead of 300 µL.

20.2 USEPA, Region 1, *Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, and Ethene*, Revision 1, 02/21/02

21.0 ATTACHMENTS/APPENDICES

21.1 Example Spreadsheet for Sample Calculations

21.2 Standards Logbook Example

21.3 Calibration Curve for Methane Example

21.4 Chromatogram Example

21.5 Preparation Batch Report Example

21.6 Data Review Report Example

21.7 Instrument Run Log Example

21.8 Method Detection Limit Study Example

Approved By: _____ 1-22-09 **QA Officer** **Approved By:** _____ 1/23/09 **Area Supervisor**

Attachment 21.1
Example Spreadsheet for Sample Calculations

Dissolved Gases in Ground Water
METHANE

Initial Cal ID:	AREA (x)	CONC. (y)
METH_CRA.mth	2048.00	0.000004
	4245.00	0.000010
	8894.00	0.000020
	24284.00	0.000050
	47083.60	0.000100
	224316.68	0.000500
	Slope:	2.23255E-09
Blank Area: 2319.00	Y Intercept:	-1.67258E-06
	R:	0.999923

Analyte:	METHANE
Molecular Weight (g/mol):	16
Henry's Law Constant:	41300
g-moles in 1 L water:	55.5
Sample Temperature(K):	298
Density Correction Factor:	0.654
Bottle Volume(ml):	44
Headspace Volume(ml):	4.4

Sample	Sample Area Count	Analyte in Original Sample (ug/L)	Dilution Factor	Final Conc. (ug/L)	Partial Pressure	Equilibrium Mole Fraction	Moles of Gas	Saturation Conc. of Gas (mg/L)	Analyte in Head-space (mL)	Analyte in Liquid Phase (mg/L)
358388 20X	89074.00	18.58	20	371.54	0.00019719	4.77E-09	2.64988E-07	0.004239813	8.68E-04	0.01434
358389	157152.00	32.90	1	32.90	0.000349177	8.45E-09	4.69233E-07	0.007507729	1.54E-03	0.02539
358390 20X	85716.00	17.87	20	357.41	0.000189693	4.59E-09	2.54914E-07	0.004078621	8.35E-04	0.01379
358391 2X	138031.00	28.87	2	57.75	0.000306488	7.42E-09	4.11867E-07	0.006589873	1.35E-03	0.02228
358392 2X	142859.00	29.40	2	58.80	0.00031209	7.56E-09	4.19394E-07	0.006710311	1.37E-03	0.02269
358393	3290.00	0.05	1	0.05	4.95226E-07	1.20E-11	6.65497E-10	1.0648E-05	2.18E-06	0.00004
358499	8290.36	1.10	1	1.10	1.16588E-05	2.82E-10	1.56674E-08	0.000250678	5.13E-05	0.00085
358499DUP	5892.00	0.59	1	0.59	6.30432E-06	1.53E-10	8.47191E-09	0.00013555	2.77E-05	0.00046
358500 2X	165060.50	34.07	2	68.14	0.000361656	8.76E-09	4.86002E-07	0.007776039	1.59E-03	0.02629
358501 10X	107523.00	21.97	10	219.69	0.000233201	5.65E-09	3.13381E-07	0.005014094	1.03E-03	0.01696
358502 2X	140815.00	28.97	2	57.94	0.000307527	7.45E-09	4.13262E-07	0.006612194	1.35E-03	0.02236
358503 2X	186467.50	38.57	2	77.15	0.000409448	9.91E-09	5.50227E-07	0.008803628	1.80E-03	0.02977
358504 2X	135637.00	27.88	2	55.76	0.000295966	7.17E-09	3.97727E-07	0.006363637	1.30E-03	0.02152
358504DUP 2X	123817.00	25.40	2	50.79	0.000269578	6.53E-09	3.62265E-07	0.005796247	1.19E-03	0.01960
358505 10X	99607.00	20.30	10	203.04	0.000215528	5.22E-09	2.89632E-07	0.004634106	9.48E-04	0.01567
358506	3376.57	0.06	1	0.06	6.88497E-07	1.67E-11	9.2522E-10	1.48035E-05	3.03E-06	0.00005
358507 10X	96972.00	19.75	10	197.50	0.000209645	5.08E-09	2.81726E-07	0.004507619	9.22E-04	0.01524
358508	3297.00	0.05	1	0.05	5.10853E-07	1.24E-11	6.86498E-10	1.0984E-05	2.25E-06	0.00004

Approved By: QA 1-22-09 **QA Officer**

Approved By: JMM 1/23/09 **Area Supervisor**

Attachment 21.2
Standards Logbook Example

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By
1	GC3.34-1	Diesel #2 w/ Trichloroethane	GC323-1	–	2/4/04	1000ug/ml	9ml	MeCl	10ml	900ug/ml	JAM	9/7/04	12/19/04	
2	GC3. -2		~~GC3~~ PL4.9-2	–	2/3/05	500ug/ml	1ml	↓	↓	50ug/ml	↓	↓	↓	
3	GC3.34-3	1242	1242	Absolute 90126 Lot#010903	10/5/05		20ul	Hex	25ml	0.80ug/ml	JAM	10/20/04	4/6/05	
4	GC3. -4		PL4.10-14	–	4/5/05		5ml			0.20ug/ml			↓	
5	GC3.34-5	1254	1254	Absolute 70010 Lot# 020100	11/20/04		20ul			0.80ug/ml			11/20/04	
6	GC3. -6		PL4.10-14	–	4/5/05		5ml			0.20ug/ml			↓	
7	GC3.34-7	Chlordane	CHLORDALE	ultra Lot S-1084	9/2005	100 mg/mL	50ul	Hexan	50mL	0.100 mg/mL	SJPS	10/27/04	2/28/05	
8	GC3. -8		PL4.10.5 (Surr)		2/2/05	1 mg/mL	5mL	↓	↓	↓	SJPS	↓	↓	
9	GC3.34-9	AP II 1-thigh	Isodrin	Absolute 70141 Lot 011302	11/2/05	1000 µg/ml	5µL	Hexan	25mL	0.2 mg/mL	mp	10/25/04	11/19/05	
10	GC3. -10		Chlorobenzilate	Absolute 70020 Lot #011702	5/11/05		50 µL			2.0 mg/mL				
11	GC3. -11		Kepone	Absolute 70117 Lot 082502	5/11/05		50 µL			2.0 mg/mL				
12	GC3. -12		Dieldrin	Absolute 70350 Lot 040902	5/11/05		50 µL			2.0 mg/mL				
13	GC3. -13		TCMX PCB			1.0 µg/mL	5mL			0.2 mg/mL				
14	GC3.34-14	Post 1	Perthane	Ultra 8 Pm grade Lot #-0602	12/05	1000 ~~µg/mL~~ mg/mL	8µL		50mL	0.16 µg/mL		11/4/04	11/26/05	
15	GC3. -15		TCMX DCB			1 µg/mL	8mL	↓	↓	0.16 µg/mL	↓	↓	↓	
16	GC3.34-16	1016/1260 10	1016/1260	Absolute 70015 Lot #011303	11/4/05	10000ppm	40ul	Hex	25ml	1.6ppm	JAM	11/4/04	5/4/05	
17	GC3. -17		1260	Absolute 70021 Lot 051600	5/16/05	↓	↓	↓	↓	1.6ppm	↓	↓	↓	
18	GC3. -18		TCMX+DCB	PL4.11-9	5/1/05	1.0 ppm	5.0ml	↓	↓	0.20ppm	↓	↓	↓	

file: Standard Logbook Manual.xls
page 34 of 50
revision: 2.0

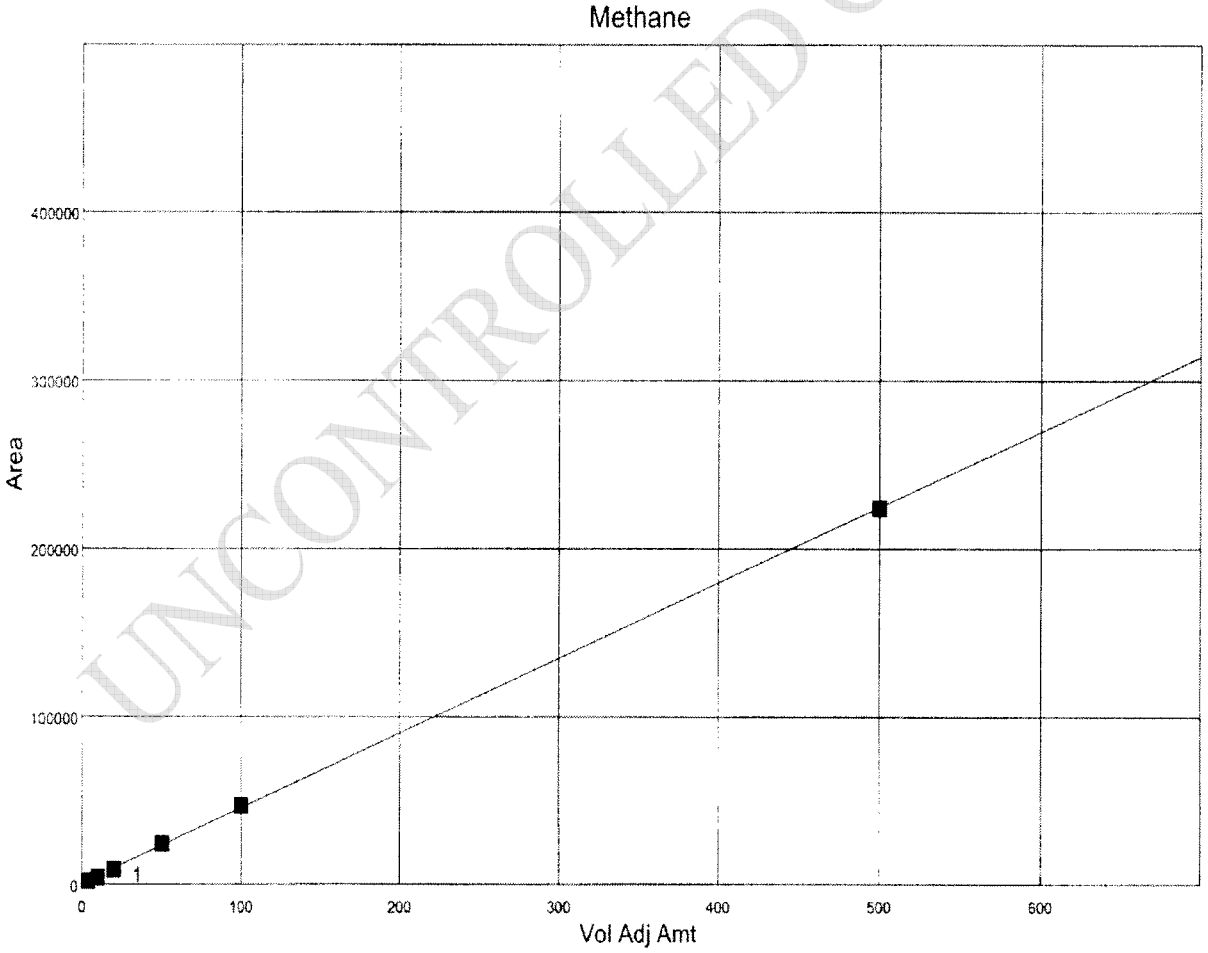
Approved By: qm 1-22-09 **QA Officer**
Approved By: JMC 1/23/09 **Area Supervisor**

Attachment 21.3
Calibration Curve for Methane Example

Fit Analysis Output For Method File: "C:\TC4\159\RSK42304.MTH"
Component Name: "Methane"
Date: 11/22/04 Time: 10:37

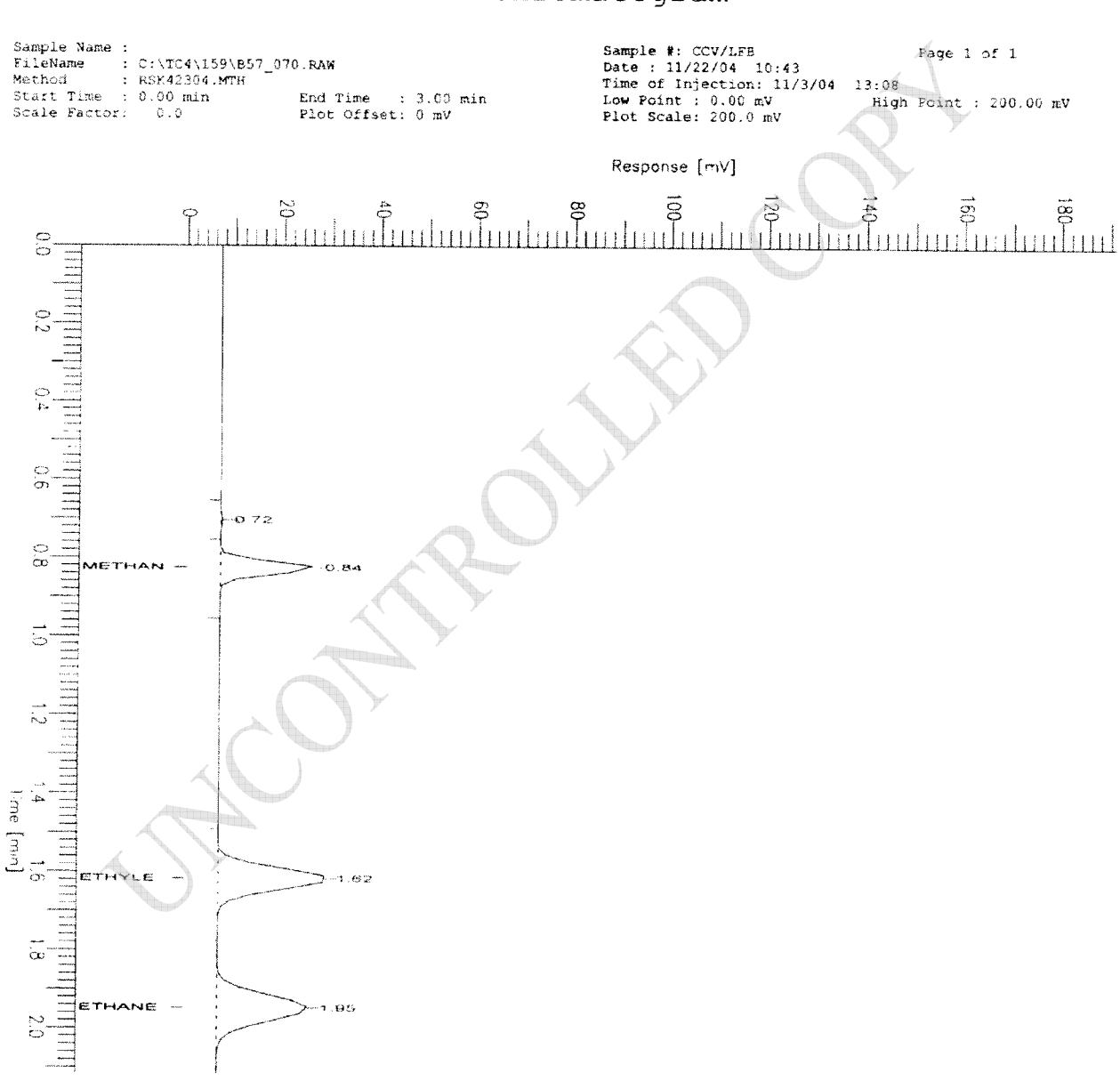
Curve Parameters:

Curve #1 : 1st Order
Weighting Factor = 1.0 (No Weighting) r^2 = 0.999846
Calibration Curve = (757.021844) + (447.849633)X



Approved By: _____ 1-22-09 **Approved By:** _____ 1/23/09
QA Officer **Area Supervisor**

Attachment 21.4
Chromatogram Example



Approved By: 1-22-09 **QA Officer**

Approved By: 1/23/09 **Area Supervisor**

Attachment 21.5
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0707324 Page 1 of 1

Printed: 7/6/2007 4:21:20PM

Semivolatiles GC, Water, Direct Injection

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0706375	RSK-175	0706397	RSK-175		

Lab Number	*Contain*	*Prepared*	*By*	*Initial (mL)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0707324-BLK1		Jun-29-07 12:33	JLW	1	1					BLANK	
0707324-DUP1		Jun-29-07 12:33	JLW	1	1		0706397-10			DUPLICATE	
0707324-BS1		Jun-29-07 12:33	JLW	1	1			6100100	10	LCS	
0706375-01	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706375-02	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706375-03	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706375-04	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706375-05	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706375-06	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-01	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-02	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-03	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-04	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-05	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-06	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-07	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-08	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-09	C	Jun-29-07 12:33	JLW	1	1					[redacted]	
0706397-10	C	Jun-29-07 12:33	JLW	1	1					[redacted]	

Comments	Analyst Initials

bch_TriMatrix.rpt

Approved By: QA 1-22-09 **QA Officer**

Approved By: [signature] 1/23/09 **Area Supervisor**

Attachment 21.6
Data Review Report Example

Sequence = 7062967 Page 1 of 1 on 7/6/2007 at 4:35

SampleID	Analysis	IResult	Qualifier	IUnits	Dilu	FResult	FUnits	IMDL	IMRL	FMRL	Recovery	RPD	Analyzed
0707324-BS1	RSK-175	17.6		ug/L	1	17.6	ug/L	0.147	1	1.0	99		6/29/2007 1:27
0707324-BS1	RSK-175	16.47		ug/L	1	16.5	ug/L	0.167	1	1.0	100		6/29/2007 1:27
0707324-BS1	RSK-175	9.08		ug/L	1	9.08	ug/L	0.0785	0.5	0.50	96		6/29/2007 1:27
0707324-BLK1	RSK-175	0		ug/L	1	0.00	ug/L	0.147	1	1.0			6/29/2007 1:34
0707324-BLK1	RSK-175	0		ug/L	1	0.00	ug/L	0.0785	0.5	0.50			6/29/2007 1:34
0707324-BLK1	RSK-175	0		ug/L	1	0.00	ug/L	0.167	1	1.0			6/29/2007 1:34
0706375-01	RSK-175	4.13		ug/L	1	4.1	ug/L	0.0785	0.5	0.50			6/29/2007 1:38
0706375-01	RSK-175	0		ug/L	1	0.0	ug/L	0.167	1	1.0			6/29/2007 1:38
0706375-01	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:38
0706375-02	RSK-175	45.88		ug/L	1	46	ug/L	0.0785	0.5	0.50			6/29/2007 1:42
0706375-02	RSK-175	2.82		ug/L	1	2.8	ug/L	0.167	1	1.0			6/29/2007 1:42
0706375-02	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:42
0706375-03	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:45
0706375-03	RSK-175	1.08		ug/L	1	1.1	ug/L	0.167	1	1.0			6/29/2007 1:45
0706375-03	RSK-175	5.75		ug/L	1	5.8	ug/L	0.0785	0.5	0.50			6/29/2007 1:45
0706375-04	RSK-175	2.22		ug/L	1	2.2	ug/L	0.0785	0.5	0.50			6/29/2007 1:49
0706375-04	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:49
0706375-04	RSK-175	0.94		ug/L	1	0.94	ug/L	0.167	1	1.0			6/29/2007 1:49
0706375-05	RSK-175	4.43		ug/L	1	4.4	ug/L	0.0785	0.5	0.50			6/29/2007 1:52
0706375-05	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:52
0706375-05	RSK-175	2.27		ug/L	1	2.3	ug/L	0.167	1	1.0			6/29/2007 1:52
0706375-06	RSK-175	0.06		ug/L	1	0.060	ug/L	0.167	1	1.0			6/29/2007 1:56
0706375-06	RSK-175	16.76		ug/L	1	17	ug/L	0.0785	0.5	0.50			6/29/2007 1:56
0706375-06	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 1:56
0706397-10	RSK-175	3.92		ug/L	1	3.9	ug/L	0.0785	0.5	0.50			6/29/2007 2:37
0706397-10	RSK-175	0		ug/L	1	0.0	ug/L	0.147	1	1.0			6/29/2007 2:37
0706397-10	RSK-175	1.31		ug/L	1	1.3	ug/L	0.167	1	1.0			6/29/2007 2:37
0707324-DUP1	RSK-175	0		ug/L	1	0.00	ug/L	0.147	1	1.0			6/29/2007 2:40
0707324-DUP1	RSK-175	1.29		ug/L	1	1.29	ug/L	0.167	1	1.0		2	6/29/2007 2:40
0707324-DUP1	RSK-175	3.93		ug/L	1	3.93	ug/L	0.0785	0.5	0.50		0.3	6/29/2007 2:40

Attachment 21.7
Instrument Run Logbook Example

TriMatrix Laboratories, Inc.

Instrument ID: Varian 3400 #159	Date: 9/8/04	Sequence #: B56	Date Archived, Disk ID

Instrument Settings/Injection Volume	Breakdown	Quantitation Information			Routine Maintenance Items
Column Type: DB624 / Porapak	Curve Date:	GC	Program 1	Program 2	New Column Date:
Injection Volume: 1 ul / 2 uL / 200 ul	Curve Type: Regression or Average CF	Initial:	45°	–	Column Clipped: Yes (No)
Head Pressure: 20 psi	Quantitated By: Curve	Hold:	3min	–	New Injection Port Liner: Yes (No)
Injector Temperature: 250° C		Final:	–	–	New Septa: Yes (No)
Detector Temperature: 280° C		Rate:	–	–	New Syringe: Yes (No)
Program Name:		Hold:	–	–	

Analyst	Run ID	File ID	Injection Time	Method	Client	Matrix	Dilution	ICV/CCV Check (85-115)	Sample Notes, Standard Numbers, Analytical Batch Information, etc.
JAM	Inst BLK	1	14 15	RSK42304		QC		PASS	
	CCV/LFB	2	14 19			QC		PASS	S-2132
	MPB	3	14 23			Wtr			
	369189	4	14 26		Merit				
	369189	5	14:47				20x		
	369190	6	14 50						
	369191	7	14 54						
	369192	8	14 57						
	369192	9	15:02				20x		
	369193	10	15:07						
	369194	11	15:10						
	369194	12	15:16 ~~15:11~~ JAM 9/8/04		✓	✓	20x ~~10x~~ JAM 9/8/04		
	CCV	13	15:20			QC		PASS	S-2132
	369194dup	14	15:25		Merit	Wtr	20x		
↓	CCV	15	15:33	↓					S-2132

Approved By: _____ 1-22-09 QA Officer
Approved By: _____ 1/23/09 Area Supervisor

Attachment 21.8
Method Detection Limit Study Example

SEMI-VOLATILE LABORATORY
INSTRUMENT NUMBER 159 2004 WATER
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average % Recovery	Standard Deviation	MDL
METHANE	RSK-175	1/28/2004	0.500	ug/L	0.160	0.090	0.120	0.130	0.090	0.060	0.130	0.111	22%	0.0334	0.105
ETHYLENE	RSK-175	1/28/2004	0.500	ug/L	0.470	0.560	0.510	0.470	0.520	0.580	0.480	0.513	103%	0.0439	0.138
ETHANE	RSK-175	1/28/2004	0.500	ug/L	0.500	0.590	0.540	0.500	0.560	0.620	0.530	0.549	110%	0.0449	0.141

file: RSK-175_WATER_Calculated_New.xls

revision: 2004.02

Approved By: PD 1-22-09 **QA Officer**

Approved By: JMW 1/25/09 **Area Supervisor**

Attachment B:

Henry's Law Constants

Temperature (^{0}C)	**Methane**	**Ethane**	**Ethene**
0	22,400	12,600	5,520
5	25,900	15,500	6,530
10	29,700	18,900	7,680
15	33,700	22,600	8,950
20	37,600	26,300	10,200
25	41,300	30,200	11,400
30	44,900	34,200	12,700
35	48,600	38,300	NA
40	52,000	42,300	NA

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Nitrate, Nitrite and Nitrate-Nitrite Nitrogen by Automated Colorimetry

Standard Methods 4500-NO_3^- F

APPROVALS:

Area Supervisor: Heather L. Brady Date: 1-31-09
Heather L. Brady

QA Officer: Tom C. Boocher Date: 1-23-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 2/4/09
Jeff P. Glaser

Procedure Number: GR-05-107
Revision Number: 3.2

Date Initiated: 6/20/94
Effective Date: 2/21/09

Date Revised: 1/23/09
Pages Revised: All

By: Jodi L. Blouw
Total Number of Pages: 22

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-19-10	Tom Boocher	4-19-11

1.0 SCOPE AND APPLICATION

1.1 Matrices applicable for nitrate and nitrite nitrogen analysis include potable water, wastewater effluents and soil extracts.

1.2 The range of analysis for potable water and wastewater effluents is 0.05 - 1.0 mg/L which may be extended by dilution.

1.3 The range of analysis for soil matrices is 1.0 - 20 mg/kg (dry weight).

1.4 The reporting limit for potable water and wastewater is 0.05 mg/L.

1.5 The reporting limit for soil matrices is 1.0 mg/kg (dry weight). The soil matrix reporting limit will vary based on sample percent solids.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1997,* 4500-NO_3^- Nitrogen (Nitrate), F, Automated Cadmium Reduction Method

2.2.1 This procedure deviates from the referenced method by use of a 10 mm path length flow cell instead of the specified 15 mm flow cell.

3.0 SUMMARY OF PROCEDURE

3.1 A filtered sample is passed through a granulated copper-cadmium (Cu-Cd) column to reduce nitrate to nitrite.

3.2 The nitrite that was originally present plus reduced nitrate is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride. A highly colored azo dye is formed which can be measured colorimetrically.

3.3 Nitrate and/or nitrite alone rather than nitrate-nitrite values are obtained by carrying out the procedure with and then without the Cu-Cd reduction step.

3.4 The magenta-colored azo dye is directly proportional to the nitrite concentration at 520 nm.

3.5 When analyzing for nitrite by carrying out the procedure without the Cu-Cd reduction step, the analysis MUST be performed on non-preserved samples. Nitrate is determined by subtracting the nitrite result from the nitrate-nitrite result.

4.0 PARAMETER OR COMPOUND LIST

4.1 Nitrate-Nitrite Nitrogen

4.2 Nitrate Nitrogen

4.3 Nitrite Nitrogen

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-05-114, *Nitrite Nitrogen by Colorimetric Spectrophotometry*, latest revision

5.2 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.4 TriMatrix SOP GR-16-103, *Glassware Cleaning for Wet Chemistry and Metals*, latest revision

5.5 Lachet QuikChem® Method 10-107-04-1-C, *Determination of Nitrate/Nitrite in Surface and Wastewaters by Flow Injection Analysis*, April 23, 1999

5.6 TriMatrix SOP GR-02-103, *Lachat QuikChem® AE8000 Operation*, latest revision

5.7 TriMatrix SOP GR-18-118, *Residual Chlorine*, latest revision

5.8 TriMatrix SOP GR-16-117, *Extraction of Soluble Inorganic Analytes from Soil*, latest revision

5.9 TriMatrix SOP GR-10-104, *Chain-of-Custody (CoC)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 A build-up of suspended matter in the reduction column will restrict sample flow. Since nitrate and nitrite are found in a soluble state, samples must be pre-filtered using a 0.45 µm membrane filter to minimize maintenance to the reduction column.

6.2 Low results will be obtained for samples that contain high iron, copper or other metal concentrations. Add EDTA (Section 11.4) to minimize such interference.

6.3 Residual chlorine can produce a negative interference by limiting reduction efficiency. Check each sample for residual chlorine before analysis and if present, de-chlorinate with sodium thiosulfate (Section 13.2).

6.4 Samples with a high oil and grease concentration will coat the cadmium surface. Remove oil & grease by extracting with methylene chloride.

6.5 Interferences may also be caused by contaminants in reagent water, reagents, glassware and/or other processing equipment that can bias response. Glassware must be scrupulously clean in accordance with TriMatrix SOP GR-16-103.

7.0 SAFETY PRECAUTIONS

7.1 The toxicity or carcinogenicity of each reagent used has not been fully established. Each chemical must be regarded as a potential health hazard and exposure must be as low as reasonably achievable.

7.2 A reference file of Material Safety Data Sheets (MSDS) in maintained on the laboratory intranet library.

7.3 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.4 Personal protective equipment is mandatory when working in the laboratory and includes but is not limited to the use of a laboratory coat, approved safety glasses and disposal gloves.

7.5 The following chemicals have the potential to be highly toxic or hazardous. At no time are these chemicals to be handled without disposable gloves. Consult the MSDS if there is any question about handling, storing and/or disposal.

7.3.1 Cadmium (Cd)

7.3.2 Phosphoric acid (H_3PO_4)

7.3.3 Hydrochloric acid (HCl)

7.3.4 Sulfuric acid (H_2SO_4)

7.3.5 Methylene chloride ($MeCl_2$)

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples must be stored after receipt/log-in at 4 ±2° C until analysis.

8.2 Hold times are as follows:

8.2.1 Nitrite N must be analyzed within 48 hours of sample collection on an un-preserved sample.

8.2.2 Un-preserved Nitrate-Nitrite N must be analyzed with 48 hours of sample collection.

8.2.3 Preserved (with sulfuric acid) Nitrate-Nitrite N must be analyzed within 28 days of sample collection.

8.3 Nitrate N can only be determined by subtracting nitrite N from the nitrate-nitrite N result.

8.4 If it is not possible to run the nitrate-nitrite analysis within 48 hours from sample collection, the nitrite N analysis must first be analyzed by this procedure or TriMatrix SOP GR-05-114 within the hold time. The rest of the sample may then be preserved with sulfuric acid to a pH <2.0 and the nitrate-nitrite N analysis performed within the 28-day hold time.

8.5 Sulfuric acid-preserved samples must be neutralized with NH_4OH to pH 5 - 9 before the nitrate-nitrite analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Lachat QuikChem® 8000 Automated Flow Injection Analyzer with the following specifications:

9.1.1 Autosampler

9.1.2 Multi-channel proportioning pump

9.1.3 Injection valve module with 22.5 cm x 0.8 mm loop

9.1.4 10 nm band pass, 80 µL, glass flow cell with 10 mm cell path

9.1.5 520 nm interference filter

9.1.6 Two-stage switching valve

9.1.7 Reaction module

9.1.8 Cadmium-copper reduction column (Lachat 50237)

9.2 Degassing wand

9.3 Vials and bottles, various sizes for holding standard solutions

9.4 Autosampler tubes

9.5 Class A volumetric pipets, various sizes

9.6 Class A Volumetric flasks, various sizes

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Refer to TriMatrix SOP GR-02-103 for preventive maintenance schedules.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM Type II, MilliQ system

11.2 Prepare ACS sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) solution for eliminating residual chlorine as follows:

11.2.1 Dissolve 0.35 g of $Na_2S_2O_3 \cdot 5H_2O$ into approximately 60 mL of laboratory reagent water in a 100 mL volumetric flask.

11.2.2 After dilution is complete, dilute to volume with laboratory reagent water. Store tightly capped.

11.2.3 Expiration is 7 days from the date prepared.

11.3 Ammonium hydroxide (NH_4OH), ACS, concentrated, 30%, for pH adjustment

11.4 Prepare the ammonium chloride-EDTA solution as follows:

11.4.1 In a fume hood, transfer approximately 1000 mL of laboratory reagent water into a 2 L volumetric flask.

11.4.2 Slowly add 210 mL of concentrated hydrochloric acid.

11.4.3 Slowly add 190 mL of ammonium hydroxide.

11.4.4 Add 2.0 g of disodium ethylenediamine tetraacetate (EDTA). Let cool to room temperature.

11.4.5 After cooling to room temperature, adjust the pH to 8.5 with concentrated NH_4OH and hydrochloric acid (HCl) then dilute to 1 L with laboratory reagent water.

11.4.6 To the diluted solution, add 0.5 mL polyoxyethylene 23 lauryl ether (Brij-35).

11.5.7 Expiration is six months from the date made.

11.5 Sulfanilamide Color Reagent is prepared as follows:

11.5.1 To a 2 L volumetric flask add about 1200 mL of laboratory reagent water.

11.5.2 Measure and add 200 mL of 85% phosphoric acid (H_3PO_4) and swirl to mix.

11.5.3 Add 80.0 g sulfanilamide.

11.5.4 Add 2.0 g N-1-naphthylethylenediamine dihydrochloride (Marshall's reagent).

11.5.5 Shake and/or stir until dissolved then dilute to the mark with laboratory reagent water. Store in a dark bottle.

11.5.6 Expiration is 30 days from the date made. The solution should be slightly pinkish but not a strong color (indicating contamination). If the solution becomes contaminated before the expiration date, it must be replaced.

11.6 Cadmium, coarse granules (0.3 - 1.5 mm diameter).

11.7 Prepare 1N hydrochloric acid (HCl) as follows:

11.7.1 In a 120 mL bottle, add 8 mL of concentrated HCl to 92 mL of laboratory reagent water.

11.7.2 Cap and shake to mix.

11.7.3 Expiration is six months from the date made.

11.8 Prepare a 2% (wt/vol) copper sulfate solution as follows:

11.8.1 In a 1 liter volumetric flask, dissolve 20 g anhydrous copper sulfate ($CuSO_4$) in about 800 mL of laboratory reagent water.

11.8.2 After dissolution, dilute to volume with laboratory reagent water and invert to mix thoroughly.

11.8.3 Expiration is six months from the date made.

11.9 All reagents must be recorded accurately and appropriately in the reagent logbook and/or in the laboratory information management system (LIMS).

11.10 All reagents must be reagent grade or better.

12.0 STANDARDS PREPARATION

12.1 Prepare a 100 mg/L nitrate (NO_3) solution as follows:

12.1.1 In a 500 mL volumetric flask, dissolve 0.361 g of potassium nitrate (KNO_3) in about 300 mL of laboratory reagent water.

12.1.12 Add 1 mL of chloroform then dilute to volume with laboratory reagent water and mix well.

12.1.13 Expiration is 6 months from the date made. Store at 4 ±2°C.

12.2 Prepare a 100 mg/L nitrite (NO_2) solution as follows:

12.2.1 In a 500 mL volumetric flask, dissolve 0.2465 g of sodium nitrite ($NaNO_2$) in 300 mL of laboratory reagent water.

Note: Sodium nitrite is extremely hydrophilic and must be stored in a desiccator.

12.2.2 Add 1 mL of chloroform then dilute to volume with laboratory reagent water and mix well.

12.2.3 Expiration is 7 days from the date made. Store at 4 ±2°C.

12.3 Calibration standards are prepared as follows:

12.3.1 When the "autodilute" instrument function is used, prepare a 10 mg/L solution from the nitrate and nitrite stock solutions by diluting 10 mL of stock into 100 mL of laboratory reagent water, in a volumetric flask.

12.3.2 Using the 10 mg/L solution, prepare a separate calibration series for nitrate and nitrite as follows:

Initial concentration (mg/L)	Volume to pipet into a 200 mL volumetric flask (mL)	Final Concentration (mg/L)
10	20	1.00
10	10	0.50
10	5.0	0.25
1.00	20	0.10
1.00	10	0.05

12.4 Record all prepared calibration standards in the appropriate standards logbook and/or into LIMS.

13.0 SAMPLE PREPARATION

13.1 If a sample has turbidity, remove by filtration with 0.45 µm membrane filter prior to analysis.

13.2 Potable water samples or treated effluents must be tested for residual chlorine.

13.2.1 Test for residual chlorine in accordance with TriMatrix SOP GR-18-118.

13.2.2 To treat a sample for residual chlorine, measure 1.0 mL of sodium thiosulfate solution per 500 mL of sample (Section 11.2) for each mg/L of residual chlorine. If treating other than 500 mL, adjust the sodium thiosulfate volume accordingly.

13.2.3 Record on the benchsheet that a sample has been checked and/or treated to remove residual chlorine. Include the test result, the volume of sodium thiosulfate used and the date treatment was performed.

13.3 Soil samples such as soil, sludge and sediment must be extracted prior to analysis in accordance with TriMatrix SOP GR-16-117 using 2.0M potassium chloride (KCl).

14.0 CALIBRATION PROCEDURES

14.1 A description of the calibration is included in Section 15.2.5. It is performed using the five standards prepared in Section 12.4 and the laboratory reagent water blank. The calibration range is 0.05 to 1.0 mg/L. With autodilute, the range may be extended to 50 mg/L.

14.2 The tray protocol for quality control includes the initial calibration water blank (ICB), the secondary calibration verification (SCV), a continuing calibration blank (CCB) and continuing calibration verifications (CCV) every ten samples.

14.3 Continuing calibration blanks and verifications are taken from the ICB and one initial calibration point in the autosampler. These are not introduces as separate solutions in the tray protocol.

14.4 Acceptable limits for these quality control are as follows:

14.4.1 Any ICB or CCB is acceptable if the result is less than the reporting limit.

14.4.2 Any ICV or CCV is acceptable if the result is within 90 - 110% of the prepared concentration.

14.5 Autodilute verifications are analyzed as part of each analysis and must be within 90-110% of the expected concentration.

15.0 ANALYTICAL PROCEDURE

15.1 The cadmium column is prepared by weighing 10 - 20 g of cadmium into a 250 mL beaker. The cadmium is comprised of granules are 0.3 - 1.5 mm in diameter and may be obtained from Lachat (part number 50231).

15.2 To the cadmium beaker, wash with 50 mL of acetone then decant and wash with laboratory reagent water to remove the acetone.

15.3 Next, wash with two 50 mL aliquots of 1.0 N hydrochloric acid, decanting between each wash.

15.4 After the acid, wash several times with laboratory reagent water to remove all acid. The color of acid-washed copper is silvery.

CAUTION: Collect and store all waste cadmium for proper disposal. Cadmium is toxic and carcinogenic. Refer to the Material Safety Data Sheet.

15.5 For copperization, add 100 mL of 2% copper sulfate to the acid-washed cadmium. Swirl for about 5 minutes until the blue copper color begins to fade. Decant and repeat with fresh copper sulfate until a brown colloidal precipitate forms then decant and wash at least 10 times with laboratory reagent water to remove the precipitate. The cadmium will be black and is ready for filling the column.

15.6 The empty cadmium column is available as Lachat part 50230. As a reminder, gloves must be worn when handling chemicals. Do all cadmium transfers over a special tray or beaker dedicated to this purpose.

15.7 First, clamp the empty column upright. Unscrew the colored fitting from top end of the column and pull it out but save the foam plug. Care must also be taken not to break or chip the column and glass threads. Fasten the loosened fitting higher than the open end of the column and completely fill the column, fittings, and tubing with ammonium chloride buffer.

15.8 After the buffer has been added, transfer copperized cadmium with a spatula into the top of the filled column so the granules sink to the bottom. Continue transferring cadmium while gently tapping the column to prevent air bubbles and gaps as it fills. When the cadmium is approximately 5 mm from the top, insert the foam plug and carefully screw on the top fitting. Rinse the outside of the column with laboratory reagent water.

Note: If air becomes trapped in the column, connect the column to the manifold, turn the pump on maximum and tap gently while working up the column until all air is removed.

15.9 Insert the filled cadmium column as follows:

15.9.1 Before inserting the column, pump all reagents into the manifold then turn the pump off.

15.9.2 A 2-stage valve is used to position the cadmium column in the manifold. Insert the column then switch the valve to the in-line position and turn the pump on again.

15.9.4 The direction of reagent flow through the column is not relevant.

15.9.5 Before proceeding, test the column efficiency in accordance with the written instructions in Attachment 23.3.

15.10 The instrument is operated as follows:

15.10.1 Inspect each module for the proper connection. Refer to the manifold diagram in Attachment 23.4.

15.10.2 Turn on the power and all modules.

15.10.3 Place each reagent feedline into the proper solution reservoir.

15.10.4 Let the system pump until a stable baseline is obtained.

15.10.5 Place the nitrate and nitrite calibration standards, and a laboratory reagent blank in the sample tray in descending order of concentration.

15.10.5.1 Initiate and run the calibration.

15.10.5.2 When the calibration run has finished, review the curve and calibration report. If the calibration is acceptable and has a correlation coefficient of greater than 0.995, approve it.

15.10.5.3 Refer to Attachment 23.5 to view a typical calibration.

15.10.6 Fill a sample tube with sample. Repeat for each sample and matrix quality control. Arrange each sample tube in the sample tray in the run sequence desired. Input each sample number into the run protocol then save under an appropriate filename.

15.10.7 Submit the sample tray to begin analysis.

15.10.8 At the end of the run, disconnect the cadmium column, place all feedlines in laboratory reagent water and rinse for at least 15 minutes.

15.10.9 After rinsing, place all feedlines on the counter and pump only air until the lines are dry.

15.10.10 Finally, turn off the pump and all modules then release each pump tube to prevent crimping.

15.11 The required quality control must be analyzed with each analysis sequence as outlined in Section 18.0.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The Lachat software compares sample response against the calibration and prints the result in concentration (mg/L as N). Manual calculation is only necessary on diluted samples not taken into account by the autodilute function.

16.2 Nitrate-nitrite N and nitrite N are calculated by the instrument. Nitrate N is calculated as follows:

A-B = C

where:
A = Nitrate plus Nitrite N, in mg/L
B = Nitrite N, in mg/L
C = Nitrate N, in mg/L

16.3 Extracted soils need reported as "exchangeable" nitrate N, nitrite N or nitrate-nitrite N.

17.0 DATA REPORTING AND DELIVERABLES

17.1 The analyst is responsible for data quality and for correctly filling in all analytical documentation. This is required for quality control and to provide the client with defensible data.

17.2 The following must be attached to the preparation batch report for archiving:

17.2.1 The calibration statistics report.

17.2.2 The peak area plot.

17.2.3 The calibration curve.

17.2.4 The datafile run report.

17.2.5 The calibration summary report.

17.2.6 Sample preparation forms, if necessary.

17.3 The following information must be recorded in the instrument run logbook for each analysis:

17.3.1 The date analyzed.

17.3.2 The analyst's initials.

17.3.3 The method parameter and reference number.

17.3.4 All calibration standards used.

17.3.5 The client name and sample numbers analyzed.

17.4 The logbook must be filled in completely and correctly. All entries are to be made in indelible blue or black ink as follows:

17.4.1 Corrections are to be made with a single lineout which is then dated and initialed. The erroneous result must remain legible.

17.4.2 The corrected data can then be written beside the lineout.

17.4.3 Write-overs are NOT permitted in any laboratory notebook or on any laboratory hardcopy.

17.4.4 Blank areas and/or pages in the logbook must be Z'd out to document the space has been intentionally left blank.

17.5 The following information must be included in the archived hardcopy for each analysis:

17.5.1 The analysis date

17.5.2 The reviewing supervisor's initials and date of data review.

17.5.3 Calibration standard numbers.

17.5.5 The wavelength used.

17.5.6 All calibration standard concentrations

17.5.7 All calibration, quality control and sample responses

17.6 If internal chain-of-custody is required it is very important the COC for be filled in correctly and completely. Refer to TriMatrix SOP GR-10-104.

17.7 Refer to Attachment 23.6 for an Instrument Logbook example.

17.8 Refer to Attachment 23.7 for a Preparation Batch Report example.

18.0 QUALITY ASSURANCE

18.1 Method quality control (QC) for aqueous samples consists of an Instrument Blank (BLK), Second-source Calibration Verification (SCV), Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verifications (CCV), Continuing Calibration Blanks (CCB) and a Reporting Limit Confirmation (CRL).

18.2 Additionally, extracted soil samples require an extracted Method Preparation Blank (MPB/BLK) and Blank Spike (LFB/BS). Spiking of the MPB/BLK and LFB/BS is discussed in TriMatrix SOP GR-16-117. All batching and recording requirements apply to the MPB/BLK and LFB/BS.

18.3 A BLK and a SCV must be analyzed with each batch analysis

18.4 The BLK is laboratory reagent water analyzed as a sample. The result must be less that the reporting limit to be acceptable.

18.5 The SCV is a commercially prepared and certified standard of known concentration that is not associated with the material used to prepare the calibration. Prepare the SCV in accordance with instructions supplied with standard. Preparation of the SCV must be recorded in the standards logbook and/or laboratory information management system (LIMS) with the following information:

18.5.1 Initials of the preparer

18.5.2 The preparation date

18.5.3 The concentration with units

18.5.4 The parameter name

18.5.5 The standards logbook number

Note: Acceptance limits for the SCV are 90-110% of the prepared value.

18.6 The ICV is the midrange calibration standard analyzed immediately after generation of the calibration. Acceptance limits are 90-110 % of the prepared concentration.

18.7 The ICB is laboratory reagent water analyzed as a sample immediately after the ICV. The result must be less than the reporting limit.

18.8 All CCVs are the midrange standard analyzed as a sample after every ten samples and at the end of the analysis sequence. Acceptance limits are 90-110 % of the prepared concentration. Included in the ten-sample count are the SCV, BLK, matrix spike (MS/SPK) and matrix spike duplicate (MSD/SPK).

18.9 All CCBs are laboratory reagent water analyzed as a sample after each CCV. The result must be less than the reporting limit.

18.10 The CRL is the 0.05 mg/L calibration standard analyzed as a sample. Acceptance limits are 0.03 - 0.07 mg/L N.

18.11 Prepare and analyze a matrix spike and matrix spike duplicate every 20 samples for each sample matrix.

18.11.1 To prepare a matrix spike for water samples, add 50 µL of the 100 mg/L standard to 10 mL of sample. The spike concentration in the sample is 0.5 mg/L. Recovery must be between 90-110% of the spiked value. Matrix spike and spike duplicate precision must be less than or equal to 20% relative percent difference.

18.11.2 Soil samples must be spiked before being extracted.

18.2 Unacceptable quality control results must be addressed in accordance with TriMatrix SOP GR-10-106 unless the appropriate corrective action is otherwise indicated. All out-of-control results must be addressed.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. While IDC studies are not necessarily instrument-dependent, one is required for each instrument used in sample analysis to demonstrate the instrument's ability to generate acceptable accuracy and precision.

19.1.1 The Initial Demonstration of Capability Study is performed as follows:

19.1.1.1 Spike four aliquots of laboratory reagent water with SCV standard so the resulting concentration is in the lower half of the calibration range. Process the four spiked aliquots as samples following every step outlined in the procedure.

19.1.1.2 Input the four results into the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average percent recovery must be 90-110 % of the spiked concentration. Relative standard deviation must be less than or equal to 20%.

19.1.1.3 Alternatively, the last four of seven results obtained from a method detection limit (MDL) study may be used as the IDC if the study is run exclusively by the analyst. Remember to use ONLY the last four results.

19.1.1.4 If any criterion fails, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure and/or techniques used. If this occurs, locate and correct the procedure and/or techniques then repeat the study.

19.1.1.5 Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

19.1.1.6 Copies IDC/instrument validation spreadsheets must be given to the Quality Assurance Department for training documentation.

19.1.2 A Continuing Demonstration of Capability (CDC) is required annually and may be completed in one or more of the following ways:

19.1.2.1 Repeating the IDC study.

19.1.2.2 Analysis of four consecutive SCV standards during the course of routine sample analysis.

19.1.2.3 Obtaining an acceptable performance testing sample result from a performance evaluation study.

19.2 Method Detection Limit Studies are performed as follows:

19.2.1 A Method Detection Limit (MDL) study must be performed annually for each instrument. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. Actual reporting limits are derived from the MDL study. The minimum possible non-estimated reporting limit is equal to the concentration spiked in the MDL study provided the MDL passes. The reporting limit actually achieved for any given sample will vary depending on instrument sensitivity, matrix effects and dilutions.

19.2.2 The procedure followed for an MDL study is based on the method given in 40 Code of Federal Regulations, part 136, Appendix B, latest revision.

19.2.3 Seven replicate analyses are performed using laboratory reagent water spiked at the estimated minimum reportable concentration.

19.2.4 The standard deviation using all seven analyses is calculated and multiplied by 3.143 with the resultant number being the calculated MDL.

19.2.5 If the concentration spiked is between the calculated MDL and less than or equal to five times the calculated MDL, and there are no zero percent recoveries, the MDL study is acceptable. If not, the study must be repeated. If a study needs repeated at a different concentration, the entire study needs repeated. If a study fails due to only one of the seven results, only that result needs

repeated. However, only one result may be rejected from the dataset before the entire study needs repeated.

19.2.6 All seven results do not need obtained from the same analytical batch.

19.2.7 If at any time a reporting limit is above a client or state desired reporting limit, the calculated MDL may be used as a reporting limit provided results are narrated. The narration must state that the reporting limit is the calculated MDL and when spiked at that level, analyte may not be observed.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library and may be accessed from any laboratory computer.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Cadmium waste must be collected in the containers designated to cadmium waste collection. Cadmium waste is to be disposed of through a contracted and licensed hazardous waste company.

21.4 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal.

22.0 REFERENCES

22.1 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1997,* 4500-NO_3^- Nitrogen (Nitrate), F, Automated Cadmium Reduction Method

22.2.1 This procedure deviates from the referenced method by use of a 10 mm path length flow cell instead of the specified 15 mm flow cell.

23.0 ATTACHMENTS/APPENDICES

23.1 Standards Logbook Example

23.2 Cadmium Column Efficiency Instructions

23.3 Manifold Diagram

23.4 Calibration Example

23.5 Instrument Logbook Example

23.6 Preparation Batch Report Example

Attachment 23.1
Standards Logbook Example

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By	Checked Against Standard #
1	IN1.21.1	orthophos	IN1.8.8			1000ppm	10 mL	H_2O	100 mL	100ppm	SAJ	11/3/09	11/4/09		
2	IN1.21.2	↓	IN1.8.9			↓	↓	↓	↓	↓	↓	↓	↓		
3	IN1.21.3	Chloride	764J.37			1000ppm	40mL	H_2O	100mL	400ppm	SAJ	11/3/09	12/3/09		
4	IN1.21.4					100ppm	15mL		100mL	150ppm					
5	IN1.21.5					1000ppm	25mL		250mL	100ppm					
6	IN1.21.6					100ppm	6mL		100mL	60ppm					
7	IN1.21.7	SAJ	764J37			1000ppm	2.0mL		100mL	20ppm					
8	IN1.21.8		IN1.21.5			100ppm	10mL		100mL	10ppm	SAJ	SAJ	SAJ		
9	IN1.21.9					100ppm	5mL		100mL	5.0ppm					
10	IN1.21.10		SAJ			100ppm	2mL		100mL	2.0ppm					
11	IN1.21.11	Chloride	IN1.21.5			100ppm	2mL	H_2O	200mL	1.0ppm	SAJ	11/3/09	12/3/09		
12	IN1.21.12	TOC	IN1.19.12			10ppm	1ml	H_2O	100ml	1.0ppm	JMB	11/6/09	11/6/09		
13	[illegible]	Sulfite				1.5176g	1000ml	H_2O	1000ml	1000ppm	JMB	10/30/09	10/30/09		
14	IN1.21.14	Sulfate	225854		5/1/01	1000mg/L	20ml	H_2O	100ml	200mg/L	JMB	11/6/09	12/6/09		
15	IN1.21.15						15mL		100mL	150					
16	IN1.21.16						20mL		200mL	100					
17	IN1.21.17	↓	↓			↓	16mL	↓	200mL	80		↓	↓		
18	IN1.21.18	Sulfate	225854			1000mg/L	5mL	H_2O	100mL	50		11/6/09	12/6/09		

file: standardslogs.xlsx page: 21 of 58 revision: 1.0

Approved By: M 1-23-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.2
Cadmium Column Efficiency Instructions

QuikChem Method 10-107-04-1-C Nitrate/Nitrite Page 8

Column Efficiency Procedure

a. Visually inspect the column. Check for air bubbles in the column or lines, gaps in the column or any change in the cadmium surface characteristics, (cadmium granules should be dark gray).

b. If air bubbles are present in column, connect the column into the manifold, turn the pump on maximum and tap firmly with a screwdriver handle, being careful not to break the column, working up the column until all air is removed. If air cannot be removed, the column should be repacked. Cadmium columns should be stored filled with buffer. If air enters the column, efficiency will decrease.

c. Check the flow efficiency by disconnecting the cadmium column from the manifold and reconnecting to a green pump tube. Pump buffer through the packed column and collect in a graduated cylindar. The flow rate with the column connected should be greater than 4.0 mL/min.

d. There are two procedures for determining column efficiency, as follows:

Slope Ratio Method

1. Calibrate with the mid-range NO_3-N standards
2. Calibrate with a matching concentration range of NO_2-N standards
3. The column efficiency is determined by the equation:

$$\frac{S_{NO3\text{-}N}}{S_{NO2\text{-}N}} \times 100 = E$$

$S_{NO3\text{-}N}$ = slope of NO_3 calibration
$S_{NO2\text{-}N}$ = slope of NO_2 calibration
E = % efficiency

4. If the efficiency is <90%, the column should be repacked

Concentration Ratio Method

1. Calibrate with the mid-range NO_2-N or NO_3-N standards
2. Run a known concentration NO_2-N standard
3. Run a matching concentration NO_3-N standard
4. The column efficiency is determined by the equation:

$$\frac{C_{NO3\text{-}N}}{C_{NO2\text{-}N}} \times 100 = E$$

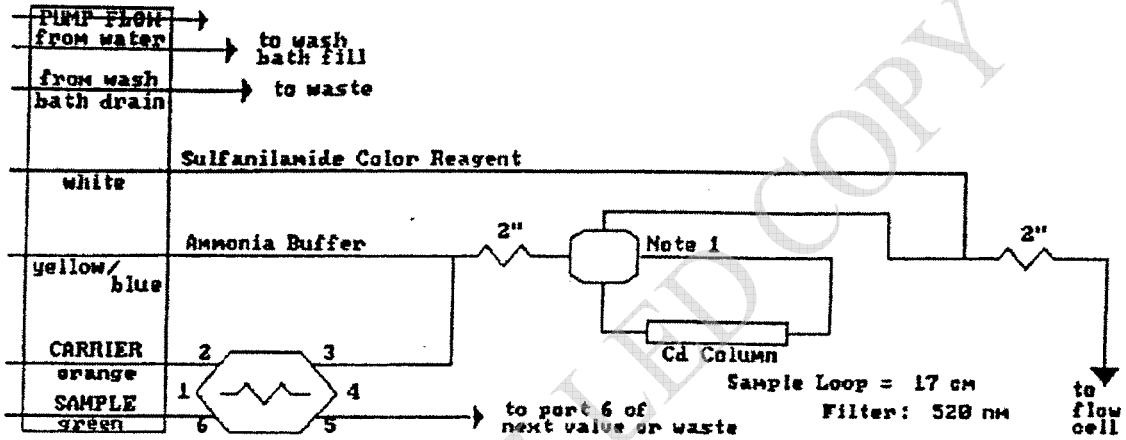
$C_{NO3\text{-}N}$ = concentration of NO_3 standard
$C_{NO2\text{-}N}$ = concentration of NO_2 standard
E = % efficiency

5. If the efficiency is <90%, the column should be repacked

Attachment 23.3
Manifold Diagram

QuikChem Method 10-107-04-1-C Nitrate/Nitrite Page 10

Manifold Diagram:



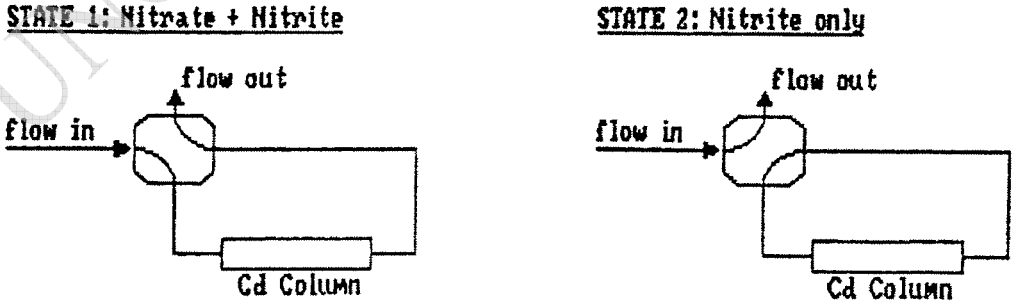
CARRIER is water.

2" is 135 cm of tubing on a 2 in coil support

APPARATUS: Standard valve, flow cell, and detector head modules are used.

All manifold tubing is 0.8 mm (0.032 in) i.d. This is 5.2 uL/cm.

Notes:
Note 1: This is a 2 state switching valve used to place the cadmium column in-line with the manifold.



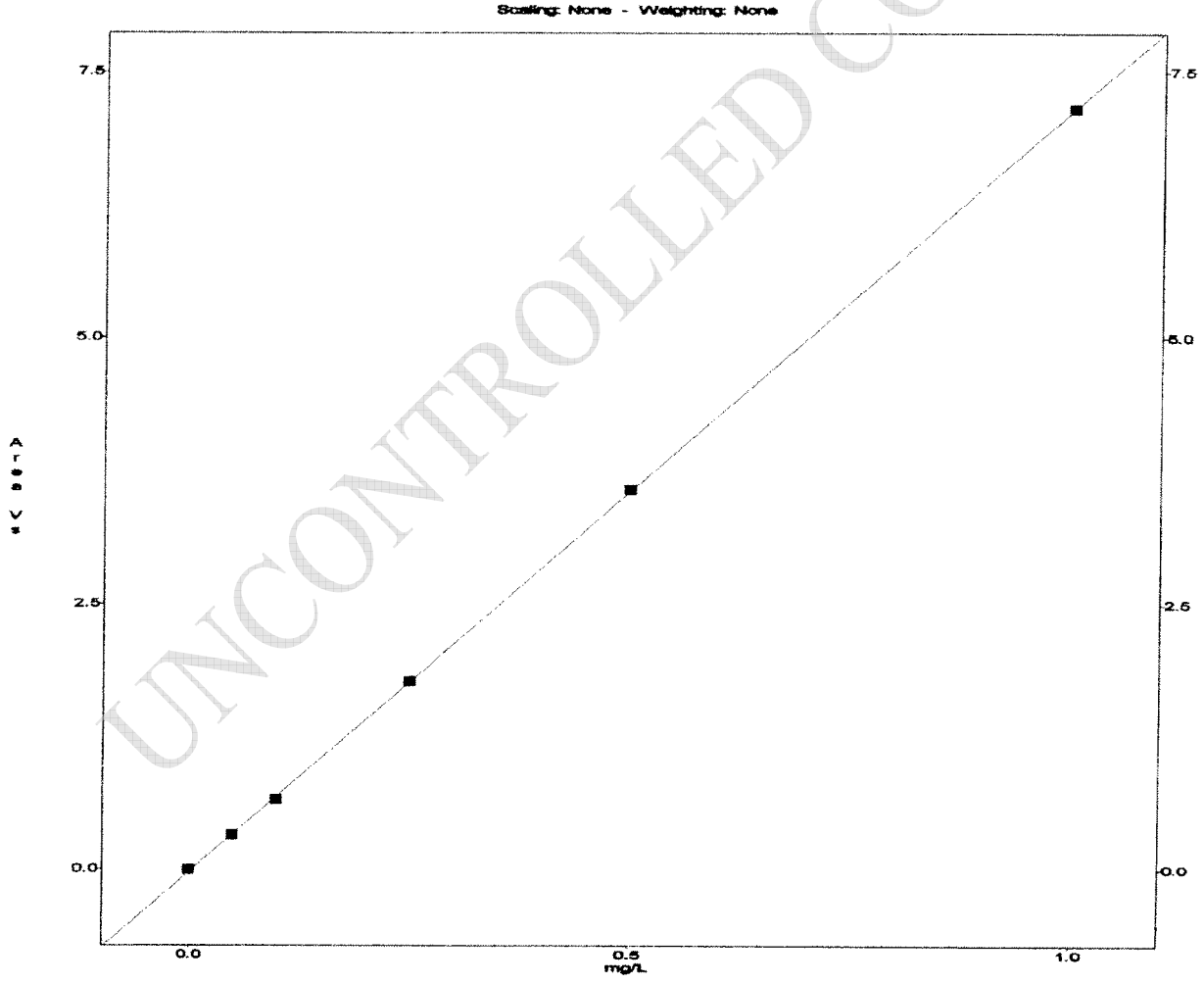
MANIFOLD DIAGRAM REVISION DATE: 4 November 1992 AS

Attachment 23.4
Calibration Example

Nitrite

Lvl	Area	mg/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
7	7161014	1.00	7161014					0.0	0.0	0.0
8	3579547	0.50	3579547					0.0	0.0	-0.3
9	1778104	0.25	1778104					0.0	0.0	-0.3
10	661353	0.10	661353					0.0	0.0	4.5
11	328138	0.05	328138					0.0	0.0	1.7
12	0	0.00	0					0.0	0.0	

1st Order Poly
Conc = 1.391e-007 Area + 3.529e-003
r = 1.0000



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Approved By: 1-23-09 QA Officer

Approved By: Heather L. Brady Area Supervisor

Attachment 23.5
Instrument Logbook Example

TriMatrix Laboratories, Inc.

Instrument Number 189 Run Logbook

Date	Analyst	Method Name or Number	Concentration of Calibration Standards Used							Units	Client Names and Sample Numbers
			No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7		
5/11/01	JW	NO_3/NO_2	1.0	0.5	0.25	0.10	0.05			mg/L	[redacted] 37343-500
			Calibration Requirements			≥ 0.995 ✓	Calibration Results			NO_3: 0.997 / NO_2: 0.999	
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				
			Calibration Requirements				Calibration Results				

file: instlogbook page: 1 of 50 revision: 2.0

Approved By: QA 1-23-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.6
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH **0702807** Page 1 of 1

Printed: 1/23/2009 9:46:47AM

Inorganic - Wet Chemistry, Waste Water, Method-Specific Preparation

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0703181	Nitrogen, NO3 353.2	0703183	Nitrogen, NO3 4500-NO3 F	0703183	Nitrogen, NO3 353.2

Lab Number	*Contain*	*Prepared*	*By*	*Initial (mL)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client QC Type*	*Extraction Comments*
0702807-BLK1		Mar-13-07 14:00	HLB	10	10					BLANK	
0702807-BS1		Mar-13-07 14:00	HLB	10	10			6110794	10000	LCS	
0702807-MS1		Mar-13-07 14:00	HLB	1	10		0703183-01	6110796	50	MATRIX SPIKE	[Spk] 10mL->10mL; 10mL->100mL; Spi
0702807-MSD1		Mar-13-07 14:00	HLB	1	10		0703183-01	6110796	50	MATRIX SPIKE DUP	[Spk] 10mL->10mL; 10mL->100mL; Spi
0703181-02	D	Mar-13-07 14:00	HLB	10	10					[redacted]	
0703183-01	A	Mar-13-07 14:00	HLB	10	10					[redacted]	this is NO2 and NO3 (plus ammonia)
0703183-01	A	Mar-13-07 14:00		10	10					[redacted]	Added for BatchQC in: 0702807

Approved By: _____ 1-23-09 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Closed System Purge and Trap Extraction for Volatile Organic Compounds

SW-846 Method 5035A

APPROVALS:

Area Supervisor: Diane VanMale Date: 1-23-09
Diane L. VanMale

QA Officer: Date: 1-22-09
Tom C. Boocher

Operations Manager: Date: 1/26/09
Jeff P. Glaser

Procedure Number: GR-04-105
Revision Number: 1.2

Date Initiated: 4/30/98
Effective Date: 2/20/09

Date Revised: 1/22/09
Pages Revised: All

By: Diane L. VanMale
Total Number of Pages: 20

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-16-10		4-16-11

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the preparation of soils, sediments and solid waste for volatile organic compounds (VOC) analysis. The procedure includes preparation of low level, high level and oily waste.

1.2 The low level soil procedure is designed to use a hermetically-sealed 40 mL sample vial, preserved with sodium bisulfate which is sealed at the time of sample collection. Since the sample is never exposed to the atmosphere after sampling, VOC losses during shipment, storage and analysis are minimized. The low level soil procedure also details steps for collecting a sample in an appropriate storage device which can then be shipped to the laboratory where it is preserved upon receipt. The applicable concentration range for low level samples is 0.001 to 0.200 mg/kg.

1.3 The high level soil procedure is based upon preserving the sample with methanol at the time of collection or upon receipt at the laboratory. It is applicable to soil samples with VOC concentrations over 0.200 mg/kg. The closed system purge and trap employed for low concentration samples is NOT appropriate for samples preserved in the field with methanol. All methanol preserved samples must be analyzed by diluting an aliquot of the methanol into laboratory reagent water then analyzing in accordance with TriMatrix standard operating procedure (SOP) GR-04-104, GR-03-105 or GR-03-121.

1.4 This procedure can be used for most volatile organic compounds with boiling points below 200° C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, reporting limits are approximately ten times higher because of poor purging efficiency.

1.5 Refer to the appropriate analytical standard operating procedure for a target analyte list and the analysis procedure.

1.6 Low level bulk soil analysis is outside of the scope of this procedure and is no longer formally recognized by the EPA as an acceptable option for VOC analysis. However, since some states and clients still require bulk soil sample collection and analysis, the technique is included in Attachment 3.0 (Bulk Soil Collection and Preparation Technique).

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, New Methods*, Method 5035A, "Closed system Purge and trap and Extraction for Volatile Organics in Soil and Waste Samples", Draft Revision 1, July 2002

3.0 SUMMARY OF PROCEDURE

3.1 Low concentration soil procedure - Applicable to soil and other solid samples with VOC concentrations in the range of 0.001 to 0.2 mg/kg. The VOCs are determined by collecting approximately 5 g of sample and shipping it to the laboratory to be analyzed, or preserved and then analyzed. One of the following procedures must be used for sampling and preparation.

3.1.1 Collect approximately 5 g of sample by using a metal or plastic coring tool, weighed in the field at the time of collection. Place the coring in a pre-weighted 40 mL VOA vial with a septum-sealed screw cap that already contains a stir bar. The vial is then sealed and shipped to the laboratory. The sample must then be frozen within 48 hours of collection. Another option is to preserve the sample at the time of collection with sodium bisulfate preservative solution. The vial is then sealed and shipped to the laboratory where it is stored in the refrigerator until time of analysis. For analysis, the entire vial is allowed to come to room temperature, and is then placed unopened into the autosampler.

3.1.2 Alternatively, samples may be collected in the field using an appropriate coring device which can also serve as the storage device for shipping such as the EnCore™ sampler. Constructed of an inert polymer, the EnCore™ has a coring/storage chamber designed to collect a 5 g sample with a press-on cap that creates a vapor tight seal to prevent analyte loss. After collection, the sample is shipped to the laboratory where it is transferred to a new pre-cleaned and tared 40 mL VOA vial containing a stir bar. The vial is then sealed and weighed, and the weight recorded. At this point the entire vial may be placed in the freezer until time of analysis, or the appropriate volume of sodium bisulfate preservative solution (1:1 w/v) may be added, and the sample stored in the refrigerator until time of analysis. As in Section 3.1.1, the entire vial is placed unopened into the autosampler for analysis.

3.1.3 Regardless of collection technique and immediately before analysis, laboratory reagent water, surrogates and internal standards (if applicable) are automatically added without opening the sample vial. The vial is then heated to 45° C, magnetically stirred and volatiles are purged into an appropriate trap using helium gas. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped volatile analytes into a gas chromatograph (GC) for analysis by the appropriate detector.

3.2 High concentration soil procedure - Applicable to soil and other solid samples with VOC concentrations greater than 0.2 mg/kg. The low concentration technique is not applicable to all samples. Particularly those containing high concentrations of VOCs which may overload the trap or exceed the calibration range. In such instances, use one of the procedures described below.

3.2.1 The first option is to collect a bulk sample in a glass container with a PTFE-lined lid. A 5 g portion of the collected sample is removed from the container at the laboratory and added to 5.0 mL of methanol to extract volatile organic constituents. After mixing by shaking or agitating using an ultrasonic bath, a volume of the solvent extract is quantitatively diluted into laboratory reagent water and placed in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

Note: This is the least desirable option as the sample contacts the atmosphere which may cause some volatile constituent losses.

3.2.2 The second option is to collect approximately 10 g of sample in a pre-weighed vial with a septum-sealed screw cap that contains 10 mL of methanol for extraction. For analysis, a volume of the solvent extract is quantitatively diluted into laboratory reagent water as in Section 3.2.1 and placed in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if

applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

3.2.3 The third option is to use a coring/storage device, such as the EnCore™ sampler described in Section 3.1.2. The EnCore™ storage chamber size can be 5 g or 25 g. Immediately after a sample is collected, seal the device and ship to the laboratory. Upon receipt, the laboratory will extrude the sample into a pre-tared 60 mL jar, weigh the sample and add methanol to the container in a 1:1 ratio of solvent volume to sample mass. At analysis, dilute an aliquot of the methanolic extract into laboratory reagent water as in Section 3.2.1 and transfer directly to a purge and trap vessel. Surrogates and internal standards (if applicable) are added by the before purging and analysis in accordance with the appropriate analytical procedure.

3.3 High concentration oily waste procedure - Applicable to oily samples with VOC concentrations greater than 0.2 mg/kg that can be diluted in a water-miscible solvent (not necessarily methanol). After demonstrating that a test aliquot is soluble in the solvent of choice, dilute a separate aliquot into the solvent. At analysis, dilute an aliquot of the water-miscible solvent extract into laboratory reagent water as in Section 3.2.1 and place in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

Note: Oily samples not soluble in a water-miscible solvent are beyond the scope of this procedure and must be analyzed by direct injection analysis after dilution in n-hexadecane.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to each analytical procedure for the appropriate parameters list.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-104, *Volatile Organic Compounds By Purge And Trap Capillary Column Gas Chromatography/Mass Spectrometry*, latest revision

5.2 TriMatrix SOP GR-03-105, *Volatile Organic Compounds By Purge And Trap Capillary Column Gas Chromatography With Photoionization And Electrolytic Conductivity Detectors in Series*, latest revision

5.3 TriMatrix SOP GR-03-121, *Method for the Determination of Gasoline Range Organics*, latest revision

5.4 TriMatrix SOP GR-03-124, *Volatile Organic Laboratory Corrective Actions*, latest revision

5.5 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.6 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Sample contamination can cause elevate reporting limits or give false positives and come from a variety of sources. Improper sampling can contaminate samples at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through the septum. A trip blank prepared from laboratory reagent water containing the sodium bisulfate preservative and carried through all sampling and handling serves as a check on contamination for the low level procedure. For the methanol preservation technique, a trip blank must be prepared using purge and trap grade methanol which must then be processed through all sample preparation and analysis steps.

6.2 To minimize contamination, the volatiles analysis laboratory must be free of solvents. The volatiles sample storage area must be isolated from atmospheric sources of methylene chloride and other solvents. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing must be stainless steel or copper. Clothing previously exposed to methylene chloride fumes during semi-volatiles extraction can contribute to sample contamination and must not be worn into the volatiles laboratory.

6.3 The sodium bisulfate preservative acts to lower sample pH which inhibits biological degradation of aromatic compounds. However, under acidic conditions, highly reactive compounds such as 2-Chloroethylvinyl ether are unstable and will be lost. Acid preservation can also degrade methyl-t-butyl ether (MtBE) to tert-butyl alcohol during purge and trap. Additional analytes affected include vinyl chloride, styrene and other fuel oxygenate ethers. If these analytes are to be reported, collect a second sample set without sodium bisulfate preservative. Acidification of certain soil types with sodium bisulfate can produce an acetone artifact which is typically between 0.025 and 0.100 mg/kg.

6.4 For the appropriate corrective actions when encountering contamination, refer to TriMatrix SOP GR-03-124.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity or carcinogenicity of reagents used in this procedure has not been precisely defined. However, each chemical must be treated as a potential health hazard and exposure must be reduced to the lowest possible level by all means available.

7.3 The laboratory maintains a current reference file of material safety data sheets (MSDS) available to all personnel.

7.4 Many of the analytes tested have been classified as known or suspected carcinogens. Consequently, all neat standard materials and standard solutions must be opened only in a fume hood.

7.5 Appropriate personal protective equipment including a laboratory coat, approved safety glasses and disposable gloves must be worn in the laboratory when handling any chemical. The only exception for wearing personal protective equipment is when working at a computer station in the laboratory.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 The specific sample container required depends on the purge and trap system. TriMatrix Laboratories currently uses the needle sparge type purging device for all low level soils. This system requires a 40 mL borosilicate glass volatile organic analysis (VOA) vial.

8.2 The specific preparation for a sample vial depends on the expected concentration range with specific preparation techniques for low concentration soils, high concentration soils and solid waste. Sample vials must be prepared in the laboratory or other controlled environment then sealed and shipped to the collection site. Disposable gloves must be worn during the preparation steps which are as follows:

8.2.1 The following instructions apply to the preparation of vials used in the collection of low concentration soil samples that are to be preserved in the field.

8.2.1.1 Add a clean magnetic stirring bar to each clean vial.

8.2.1.2 Add 5.0 mL of 20% sodium bisulfate preservative solution to each vial. This should be sufficient to ensure a sample pH of <2 to prevent biodegradation.

8.2.1.3 Seal the vial with the screw-cap septum lid.

8.2.1.4 Affix a label to each vial. This eliminates the need to label vials in the field and assures that the vial tare weight is included on the label.

8.2.1.5 Weigh the prepared vial to the nearest 0.01 g, record the tare weight and write it on the label.

8.2.1.6 Because volatile organics will partition into the vial headspace from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes and internal standards (if applicable) must only be added after sample has been added. Surrogates, matrix spikes and internal standards (if applicable) must be injected automatically by the autosampler just prior to analysis.

8.2.2 When high concentration samples are collected without a preservative, a variety of sample containers may be employed including 40, 60 and 125 mL glass jar with a PTFE liner. This collection technique must only be used when sample solubility in methanol is questionable.

8.2.3 The following steps apply for high concentration soil samples collected and preserved in the field with methanol as described in Section 3.2.

8.2.3.1 Use pre-tared 40, 60 or 125 mL jars with screw cap lids and PFTE liners.

8.2.3.2 To obtain the pre-tared weighing, weigh the container with the label, lid and liner in place then record the tare weight on the label to the nearest 0.01 g. The mass of the ink is negligible.

8.2.3.3 Include a purchased 10 mL ampule of purge and trap methanol with each sample container.

8.2.4 When oily waste samples are known to be soluble in methanol, sample containers may be used as described in Section 8.2.3, preserving with methanol. However, when methanol solubility is unknown, collect the sample without preservative in a 40, 60 or 125 mL glass jar with a PTFE liner.

8.2.5 Preservation by freezing to less than -7° C may be used unless otherwise specified and is as follows:

8.2.5.1 If freezing at collection, add sample to an empty sample container and freeze to less than -7° C until analysis.

Note: When freezing, always place the sample container on its side to prevent the glass container from breaking.

8.2.5.2 Samples received by the laboratory unfrozen and at 4 ±2° C, and are within 48 hours of collection may be frozen to less than -7° C at the time of receipt.

8.2.5.3 Samples must be thawed for preparation and analysis within 14 days of collection.

8.2.5.4 Thawed samples must be prepared and analyzed within 24 hours of thawing.

8.3 Sample Collection

8.3.1 Collect samples according to the procedure outlined in the project sampling plan. In general, after a fresh surface of the solid material is exposed, collect a subsample in the least amount of time and with as little disruption as possible to minimize the loss of volatiles. Use a metal or rigid plastic coring tool to collect the subsample. These devices help maintain sample structure during collection and transfer to the sample container. When inserting the coring device into solid material, use care not to trap air behind the sample. If air is trapped, it can cause VOC loss by passing through the sample or cause the sample to be pushed from coring device before a transfer can be made to the sample container. The following devices have been designed to prevent headspace during collection and are approved for sampling:

8.3.1.1 EasyDraw Syringe™ with Powerstop Handle™

8.3.1.2 Terra Core™

8.3.1.3 EnCore™

8.3.2 Low level soil samples may be collected as follows:

8.3.2.1 Collect approximately 5 g of soil using a coring device as described in Section 8.3.1. After taking the sample, remove the filled corer and quickly wipe the barrel with a clean disposable wipe.

8.3.2.2 If a sample is to be preserved at collection, add to a sample jar containing sodium bisulfate preservative by holding the sample jar at an angle then extrude into the

container to minimize splashing. Quickly brush or wipe the vial threads to remove residual soil and immediately seal the vial tightly with the septum screw cap.

Note: An improper seal due to soil remaining on the sample jar threads and improper lid tightening are the primary factors in volatiles loss during sample collection. This can also cause problems during analysis by preventing the vial from pressurizing during the purge step, resulting in low or no recovery.

8.3.2.3 If a sample is shipped to the laboratory for preserving, cap the open end of the coring/storage device after ensuring all sealing surfaces are clean then store and transport at 4 ±2° C.

Note: An individual EnCore™ is needed for each sample aliquot.

8.3.2.4 If samples are expected to contain target analytes over a wide concentration range and high level samples are not being collected, collect an additional sample aliquot in a low concentration preserved VOA vial with 1 - 2 g rather than 5 g. If necessary, the low mass sample can be analyzed for analytes in the 5 g sample that exceed the calibration range.

8.3.2.5 Samples that contain carbonate minerals may effervesce upon contact with the sodium bisulfate preservative. If the gas volume generated is small, volatiles loss may be minimal. However, if large gas volumes are generated, volatiles are likely to be lost and the vial may rupture. If carbonate minerals are expected, collect a test sample to check for effervescence. If the sample effervesces when sodium bisulfate is added, discard the test sample and collect additional sample without preserving.

8.3.3 High level soil samples may be collected as follows:

8.3.3.1 Preserve in the field as follows:

8.3.3.1.1 Use a coring device to collect a 5 g or 25 g sample as soon as possible after solid surface material is exposed. Wipe the exterior of the collection device with a clean disposable wipe.

8.3.3.1.2 Quickly transfer to a 40 mL vial for 5 g, or to a 60 or 125 mL sample jar for a 25 g sample.

8.3.3.1.3 Add a volume of methanol in a 1:1 ratio of methanol volume to sample mass.

8.3.3.2 Preserved in the laboratory as follows:

8.3.3.2.1 If a sample is to be preserved in the laboratory, collect in an EnCore™ sampler, cap the open end after ensuring all sealing surfaces are clean and store at 4 ±2° C until transport.

8.3.3.2.2 Upon receipt at the laboratory, the sample must be extruded from the EnCore™ into a new sample jar then weighed and a 1:1 ratio of methanol volume to sample mass added. Refer to Section 13.0 for detailed instructions.

8.4 Sample Storage

8.4.1 Once in the laboratory, store samples at 4 ±2° C (or freeze to -7° C within 48 hours of collection) until analysis. The volatiles sample storage area must be free of organic solvent vapors.

8.4.2 All samples must be analyzed as soon as practical and within the designated holding time. Samples not analyzed within the holding time must be reported as exceeding the holding time. Results must be reported as estimated minimum values.

8.4.4 When low concentration samples are strongly alkaline or highly calcareous, the sodium bisulfate preservative solution may not be strong enough to reduce pH to below 2.

8.4.4.1 When low concentration soils are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required for preservation. Such steps include:

8.4.4.1.1 Addition of a larger sodium bisulfate preservative volume

8.4.4.1.2 Storage at below -10° C (taking care not to fill the vial it breaks from expansion)

8.4.4.1.3 Significantly reducing the maximum holding time.

8.4.4.2 The preservation used must be clearly described in the sampling and quality assurance (QA) project plan for distribution to both field and laboratory personnel.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Sample containers are as follows:

9.1.1 Vials, 20 or 40 mL with PTFE-lined septum-sealed screw cap lids

Note: Examine each vial prior to use to ensure the vial has a flat, uniform sealing surface.

9.1.2 Glass borosilicate jars, 60 or 125 mL with PTFE-lined screw cap lids

9.2 EnCore™ sampler (EnNovative Technologies, Inc.)

9.3 microSyringes, 100 µL and 1000 µL

9.4 Balance, top-loading, capable of accurately weighing to 0.01 g

9.5 Disposable Pasteur pipets

9.6 Magnetic stirring bars, PTFE of the appropriate sizes to fit sample containers.

Note: Stirring bars may be reused provided they are thoroughly cleaned between each use.

9.7 Field equipment is as follows:

9.7.1 Terra Core™ sampler, EnNovative Technologies, Inc.

9.7.2 EasyDraw Syringe™ with Powerstop Handle™, US Oil Company

9.7.3 Portable balance, capable of accurately weighing to 0.01g

9.8 Volumetric flask, 500 mL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no routine preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, organic-free

11.2 Methanol (CH_3OH), purge and trap grade

Note: Store away from other solvents

11.3 The low concentration sample preservative is prepared as follows:

11.3.1 Sodium bisulfate ($NaHSO_4$), certified

11.3.2 Weigh 100 g of $NaHSO_4$ into a 500 mL volumetric flask. Mix to dissolve the $NaHSO_4$ then bring to volume with laboratory reagent water (organic-free). Record the preparation in the laboratory information system (Element™) and/or in the reagent preparation logbook. Concentration is 20% (w/v) $NaHSO_4$.

11.3.3 Expiration is 6 months from the date prepared.

11.4 Refer to the appropriate analytical procedure for guidance on preparing internal standards and/or surrogates.

12.0 STANDARDS PREPARATION

12.1 There are no analytical standards directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 Prepare low level soils collected with an Encore sampler (5 g) as follows:

13.1.1 Place a 40 mL vial on the top-loading balance and tare with the cap off.

13.1.2 Empty the 5 g Encore sample into the vial.

13.1.3 Weigh to the nearest 0.01 g and record on the vial label.

13.1.4 Add the equivalent volume of 20% $NaHSO_4$ to the vial to achieve a 1:1 ratio of sample mass to preservative volume.

13.1.5 Add a clean magnetic stir bar to the vial and seal the vial.

13.1.6 Remember to attach the label to the vial.

Note: **Sodium bisulfate degrades 2-chloroethylvinyl ether. TriMatrix Laboratories has demonstrated that this preservative will not give acceptable quality control recovery so 2-chloroethylvinyl ether will not be reported when used.**

13.2 Prepare high level soils collected with an Encore sampler (25 g) as follows:

13.2.1 Place 60 mL sample jar on the top-loading balance and tare with the cap off.

13.2.2 Empty the 25 g Encore sample into the jar

13.2.3 Weigh to the nearest 0.01 g and record on the jar label.

13.2.4 Add the equivalent volume of methanol to the vial to achieve a 1:1 ratio of sample mass to preservative volume then seal the jar.

13.2.6 Remember to attach the label to the jar.

13.2.7 Shake the sample jar for 2 minutes. Then sonicate the jar for 20 minutes.

13.2.8 After shaking and sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.2.9 After settling, carefully decant enough methanol extract to fill a 4 mL vial without headspace and seal with the septum screw cap. Store at 4 ±2° C until analysis.

13.3 Prepare high level soils pre-preserved in the field as follows:

13.3.1 Weigh the sample jar containing soil sample and methanol, and record on the label.

13.3.2 Enter the sample jar size and mass into the Volatile Soil Receipt Information spreadsheet located on the laboratory intranet library (Attachments 1 and 2).

13.3.4 The spreadsheet determines if additional methanol needs added and/or whether the sample can be used, as follows:

13.3.4.1 If the mass collected in ratio with the methanol added is less than a 1:1 ratio of sample to preservative, consult the project chemist. The sample must be reported with a low weight narration.

13.3.4.2 If the mass collected requires additional methanol that exceeds the sample jar volume, consult the project chemist. The sample may be rejected for analysis.

13.3.4.3 If the mass collected in ratio with the methanol added is greater than a 1:1 ratio of sample to preservative, add the appropriate methanol volume.

13.3.5 Shake the sample jar for 2 minutes. Then sonicate the sample jar for 20 minutes.

13.3.6 After shaking and sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.3.7 After settling, carefully decant enough methanol extract to fill a 4 mL vial without headspace and seal with the septum screw cap. Store at 4 ±2° C until analysis.

13.4 Prepare oily waste samples soluble in a water-miscible solvent as follows:

13.4.1 Place a 20 mL vial on the top-loading balance and tare with the cap off.

13.4.2 Weigh out 1 g of sample to the nearest 0.01 g.

13.4.3 Add 10 mL of water-miscible solvent and quickly cap the vial.

13.4.4 Shake for 2 minutes. After shaking or sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.4.5 After settling, carefully decant enough of the mixture to fill a 4 mL vial without headspace and seal with the septum screw cap. A 1:50 dilution in the water-miscible solvent will be made unless the analyst suspects higher volatile compound concentrations.

Note: Oily samples that are not soluble in a water-miscible solvent are beyond the scope of this procedure.

14.0 CALIBRATION PROCEDURES

14.1 There are no calibration procedures directly associated with this procedure.

15.0 ANALYTICAL PROCEDURE

15.1 Analyze a low level soil as follows:

15.1.1 Remove the sample vial from storage and bring to room temperature. Shake the vial gently, to ensure the contents move freely and that stirring will be effective. Place the sample vial in the autosampler.

15.1.2 For matrix spiked samples, add the matrix spiking solution described in the appropriate analytical procedure. The spiking solution concentration and the volume injected will vary.

15.1.3 Perform the qualitative and quantitative analysis in accordance with every step in the analytical procedure.

15.1.4 If any target analyte concentration exceeds the calibration range, reanalyze by the high concentration procedure. Reanalysis need only address analytes exceeding the calibration range.

15.1.5 Alternatively, analyze the 1-2 g sample aliquot if collected (Refer to Section 8.2.2.7). Reanalysis need only address analytes exceeding the calibration range in the 5 g sample analysis.

15.2 Analyze a high level soil or waste as follows:

15.2.1 Pipet 1.0 mL of solvent extract or diluant to a 50 mL volumetric flask approximately ¾ full of laboratory reagent water (organic-free). Dilute to volume with laboratory reagent water organic-free) and invert 3 times to mix. Transfer to a 40 mL vial and place on the autosampler for analysis.

15.2.2 For matrix spiked samples, add the matrix spiking solution described in the appropriate analytical procedure. The spiking solution concentration and the volume injected will vary.

15.2.3 Perform the qualitative and quantitative analysis in accordance with every step in the analytical procedure.

15.2.4 If any target analyte concentration exceeds the calibration range, reanalyze by diluting a lesser amount of the solvent extract into a 50 ml volumetric flask. Reanalysis need only address analytes exceeding the calibration range.

16.0 CALCULATIONS AND DATA HANDLING

16.1 There are no analytical calculations directly associated with this procedure.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Volatiles Soil Receipt Information Spreadsheets are to be archived as raw data.

17.2 Refer to the analytical procedure for complete data reporting and deliverable requirements.

18.0 QUALITY ASSURANCE

18.1 Refer to the analytical procedure for complete quality control/quality assurance requirements.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Refer to the analytical procedure for demonstration of capability study and instrument validation requirements.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, New Methods*, Method 5035A, "Closed system Purge and trap and Extraction for Volatile Organics in Soil and Waste Samples", Draft Revision 1, July 2002

23.0 ATTACHMENTS

23.1 Volatile Soil Sample Receipt Information Spreadsheet, Page 1

23.2 Volatile Soil Sample Receipt Information Spreadsheet, Page 1

23.3 Bulk Soil Collection and Preparation

Attachment 23.1
Volatile Soil Sample Receipt Information Spreadsheet, Page 1

TriMatrix Laboratories, Inc.

Volatile Soil Sample Receipt Information Sheet

Client: _____ Date Received: 08/29/2003 Sheet Completed By: JDM

Project-Submittal: _____ Date Form Completed: 09/03/2003 Sheet Reviewed By: _____

Low Level Soils	Yes	No	N/A
Were Samples Received in 40 mL VOA Vials Containing a Stir Bar and Pre-Preserved with Sodium Bisulfate?	☐	☐	☐
Were Samples Received Non-Preserved in Encore Samplers?	☐	☐	☐
If Received in Encore Samplers, Was Sample Received Within 48 Hours?	☐	☐	☐
Volatile Lab Informed That Samples Must Be Preserved AND Analyzed Within 48 Hours?	☐	☐	☐

High Level Soils	Yes	No	N/A
Extra Sample Containers Received for Dry Weight Determination?	☐	☐	☐
MeOH Trip Blank Received (if MeOH not added from sealed ampules)?	☐	☐	☐
Samples Collected in Which of the Following Ways?			
Tared 40, 60, or 120 mL Containers:			
Were Containers Supplied by TriMatrix?	☐	☐	☐
Were Samples Received Within 4 Days After Collection, and Were They MeOH Preserved?	☐	☐	☐
Packed With No Headspace into Brass Tubes:			
Were Samples Received Within 4 Days After Collection, and Preserved Within 2 Hours of Sample Receipt?	☐	☐	☐
Packed With No Headspace in En Core Samplers:			
Were Samples Received Within 40 Hours After Collection and Preserved Within 48 Hours of Collection?	☐	☐	☐

Follow-Up	Yes	No	N/A
Has Soil Weight Table Been Completed?	☐	☐	☐
Has Project Chemist Been Informed of any Out-of-Compliance Samples?	☐	☐	☐

0903C xls page #: 1 of 2 revision 0.0

Approved By: QA 1-22-09 **QA Officer**

Approved By: DW 1-23-09 **Area Supervisor**

Attachment 23.2
Volatile Soil Sample Receipt Information Spreadsheet, Page 2

TriMatrix Laboratories, Inc.

Volatile Soil Sample Receipt Information Sheet

Client: _____ Date Received: 08/29/2003 Sheet Completed By: JDM

Project-Submittal: _____ Date Form Completed: 09/03/2003 Sheet Reviewed By: _____

Client Sample Identification	TriMatrix Sample Number	Vial Size: 40, 60, or 120 mL	Empty Weight of Bottle (g)	MeOH Added: 10, 25, or 50 mL	Full Weight of Bottle (g)	Date Sample Weighed	Time Sample Weighed	Weight of Soil (g)	Low Wt. Flag Required?	Additional MeOH Required?	Accept/ Reject Sample	MeOH to Add (mL)	mL MeOH Added: By, Date, Time
1	341527	40	25.5	10	43.6			10.2			Accept Sample		
2	341528	40	25.6	10	43.6			10.1			Accept Sample		
3	341543	40	25.6	10	43.4			9.9			Accept Sample		
4	341544	40	25.3	10	43.0			9.8			Accept Sample		
5	341546	40	25.6	10	43.7			10.2			Accept Sample		
6	341547	40	25.3	10	43.3			10.1			Accept Sample		
7	341548	40	25.3	10	43.3			10.1			Accept Sample		
8	341549	40	25.3	10	43.4			10.2			Accept Sample		
9	341550	40	25.4	10	43.9			10.6			Accept Sample		

Approved By: _____ 1-22-09 **Approved By:** _____ 1-23-09

QA Officer **Area Supervisor**

Attachment 23.3
Bulk Soil Collection and Preparation

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the collection and preparation of bulk soils and sediments for volatile organic compounds (VOC) analysis. Low level bulk soil analysis is no longer formally recognized by the EPA as an acceptable option for VOCs. However, some states and/or clients still request bulk soil sample collection and analysis.

2.0 SUMMARY OF PROCEDURE

2.1 Bulk soil samples are collected with minimal headspace in either 60 or 125 mL glass jars. After collection, samples must be stored at 4 ±°2 C until transport to the laboratory.

2.2 When received by the laboratory, samples remain stored at 4 ±°2 C until the analyst transfers a 1-5 g subsample to a new 40 mL vial containing a stir bar for preparation.

2.3 The vial is then loaded onto the autosampler for analysis.

3.0 INTERFERENCES AND CORRECTIVE PROCEDURES

3.1 Bulk soil sampling is inaccurate and produces results that are biased low. Bulk samples can lose more than 90% VOC content prior to analytical measurement.

3.2 Reasons for such losses include the following:

3.2.1 Volatilization from exposure of the solid surface near the time of collection

3.2.2 Volatilization from intermediate storage containers

3.2.3. Volatilization from the disaggregation of the solid material during collection

3.2.4 Volatilization from failed seals on sample jar lids

3.2.5 Volatilization during laboratory subsampling

3.2.6 Biodegradation (primarily aromatic compounds) during storage

3.2.7 Chemical reactions during storage

4.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

Attachment 23.3 (continued)
Bulk Soil Collection and Preparation

4.1 Solid samples are collected using a stainless steel spatula-type device to completely fill a 40, 60 or 125 mL glass jar.

4.2 The sample container is then closed using PTFE-lined caps and stored at 4 ±2° C with ice throughout the collection event, during transport to the laboratory and during storage at the laboratory until just prior to analysis.

4.3 The holding time for samples is 14 days from the time of collection.

4.4 If samples require high level preparation/analysis based on low level results or the sample matrix, a subsample must be extracted using methanol.

4.5 The holding time for a methanol extract is also 14 days from the time of collection.

5.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

5.1 Refer to Section 9.0 in the main body of the procedure.

5.0 SAMPLE PREPARATION

6.1 Prepare a low level sample as follows:

6.1.1 From the original sample container, remove a representative subsample and transfer to a tared 40 mL vial containing a PTFE stir bar. Record the weight to the nearest 0.01 g. Use a 5 g sample unless higher level concentrations are expected and a smaller aliquot is needed.

Note: Do not use less than a 1 g sample aliquot.

6.1.2 Quickly wipe any residual soil from the vial threads and seal then load onto the instrument autosampler. The autosampler will add 10 mL of laboratory reagent water (organic-free) and applicable internal standards and/or surrogates immediately prior to the purge cycle.

6.2 Prepare a high level sample as follows:

6.2.1 If low level analysis results are outside the instrument calibration range or if the sample matrix indicates an unsuitability for low level analysis, the sample requires high level preparation and analysis.

6.2.2 From the original sample jar, remove a representative subsample and transfer to a tared 20 mL sample vial. Weigh out 5 g to the nearest 0.1 g and record on the label. Remove any residual

Attachment 23.3 (continued)
Bulk Soil Collection and Preparation

sample from the vial threads and pipet in 5.0 mL of methanol. Cap and shake the container by hand for 2 minutes. Place in the refrigerator at 4 ±2° C to settle out.

Note: Some samples may require letting stand overnight for sufficient phase separation.

6.2.3 Once the phases separate, decant the methanol phase into a new 4 mL vial for storage. Store at 4 ±2° C until analysis.

Note: The methanol must be withdrawn from the sample within 24 hours of extracting the sample to keep the extraction times constant.

7.0 CHEMICALS AND REAGENTS

7.1 Refer to Section 11.0 in the main body of the procedure.

8.0 ANALYTICAL PROCEDURE

8.1 Once prepared, low level soils can be directly loaded onto the purge and trap autosampler.

8.2 The autosampler will add laboratory reagent water (organic-free), internal standards and surrogates.

8.3 If the results of the analysis are outside the calibration range, reanalyze as a high level sample as follows:

8.2.1.1 Pipet 1.0 mL of extract into a 50 mL volumetric flask approximately ¾ full of laboratory reagent water (organic-free).

8.2.1.2 Dilute to volume with laboratory reagent water (organic-free).

8.2.1.3 Transfer to a 40 mL vial and place vial on autosampler for analysis.

8.4 If analysis of the high level preparation exceeds the calibration range, reanalyze by using an appropriate smaller extract volume to dilute into the 50 mL volumetric flask or perform a serial dilution until response is with the calibration range.

8.5 Follow the main body of the procedure to complete analysis and data reduction.

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Turbidimetric Sulfate (Konelab)

ASTM Method D 516-02
SW-846 Method 9038

APPROVALS:

Area Supervisor: Heather L. Brady Date: 1-30-09
Heather L. Brady

QA Officer: Date: 1-30-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 1/30/09
Jeff P. Glaser

Procedure Number: GR-05-124
Revision Number: 0.3

Date Initiated: 8/9/04
Effective Date: 2/24/09

Date Revised: 1/24/09
Pages Revised: All

By: Jodi L. Blouw
Total Number of Pages: 17

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-19-10		4-19-11

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to effluents, surface water, groundwater and aqueous extracts of domestic and industrial soil or waste.

1.2 The applicable concentration range is 1.0 to 35 mg/L. Higher concentrations may be analyzed by using a sample dilution.

1.3 The minimum reporting limit is 1.0 mg/L for aqueous samples and 10 mg/kg for solids extracted with laboratory reagent water.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *American Society of Testing and Materials,* approved January 2002, "Standard Test method for Sulfate Ion in Water", D 516-02, published April 2002

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, December 1996, Method 9038, "Sulfate (Turbidimetric)", Revision 0, September, 1986

3.0 SUMMARY OF PROCEDURE

3.1 A conditioning reagent is added to samples then barium chloride is added to convert sulfate to a barium sulfate suspension under controlled conditions.

3.2 Turbidity from the barium chloride is measured by semi-automated spectrophotometry (Konelab) and absorbance is compared to standards of known sulfate concentration.

4.0 PARAMETER OR COMPOUND LIST

4.1 Sulfate

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-05-129, *Konelab Aqua 20 Spectrophotometer Operation*, latest revision

5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratory Corrective Action,* latest revision

5.4 TriMatrix SOP GR-10-125, *Method Detection Limit*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Color or suspended particles in large amounts will interfere with analysis. Correct for color by running a sample blank from which the barium chloride has been omitted. Remove suspended material by filtration.

6.2 Silica in excess of 500 mg/L will cause interference.

6.3 Analysis of non-aqueous liquid organic matrices such as oils and solvents are beyond the scope of this procedure.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Personal protective equipment must be worn in the laboratory, including disposable gloves, approved eye protection and a laboratory coat.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Collect samples in inert plastic bottles and preserve at 4 ±2° C until analysis.

8.2 Let samples return to room temperature just before analysis.

8.3 Perform the analysis within 28 days of sample collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Thermo-Electron Konelab Aqua 20 discrete analyzer, model 968, operated at 420 nm

9.2 Graduated cylinders, various sizes

9.3 Volumetric flasks, Class A

9.4 Volumetric pipets, Class A

9.5 Autopipetters, various sizes, NIST traceable

9.6 Beakers, Pyrex, 1000 mL

9.7 Hotplate, stirring with PTFE-coated stir-bars

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Refer to TriMatrix SOP GR-05-129 for instructions on performing preventive and corrective maintenance.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM Type II, Milli-Q laboratory system

11.2 Using ACS grade chemicals, prepare the conditioning reagent as follows:

11.2.1 Add 10.0 g anyhydrous/dried barium chloride ($BaCl_2$) and 20 g anhydrous/dried sodium chloride (NaCl) to about 300 mL reagent water in a 1000 mL beaker.

Note: Do not use reagent crystals larger than 20-30 mesh.

11.2.2 Gently boil on a hotplate while stirring then add 0.5 g gelatin and continue stirring to dissolve. Cool to room temperature.

11.2.3 After cooling, carefully add 5 mL concentrated hydrochloric acid (HCl).

11.2.4 Quantitatively transfer to a 1000 mL volumetric flask and dilute to 1000 mL with reagent water.

11.2.5 Expiration is six months from the date prepared.

12.0 STANDARDS PREPARATION

12.1 Primary stock standard (1000 mg/L): Dissolve 1.479 g anhydrous sodium sulfate (Na_2SO_4) in approximately 900 mL reagent water in a one liter volumetric flask. Dilute to the mark with reagent water. Expiration is six months from the date prepared.

12.2 Intermediate standard (100 mg/L): An intermediate standard is prepared by pipetting 20 mL of 1000 mg/L stock into about 150 mL of reagent water, in a 200 mL volumetric flask. Dilute to volume with reagent water and invert ten times to mix. Prepare just before setting up working calibration standards.

12.3 Prepare working standards by diluting the following volumes of intermediate 100 mg/L standard in a 100 mL volumetric flask and bringing to the mark with reagent water. Expiration is 30 days from the date made.

Volume of 100 mg/L Standard to add (mL)	Final Concentration (mg/L SO_4^{-2})	Calibration
35	35	high level
30	30	high level
25	25	high level
20	20	high level
15	15	low and high level
10	10	low and high level
5.0	5.0	low and high level
2.0	2.0	low level
1.0	1.0	low level

12.4 Label all standards with the following information:

12.4.1 Preparer's initials

12.4.2 Preparation date

12.4.3 Concentration and units

12.4.4 Parameter name

12.4.5 Standard log number as it appears in the standards logbook

12.5 Record all standards in the standards log under a unique ID number. Refer to Attachment 23.1 for a standards record example.

12.6 Sulfate spiking solution (10,000 mg/L): Dissolve 1.479 g anhydrous sodium sulfate (Na_2SO_4) in approximately 50 mL reagent water in a 100 mL volumetric flask. Dilute to the mark with laboratory reagent water. Expiration for this solution is six months from the date made.

13.0 SAMPLE PREPARATION

13.1 Pretreatment is not necessary unless color or turbidity is observed and sample dilution is required.

13.2 If the reporting limit is elevated from diluting and the result is less than the new reporting limit, qualify as being raised due to color or turbidity.

14.0 CALIBRATION PROCEDURES

14.1 Analyze standards in increasing order.

14.2 The sulfate analysis may give a nonlinear response. For non-linear calibration, set the instrument to "second-order" regression instead of linear.

14.3 Do not use the cubic spline fit function to calibrate the instrument for sample analysis.

15.0 ANALYTICAL PROCEDURE

15.1 Refer to TriMatrix SOP GR-05-129 for detailed instructions on operating the Konelab.

15.2 Fill sample cups with appropriate standards and samples. Place in the instrument.

15.3 The instrument performs the following steps:

15.3.1 Takes 120 µL of standard or sample to be analyzed

15.3.2 Performs a background

15.3.3 Adds 40 µL conditioning reagent to the 120 µL aliquot

15.3.4 Incubates for one minute at 37° C

15.3.5 Stirs the sample and reagent mixture

15.3.6 Incubates for four minutes at 37° C

15.3.7 Reads absorbance at 420 nm, at 4.0 minutes.

15.4 After sample analysis, perform a standby and shut down the instrument.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The Konelab calculates all results against the calibration curve and prints out concentration in mg/L sulfate.

16.2 No manual calculations are necessary except to account for sample dilutions.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Attach the following to the benchsheet for each sample batch analyzed:

17.1.1 A copy of the calibration curve.

17.1.2 A copy of the raw data printout.

17.1.3 Refer to Attachments 21.2 and 21.3 for examples of a calibration curve and raw data.

17.2 The instrument run logbook must be filled in for each batch analyzed with the following information:

17.2.1 Date analyzed

17.2.2 Analyst initials

17.2.3 Method name and number

17.2.4 Calibration standards used

17.2.5 Client names and sample numbers analyzed

17.3 Record the following data on the benchsheet, for each batch analyzed:

17.3.1 SCV, BLK, sample concentrations, CCV, CCB, SPK, MSD

17.3.2 Instrument number, analyst, date run, stock standard numbers, reagent numbers

17.3.3 Calibration standard concentrations, observed concentrations, Element™ identifications.

17.4 Refer to Attachments 23.4 through 23.7 for examples of the instrument run logbook, analytical benchsheet, run sequence sheet and batch detail report.

18.0 QUALITY ASSURANCE

18.1 Method QC must be analyzed with each sample batch and consists of a Second-source Calibration Verification (SCV), Instrument Blanks (BLK), Continuing Calibration Verifications (CCV), Continuing Calibration Blanks (CCB) and Detection Limit Confirmation Standards (CRDL).

18.1.1 The SCV is an aliquot of purchased Analytical Products Group (APG) standard.

18.1.1.1 Record all APG standard preparations in the standards record. Refer to Attachment 23.1 for a standards record example.

18.1.1.2 Acceptance limits for SCV percent recovery are listed in the laboratory information management system (Element™). However, if LIMS limits are wider than APG limits, use APG limits to determine acceptability.

18.1.2 A BLK is an aliquot of laboratory reagent water analyzed like a sample. Results must be less than the minimum reporting limit of 1.0 mg/L (or 5.0 mg/L if using the high-level calibration).

18.1.3 CCVs are aliquots of calibration standards analyzed at the start of the analytical sequence, every four samples and at the end of the analytical sequence.

18.1.3.1 Acceptance limits for CCVs are 85 – 115% of the prepared value.

18.1.3.2 Include in the counting any SCV, BLK, MS or MSD.

18.1.3.3 The CCV concentration must be varied throughout the run using calibration standards.

18.1.4 CCBs are aliquots of reagent water analyzed after every CCV and at the end of each analytical run sequence. Results must be less than the minimum reporting limit of 1.0 mg/L (or 5.0 mg/L if using the high-level calibration).

18.1.5 A CRDL is an aliquot of the 1.0 mg/L working standard analyzed like a sample. Acceptance limits for the 1.0 mg/L CRDL are 40 – 160% of the prepared value.

18.2 Matrix QC which must be done for every sample batch, consists of a Matrix Spike (MS) and Matrix Spike Duplicate (MSD).

18.2.1 The MS/MSD are prepared as follows: Pipette 100 µL of the 10,000 mg/L sulfate spiking solution into a 50 mL sample aliquot. Run in the same way as the unspiked sample.

18.2.2 The sulfate spike concentration in the sample aliquot is 20 mg/L.

18.2.3 Acceptance limits for the MSD relative percent difference result are listed in LIMS.

18.3 Refer to TriMatrix SOP GR-10-103 for corrective action requirements when quality control results are out of acceptance limits.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 Before analyzing samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by performing a successful demonstration of capability study.

19.2 To perform a demonstration of capability study, prepare four 50 mL aliquots of APG LCS or four 50 mL aliquots of a 20 mg/L sulfate solution prepared separately from the 1000 mg/L calibration stock (use the 10,000 mg/L matrix spiking solution).

19.3 Analyze the four aliquots following every step in the procedure.

19.4 Calculate average percent recovery and relative standard deviation using the four results obtained.

19.4.1 Average recovery must be within 85 – 115% and RSD must be less than or equal to 20%.

19.4.2 If these criteria are met, the capability study is acceptable and complete except for entering into the IDC spreadsheet on the library and printing a copy for the QA department. Upon acceptable completion, the analyst is authorized to perform sulfate analysis.

19.4.3 If these criteria are not met, repeat the IDC study as follows:

19.4.3.1 Locate and correct the problem then repeat the study successfully.

19.4.3.2 A repeat of the failure indicates a problem with the procedure and/or techniques used. If this occurs, locate and correct the problem, modify the procedure and/or techniques which caused the error then repeat the study successfully.

19.4.4 Sulfate may not be analyzed by the analyst until a demonstration of capability study has been completed successfully and submitted to the QA department.

19.4.5 A demonstration of capability study is required annually by any of the following when analyzed exclusively by the analyst:

19.4.5.1 By repeating the IDC study.

19.4.5.2 By inputting the last four results from a method detection limit study to the IDC spreadsheet.

19.4.5.3 By inputting four consecutive blank spikes obtained during routine sample analysis to the IDC spreadsheet.

19.4.5.3 By successfully analyzing a blind Performance Testing sample from a source outside the laboratory.

19.5 For each instrument, a method detection limit (MDL) study is required annually in accordance with TriMatrix GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate material safety data sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library and accessed from any computer.

21.2 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal.

22.0 REFERENCES

22.1 *American Society of Testing and Materials,* approved January 2002, "Standard Test method for Sulfate Ion in Water", D 516-02, published April 2002

22.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, December 1996, Method 9038, "Sulfate (Turbidimetric)", Revision 0, September, 1986

23.0 ATTACHMENTS

23.1 Standards Record Example

23.2 Calibration Example

23.3 Data Printout Example

23.4 Instrument Run Logbook Example

23.5 Analytical Benchsheet Example

23.6 Run Sequence Sheet Example

23.7 Batch Detail Report Example

Attachment 23.1
Standards Record Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8020936

Description	Sulfate 20 mg/L	Expires	Mar-29-08
Standard Type	Calibration Star	Prepared	Feb-29-08
Solvent	Solvent Lot #	Prepared By	Gretchen E. Housekeeper
Final Volume (mls)	100	Department	Expired
Vials	1	Last Edit	Apr-03-08 14:30 by GEH

Analyte	CAS Number	Concentration	Units
Sulfate as SO4	148-08-798	20	mg/L
Sulfate (soluble)	14808-79-8	20	mg/L
Sulfate	14808-79-8	20	mg/L

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
8020931	Sulfate 100 mg/L	Feb-29-08	Gretchen E. Housek	Mar-29-08	Apr-03-08 14:29 by GEH	20

Approved By: QA 1-26-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 23.2
Calibration Example

Calibration results AquaKem 6.5 Page: 1

Laboratory
Analyzer User

21.12.2005 05:42

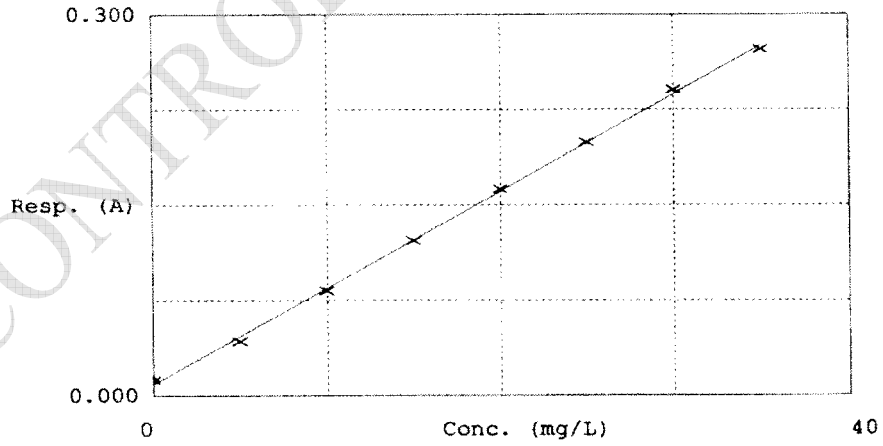
Test SO4

Accepted 20.12.2005 06:39

Resp. = A * Conc. ^ 2 + B * Conc. + C
A = 0
B = 0.008
C = 0.009

Coeff. of det. 0.999076

Errors Meas. error



	Calibrator	Response	Calc. con.	Conc.	Errors
1	SO4-0	0.012	0.462	0.000	Blank resp. low
2	SO4-5	0.043	4.504	5.000	Blank resp. low
3	SO4-10	0.083	9.739	10.000	Blank resp. low
4	SO4-15	0.122	14.927	15.000	Blank resp. low
5	SO4-20	0.163	20.247	20.000	
6	SO4-25	0.199	25.069	25.000	Blank resp. low
7	SO4-30	0.241	30.457	30.000	Blank resp. low
8	SO4-35	0.273	34.594	35.000	Blank resp. low

Approved By: _____ 1-26-09 **Approved By:** Heather L Brady
QA Officer **Area Supervisor**

Attachment 23.3
Data Printout Example

Result Report — Konelab 6.0.2 — Page: 1

Laboratory
Konelab User

Date : 2005-10-03
Time : 13.55

Test: SO4
Unit: mg/l

Sample ID:	Result	Date and Time	Man.dilut	Dilut	Resp.
SO4ccv	10.16	2005-10-03 06.25			0.083
SO4ccb	0.01	2005-10-03 06.25			0.001
blk	<0.00	2005-10-03 06.25			0.001
lcs	23.70	2005-10-03 06.25			0.179
crdl 5	4.94	2005-10-03 06.25			0.034
SO4ccv	10.03	2005-10-03 06.32			0.082
SO4ccb	<0.00	2005-10-03 06.32			0.000
SO4ccv	9.59	2005-10-03 06.36			0.078
SO4ccb	0.02	2005-10-03 06.36			0.001
40	37.62	2005-10-03 07.13		1+1.0	0.151
100	93.73	2005-10-03 07.13		1+4.0	0.151
500	504.98	2005-10-03 07.13		1+19.0	0.187
200	188.05	2005-10-03 07.22		1+9.0	0.151
SO4ccv	10.01	2005-10-03 07.24			0.082
SO4ccb	0.24	2005-10-03 07.24			0.002
0509269-03	20.25	2005-10-03 07.30			0.161
0509367-01	21.85	2005-10-03 07.30			0.170
SO4ccv	10.20	2005-10-03 07.39			0.083
SO4ccb	0.60	2005-10-03 07.39			0.004
0509269-02	34.35	2005-10-03 07.42		1+1.0	0.139
0509269-02 spk1	55.04	2005-10-03 07.42		1+1.0	0.201
0509269-02 spk2	56.10	2005-10-03 07.42		1+1.0	0.204
SO4ccv	10.31	2005-10-03 07.45			0.084
SO4ccb	1.20	2005-10-03 07.45			0.008
0509367-02	25.87	2005-10-03 07.58			0.190
0509367-03	24.40	2005-10-03 07.58			0.182
SO4ccv	10.18	2005-10-03 07.58			0.083
SO4ccb	<0.00	2005-10-03 07.58			0.000
0509367-04	22.38	2005-10-03 08.05			0.173
0509367-05	21.70	2005-10-03 08.05			0.169
0509367-06	21.59	2005-10-03 08.06			0.169
SO4ccv	9.91	2005-10-03 08.06			0.081
SO4ccb	<0.00	2005-10-03 08.06			0.000
0509367-09	26.45	2005-10-03 08.09			0.194
0509367-10	0.01	2005-10-03 08.09			0.001
0509374-01	<0.00	2005-10-03 08.09			-0.003
SO4ccv	15.65	2005-10-03 08.13			0.126
SO4ccb	<0.00	2005-10-03 08.13			-0.000
0509402-01	0.12	2005-10-03 08.21			0.001
0509402-02	17.21	2005-10-03 08.21			0.139
0509402-03	16.68	2005-10-03 08.21			0.135
0509402-04	<0.00	2005-10-03 08.21			-0.012
SO4ccv	15.31	2005-10-03 08.21			0.123
SO4ccb	<0.00	2005-10-03 08.21			0.000
0509402-05	0.10	2005-10-03 08.25			0.001
SO4ccv	15.14	2005-10-03 08.32			0.122
SO4ccb	<0.00	2005-10-03 08.32			0.000
0509372-02	<0.00	2005-10-03 08.36			-0.082
0509372-03	29.18	2005-10-03 08.36			0.210
SO4ccv	19.37	2005-10-03 08.36			0.155

Approved By: _____ 1-26-09 — **QA Officer**

Approved By: Heather L Brady — **Area Supervisor**

Attachment 23.4
Instrument Run Logbook Example

TriMatrix Laboratories, Inc.

Instrument Number 298 Run Logbook

Date	Analyst	Method Name or Number	Concentration of Calibration Standards Used							Units	Client Names and Sample Numbers
			No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7		
8.11.04	JAB	SO4	0	5/10	15	20	25	30	35	mg/L	[redacted] 367347-409 FB 367154 [redacted] 366123 Calibration Requirements ≥0.995 Calibration Results 1.0000 [redacted] 367158.155, FB H 367164-156,157-163 [redacted] 367165, 169, 357, 358
8-11-04	INR	TPO4	0	0.01	0.05	0.1	0.5	1.0		mg/L	[redacted] 367361 + 6more, 367251-256, 313 322 [redacted] 367348, CCV 367-378 [redacted] 367135 [redacted] 367137 Calibration Requirements ≥0.995 Calibration Results 0.999781
8.12.04	EGB	NH4	2.0	1.0	0.5	0.1	0.05	0.01		ppm	[redacted] 367361, 367388-93, 367320-22 367325-56 [redacted] 367348-49, 367387-88 [redacted] 367308 [redacted] 367397-410, 367374-87 50 367276 [redacted] 367274-75 Calibration Requirements ≥0.995 Calibration Results 0.999944
8.13.04	JAB	Cl-	0	1	5/10	20	40	75	100	mg/L	[redacted] 367574-87 [redacted] 367437,438 [redacted] 367435,436 Calibration Requirements ≥0.995 Calibration Results 1.0000 JAB 8/13/04
8.17.04	JAB	SO4	0	5/10	15	20	25	30	35	mg/L	[redacted] 366151-154, 156, 262-273 [redacted] 367410, 574-587 [redacted] 367225, 269, 435, 436 Calibration Requirements ≥0.995 Calibration Results 1.0002
8.19.04	JAB	CN	0	.005	.01	.02	.05	.10	0.20	mg/L	[redacted] 367152-161, 189-193 [redacted] 367411-415, 432-431 [redacted] 367387- [redacted] 367196-202 [redacted] 367361, 555 597 Calibration Requirements ≥0.995 Calibration Results 0.996708
8.17.04	JAB	Phenol	0.0	0.2	0.4	0.6	0.8	0.10	0	mg/L	[redacted] 367196-201, 224 [redacted] 367-357 [redacted] 367 [redacted] 574-587 Calibration Requirements ≥0.995 Calibration Results 0.9975

Approved By: [signature] 1-26-09 **QA Officer**

Approved By: Heather L Brady **Area Supervisor**

Attachment 23.5
Analytical Benchsheet Example

PREPARATION BENCH SHEET

0509158

TriMatrix Laboratories, Inc.

inted: 10/10/2005 9:34:40AM

Matrix: Water **ed using: Inorganic - Wet Chemistry - General Inorgan** **(No Surrogate)**

Lab Number	Analysis	Prepared	Initial (mL)	Final (mL)	Spike ID	Source ID	ul Spike	ul Surrogate	Client	Extraction Comments
0509158-BLK1	QC	Oct-03-05 08:30	10	10						
0509158-BS1	QC	Oct-03-05 08:30	10	10	A507183		10000			
0509158-MS1	QC	Oct-03-05 08:30	10	10	A508124	0509308-13	20			
0509158-MSD1	QC	Oct-03-05 08:30	10	10	A508124	0509308-13	20			
0509285-09	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509285-11	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509285-12	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509285-13	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509285-14	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509285-15	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509285-16	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509285-17	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-01	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-03	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-04	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-08	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-09	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-10	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-11	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-12	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-13	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l
0509308-14	Sulfate 375.4 (low level	Oct-03-05 08:30	10	10					[redacted]	1 mg/l

Spiking Witnessed By Date | Preparation Reviewed By Date | Extracts Received By Date

Approved By: 1-26-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 23.6
Run Sequence Sheet Example

ANALYSIS SEQUENCE

5101009

Instrument: 298

Calibration ID: UNASSIGNED

Printed: 10/10/2005 9:34:32AM

Lab Number	Analysis	Container	Order	Position	STD ID	ISTD ID	Client	Comments
0509285-16	Sulfate 375.4 (low level)	D	24				[redacted]	1 mg/l
5101009-CCV4	QC		25		A509300			
5101009-CCB4	QC		26					
0509308-01	Sulfate 375.4 (low level)	D	27				[redacted]	1 mg/l
0509308-03	Sulfate 375.4 (low level)	D	28				[redacted]	1 mg/l
5101009-CCV5	QC		29		A509300			
5101009-CCB5	QC		30					
0509308-08	Sulfate 375.4 (low level)	D	31				[redacted]	1 mg/l
0509308-09	Sulfate 375.4 (low level)	D	32				[redacted]	1 mg/l
5101009-CCV6	QC		33		A509300			
5101009-CCB6	QC		34					
0509308-11	Sulfate 375.4 (low level)	D	35				[redacted]	1 mg/l
0509308-12	Sulfate 375.4 (low level)	D	36				[redacted]	1 mg/l
5101009-CCV7	QC		37		A509300			
5101009-CCB7	QC		38					
0509285-12	Sulfate 375.4 (low level)	D	39				[redacted]	1 mg/l
0509285-13	Sulfate 375.4 (low level)	D	40				[redacted]	1 mg/l
5101009-CCV8	QC		41		A509300			
5101009-CCB8	QC		42					
0509308-10	Sulfate 375.4 (low level)	D	43				[redacted]	1 mg/l
0509308-13	Sulfate 375.4 (low level)	D	44				[redacted]	1 mg/l
0509158-MS1	QC		45					
0509158-MSD1	QC		46					

Samples Loaded By _____ Date _____ Data Processed By _____ Date _____

Approved By: _____ 1-26-09 **QA Officer**

Approved By: Heather L Brady **Area Supervisor**

Attachment 23.7
Batch Detail Report Example

Data Review Report

0509285-09	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	7.07	7.1mg/L			
0509285-11	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	32.19	32mg/L			
0509285-12	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	169.44	170mg/L			
0509285-13	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	288.11	290mg/L			
0509285-14	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	1,112.80	1100mg/L			
0509285-15	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	7.00	7.0mg/L			
0509285-16	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	7.63	7.6mg/L			
0509285-17	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	1,086.40	1100mg/L			
0509308-01	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	32.17	32mg/L			
0509308-03	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	14.45	14mg/L			
0509308-04	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	34.28	34mg/L			
0509308-08	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	33.14	33mg/L			
0509308-09	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	30.00	30mg/L			
0509308-10	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	42.88	43mg/L			
0509308-11	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	22.25	22mg/L			
0509308-12	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	26.18	26mg/L			
0509308-13	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	35.73	36mg/L			
0509308-14	BWC-05-09-MW	Sulfate 375.4 (low level)	Sulfate	41.39	41mg/L			
5101009-CAL1	Cal Standard	Sulfate 375.4 (low level)	Sulfate	0.00	0.00mg/L			
5101009-CAL2	Cal Standard	Sulfate 375.4 (low level)	Sulfate	0.00	0.00mg/L	5.00		
5101009-CCV2	Calibration Check	Sulfate 375.4 (low level)	Sulfate	9.81	9.81mg/L	10.0	98	
5101009-CCB2	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.00	0.0mg/L			
5101009-CCV3	Calibration Check	Sulfate 375.4 (low level)	Sulfate	9.74	9.74mg/L	10.0	97	
5101009-CCB3	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.00	0.0mg/L			
5101009-CCV4	Calibration Check	Sulfate 375.4 (low level)	Sulfate	10.07	10.1mg/L	10.0	101	
5101009-CCB4	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.27	0.27mg/L			
5101009-CCV5	Calibration Check	Sulfate 375.4 (low level)	Sulfate	9.19	9.19mg/L	10.0	92	
5101009-CCV7	Calibration Check	Sulfate 375.4 (low level)	Sulfate	9.99	9.99mg/L	10.0	100	
5101009-CCB7	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.21	0.21mg/L			
5101009-CCV8	Calibration Check	Sulfate 375.4 (low level)	Sulfate	10.17	10.2mg/L	10.0	102	
5101009-CCVA	Calibration Check	Sulfate 375.4 (low level)	Sulfate	13.67	13.7mg/L	15.0	91	
5101009-CCBA	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.03	0.030mg/L			
5101009-CCVB	Calibration Check	Sulfate 375.4 (low level)	Sulfate	23.64	23.6mg/L	25.0	94	
0509158-MSD1	Matrix Spike Dup	Sulfate 375.4 (low level)	Sulfate	54.12	54.1mg/L	20.0	92	5
0509158-BS1	LCS	Sulfate 375.4 (low level)	Sulfate	23.70	23.7mg/L	23.8	100	
5101009-CAL3	Cal Standard	Sulfate 375.4 (low level)	Sulfate	0.00	0.00mg/L	10.0		
5101009-CAL7	Cal Standard	Sulfate 375.4 (low level)	Sulfate	0.00	0.00mg/L	30.0		
5101009-CAL8	Cal Standard	Sulfate 375.4 (low level)	Sulfate	0.00	0.00mg/L	35.0		
5101009-ICV1	Initial Cal Check	Sulfate 375.4 (low level)	Sulfate	10.16	10.2mg/L	10.0	102	
5101009-CCV1	Calibration Check	Sulfate 375.4 (low level)	Sulfate	10.03	10.0mg/L	10.0	100	
5101009-CCB1	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.00	0.0mg/L			
5101009-CCB5	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.32	0.32mg/L			
5101009-CCV6	Calibration Check	Sulfate 375.4 (low level)	Sulfate	9.94	9.94mg/L	10.0	99	
5101009-CCB6	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.00	0.0mg/L			
5101009-CCB8	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.00	0.0mg/L			
5101009-CCV9	Calibration Check	Sulfate 375.4 (low level)	Sulfate	25.91	25.9mg/L	25.0	104	
5101009-CCB9	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.02	0.020mg/L			
5101009-CCBB	Calibration Blank	Sulfate 375.4 (low level)	Sulfate	0.07	0.070mg/L			
0509158-BLK1	Blank	Sulfate 375.4 (low level)	Sulfate	0.01	0.010mg/L			
0509158-MS1	Matrix Spike	Sulfate 375.4 (low level)	Sulfate	56.93	56.9mg/L	20.0	106	
5101009-CAL4	Cal Standard	Sulfate 375.4 (low level)	Sulfate	0.00	0.00mg/L	15.0		
5101009-CAL5	Cal Standard	Sulfate 375.4 (low level)	Sulfate	0.00	0.00mg/L	20.0		
5101009-CAL6	Cal Standard	Sulfate 375.4 (low level)	Sulfate	0.00	0.00mg/L	25.0		
5101009-ICB1	Initial Cal Blank	Sulfate 375.4 (low level)	Sulfate	0.01	0.0100mg/L			
5101009-SCV1	Secondary Cal Ch	Sulfate 375.4 (low level)	Sulfate	23.70	23.7mg/L	23.8	100	

Approved By: _____ 1-26-09 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Total Organic Carbon (TOC)
Dissolved Organic Carbon (DOC)

Standard Methods 5310 C
SW-846 Method 9060A

APPROVALS:

Area Supervisor: Heather L. Brady Date: 1-27-09
Heather L. Brady

QA Officer: Date: 1-26-09
Tom C. Boocher

Operations Manager: Date: 1/27/09
Jeff P. Glaser

Procedure Number: GR-05-105
Revision Number: 3.4

Date Initiated: 3/29/94
Effective Date: 2/21/08

Date Revised: 1/23/09
Pages Revised: All

By: Lucy Asbury
Total Number of Pages: 19

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-19-10		4-19-11

1.0 SCOPE AND APPLICATION

1.1 Total Organic Carbon (TOC) is the sum of purgeable (POC), non-purgeable (NPOC) and dissolved (DOC) organic carbon.

1.2 This procedure includes the analysis of TOC, TOC (as NPOC) and DOC. Applicable matrices include drinking water, surface water, saline water, and domestic and industrial wastes that can be drawn from a vial by septum needle.

1.2 Analysis reporting limits are 0.5 to 100 mg/L. This range may be extended by sample dilution.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998,* 5310 Total Organic Carbon, C. Persulfate-Ultraviolet or Heated-Persulfate Oxidation Method

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, August, 2002, Method 9060A, "Total Organic Carbon"

3.0 SUMMARY OF PROCEDURE

3.1 Organic carbon determinations are based on the oxidative release of carbon dioxide. Samples are acidified and, inorganic and purgeable organic carbon is removed by sparging with nitrogen.

3.2 After removal of inorganic carbon, sodium persulfate, a strong oxidizer is added. The oxidizer reacts quickly with organic carbon at 100° C to form carbon dioxide (CO_2).

3.3 The carbon dioxide is purged from solution and concentrated on a trap then desorbed and carried into a non-dispersive infrared detector (NDIR). The instrument is calibrated to display carbon dioxide mass. The resultant mass is equivalent to the organic carbon in the sample volume purged.

4.0 PARAMETER OR COMPOUND LIST

4.1 Total Organic Carbon (TOC as NPOC)

4.2 Dissolved Organic Carbon (DOC)

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 USEPA Great Lakes National Program Office, *Standard Operating Procedure for the Sampling of Particulate-Phase and Dissolved Phase Organic Carbon in Great Lakes Waters,* Revision 1, March 4, 1993

5.3 Operators Manual (OI Analytical), *Aurora 1030 Wet Oxidation TOC Analyzer Operator's Manual*, Revision 1.3.1 – November 2007

5.4 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Suspended solids can cause high and erratic results.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Personal protective equipment must include a laboratory coat, disposable gloves and approved safety glasses when working in the laboratory. Safety glasses must be worn at all times in the inorganics laboratory.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples for TOC are collected in triplicate, in 40 mL amber VOA vials. Vials must be preserved with sulfuric acid. Fill each vial carefully and almost to overflowing. There must be no headspace or bubbles in collected samples.

8.2 DOC samples are collected in a single 500 mL amber glass bottles. No preservative is required. Rinse bottles several times with sample before collecting.

8.3 Samples must be stored at 4 ±2° C until time of analysis.

8.4 All analyses must be performed within 28 days of collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 OI Analytical, model 1030 TOC analyzer, 10 mL syringe

9.2 OI Analytical, autosampler, model 1088

9.3 Printer

9.4 Glass pipettes, volumetric, class "A"

9.5 Amber VOA vials, with PTFE-lined septum caps

9.6 Volumetric flasks, 100 mL

9.7 Glass fiber filters, 47 mm /0.45 µm

9.8 Syringe 50 mL filtration

9.9 Reagent bottles, polyethylene, various sizes

9.10 Hotplate, stirring

9.11 Drying oven

9.12 Desiccator, with desiccant

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Routine preventive maintenance is performed every two weeks.

10.2 Maintenance includes changing the reagent tubing and the check valves. Maintenance involves cleaning the system with sodium persulfate and performing a leak check. Refer to the instrument operator's manual for further maintenance instructions.

11.0 CHEMICALS AND REAGENTS

11.1 Reagent water, organic free ASTM Type II, MilliQ system, degassed for 30 minutes

11.2 Potassium hydrogen phthalate or KHP (potassium biphthalate) - Dry at 120° C for two hours, store in a desiccator

11.3 Phosphoric acid (H_3PO_4), concentrated (~85%), certified ACS grade

11.4 Phosphoric acid (H_3PO_4), 5% (v/v) - Dilute 59 mL of concentrated H_3PO_4 (~85%) to 1 L with degassed reagent water. Expiration is six months from the date made.

11.5 Sodium persulfate ($Na_2S_2O_8$), 98% minimum purity

11.6 Sodium persulfate ($Na_2S_2O_8$) solution

11.6.1 Dissolve 200 g $Na_2S_2O_8$ in reagent water and dilute to 1 L.

11.6.2 Heat to boiling on the hotplate while stirring then cool to room temperature.

11.6.3 Expiration is six months from the date made.

12.0 STANDARDS PREPARATION

12.1 TOC stock solution (1000 mg/L as carbon):

12.1.1 Dissolve 2.128 g of KHP in a 1 L volumetric, partially filled with degassed reagent water. Acidify to pH <2 with concentrated phosphoric acid then dilute to volume with degassed reagent water. Expiration is six months from the date made.

12.1.2 The molecular weight of KHP [2-$(HO_2C)C_6H_4CO_2K$] is 204.22 g/mol and is 47% carbon (0.47 as a fraction). Calculate percent carbon as follows:

$$\frac{12 \text{ g Carbon}}{\text{mol}} \left| \frac{\text{mol}}{204.22 \text{ g 2-}(HO_2C)C_6H_4CO_2K} \right| 8 \left| 100 \right. = 47\%$$

where:
8 = the number of carbons in the molecular formula
12 = the molecular weight of carbon, in g/mol

12.1.2 Calculate the TOC stock carbon concentration as follows:

$$\frac{2.128 \text{ g KHP}}{\text{L}} \left| \frac{0.47 \text{ g Carbon}}{\text{g KHP}} \right| \frac{1000 \text{ mg}}{\text{g}} = \frac{1000 \text{ mg Carbon}}{\text{L}}$$

Where:
KHP = potassium biphthalate

12.2 Prepare low level calibration standards as follows

12.2.1 Calibration standard and upper level calibration verification standard (10 mg/L as carbon)

12.2.1.1 Pipette 5 mL of 1000 Mg/L carbon (Section 12.1) into a 500 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration is 30 days from the date made.

12.2.2 Calibration standard and CCV (5 mg/L as carbon)

12.2.2.1 Pipette 5 mL of 1000 mg/L (section 12.1) into a 1000 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration is 30 days from the date made.

12.2.3 Calibration standard (2.5 mg/L as carbon)

12.2.3.1 Pipette 25 mL of 10 mg/L carbon (Section 12.2.1) into a 100 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Prepare fresh when needed for calibration.

12.2.4 Calibration standard and CRDL (0.5 mg/L as carbon)

12.2.4.1 Pipette 5 mL of 10 mg/L carbon (section 12.2.1) into a 100 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Prepare fresh each day low level TOC samples are analyzed.

12.3 Prepare High level calibration standards as follows:

12.3.1 Calibration standard and upper level calibration verification (100 mg/L as carbon)

12.3.1.1 Pipette 10 mL of 1000 mg/L carbon (Section 12.1) into a 100 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration is 30 days from date made.

12.3.2 Calibration standard (75 mg/L as carbon)

12.3.2.1 Pipette 15 mL of 1000 mg/L carbon (Section 12.1) into a 200 volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration is 30 days from the date made.

12.3.3 Calibration standard and CCV (50 mg/L as carbon)

12.3.3.1 Pipette 10 mL of 1000 mg/L carbon (Section 12.1) into a 200 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration is 30 days from the date made.

12.3.4 Calibration standard (25 mg/L as carbon)

12.3.4.1 Pipette 5 mL of 1000 mg/L carbon (Section 12.1) into a 200 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration is 30 days from the date made.

12.3.5 Calibration standard (10 mg/L as carbon)

12.3.5.1 Pipette 5 mL of 1000 mg/L carbon (Section 12.1) into a 500 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration is 30 days from the date made.

12.3.6 Calibration standard and CRDL (1.0 mg/L as carbon)

12.3.6.1 Pipette 10 mL of 10 mg/L carbon (Section 12.3.5) into a 100 volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Prepare fresh each day high level TOC samples are analyzed.

12.4 All standards must be recorded in the appropriate standards logbook under a unique ID number. Refer to Attachment 23.1. All standards must be entered into Element.

13.0 SAMPLE PREPARATION

13.1 There is no preparation required for total organic carbon (TOC) samples before analysis.

13.2 Dissolved organic carbon (DOC) samples require filtration through a 0.45 um glass fiber filter.

13.2.1 Method preparation blanks (BLK), blank spike (BS), and contract-required detection limit (CRDL) samples:

13.2.1.1 Prime syringe by filtering 50 mL of BLK, BS, CRDL. Discard the filtrate.

13.2.1.2 Syringe filter an additional 40 mL and filter into one 40 mL amber vial. Cap with no headspace. Discard remaining filtrate.

13.2.2 Sample preparation:

13.2.2.1 Prime syringe by filtering 50 mL of sample. Discard the filtrate.

13.2.2.2 Syringe filter an additional 40 mL and filter into one 40 mL amber vial. Cap with no headspace. Discard remaining filtrate.

13.2.2.3 Use a new syringe and 0.45 um glass fiber filter per sample.

14.0 CALIBRATION PROCEDURES

14.1 Low Level Calibration (0.5 to 10 mg/L):

14.1.1 Set mode to NPOC only

14.1.2 Set the persulfate volume to 2.0 mL

14.1.3 Set acid volume to 1.0 mL

14.1.4 Set TOC React times to 2:30

14.1.5 Set TOC Detect times to 3:00

14.1.6 Set rinses 1 per sample, 1 per rep.

14.1.7 Set instrument to subtract RW

14.1.8 Set reagent blanks to 5

14.1.9 Set the sample volume to 5.0 mL. Refer to the instrument Operator's Manual for detailed instructions.

14.1.10 Run 1 cleanup, 2 rinse blanks at 3 replicates to stabilize the instrument. Refer to the instrument Operator's Manual for detailed instructions.

14.1.11 Run the standards in ascending order. The correlation coefficient must be ≥0.995, for calibration to be acceptable. If calibration is unacceptable, determine the cause and re-run before samples are analyzed.

14.2 High level calibration (1.0 to 100 mg/L)

14.2.1 Set mode to NPOC only

14.2.2 Set sample volume to 2.0 mL

14.2.3 Set persulfate volume to 2.0 mL

14.2.4 Set acid volume to 1.0 mL

14.2.5 Set TOC react time at 2:30

14.2.6 Set TOC Detect time at 3:00

14.2.7 Set rinses at one rinse per sample, per rep

14.2.8 Set instrument to subtract the offset counts

14.2.9 Set reagent blanks to 5. Refer to the instrument Operator's Manual for detailed instructions.

14.2.10 Run one cleanup by default cleanup method and two rinses blanks at 3 replicates to stabilize the instrument.

14.2.11 Run the standards in ascending order (same as low level)

14.3 Calibration is required when calibration verification or other quality control are out of established acceptance limits. Calibration is also required after preparing a new 1000 mg/L carbon stock.

15.0 ANALYTICAL PROCEDURE

15.1 Powering Up

15.1.1 Turn on the gas supply (20 psi purified nitrogen).

15.1.2 Turn on the instrument.

15.1.3 Turn on the computer.

15.1.4 Turn on the rinse water flow, and the gas control to the rinse water.

15.1.5 Check the acid and oxidant bottles for adequate levels.

15.2 Sample analysis

15.2.1 Create sequences refer to the instrument Operator's Manual for detailed instructions.

15.2.2 Add a cleanup at 3 reps using default cleanup method at the beginning of every sequence.

15.2.3 Add 2 rinse blanks at 3 reps as a sample with the appropriate method reverence after the cleanup on every sequence.

15.2.4 Add QC (CCV, CCB, SCV, CRDL, ULCV) as check standards with 1 rep per vial referencing the appropriate method.

15.2.5 Add BLK and BS as samples with 1 rep per sample referencing the appropriate method.

15.2.6 Add samples as samples with 1 rep per sample referencing the appropriate method.

15.2.7 Add QC (CCV, CCB) as check standards 1 rep each referencing appropriate method every 10 samples.

15.2.8 Save sequence as mm/dd/yy.

15.2.9 Load active sequence, refer to the instrument Operator's Manual for detailed instructions.

15.2.10 Start sequence, refer to the instrument Operator's Manual for detailed instructions.

15.2.11 After sequence has shut down, unload active sequence, refer to the instrument Operator's Manual for detailed instructions.

15.2.12 Check the pH of the analyzed vial. Record on the benchsheet.

15.2.13 Generate new report using the Reporter. Refer to the instrument Operator's Manual for detailed instructions.

15.2.14 Print Report, refer to the instrument Operator's Manual for detailed instructions.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The TOC analyzer prints results in mg/L. All four results and an average are printed if running by method 9060A. Report the average, with narrative stating the lowest and highest result. Otherwise, report the single result obtained without narrative.

16.2 Report results as TOC or DOC, in mg/L.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Attach the following to the laboratory benchsheet, for each analysis batch handed in:

17.1.1 Raw data report (Refer to Attachment 23.2)

17.1.2 Sequence Summary.

17.1.3 Method Summary.

17.1.4 Calibration results printout.

17.2 Fill out the instrument logbook for each batch handed in, with the following information:

17.2.1 Date analyzed.

17.2.2 Analyst's initials.

17.2.3 Method name and number.

17.2.4 Calibration date.

17.2.5 Calibration standards used.

17.2.6 Check standards used.

17.2.7 Reagents used.

17.2.8 Sample numbers analyzed.

17.2.9 Refer to the Attachment for an instrument logbook example.

17.3 Record the following data on the laboratory benchsheet, for each analysis batch handed in:

17.3.1 CCV, CCB, SCV, CRDL, BLK, BS, MS and MSD or DUP sample concentration and range, CCV, CCB/BLK, SPK, MSD or DUP (refer to Section 18.0).

17.3.2 Instrument number, operator, date run, supervisor, stock standard number.

17.3.3 Refer to Attachments 23.4 and 23.5 for preparation batch and analysis sequence report examples.

18.0 QUALITY ASSURANCE

18.1 Method and matrix quality control (QC) must be analyzed with each analysis batch.

18.1.1 Method QC consists of a second-source calibration verification (SCV), an Instrument Blank (BLK), Continuing Calibration Verifications (CCV), Continuing Calibration Blanks (CCB), an Upper Level Calibration Verification (ULCV), and a Contract-Required Detection Limit (CRDL) standard.

18.1.1.1 The SCV is a standard prepared from a vendor separate from the one used for calibration. The SCV (1000 mg/L carbon) is prepared as follows:

18.1.1.1.1 Add 0.4256 g potassium biphthalate from an alternate vendor, to a 200 mL volumetric flask partially filled with degassed reagent water. Acidify to <2 using concentrated phosphoric acid. Dilute to volume with degassed reagent water. Concentration is 1000 mg/L carbon. Expiration is six months from the date made.

18.1.1.1.2 High level SCV (50 mg/L carbon): Pipette 5 mL of the SCV stock into a 100 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration date is 30 days from the date made.

18.1.1.1.3 Low-level SCV (5 mg/L as carbon): Pipette 1 mL of SCV stock into a 200 mL volumetric flask, partially filled with degassed reagent water. Dilute to volume with degassed reagent water. Expiration is 30 days from the date.

18.1.1.1.4 SVC acceptance limits are 90% to 110% of the expected value. If an SCV fails, remake and re-analyze the SCV. If acceptable, proceed with sample analysis. If unacceptable, recalibrate with fresh calibration standards. An acceptable calibration with a passing SCV must be obtained before processing samples.

18.1.1.2 A BLK is an aliquot of reagent water analyzed as a sample. Results must be less that the reporting limit. If a BLK fails, contamination must be eliminated and samples re-run.

18.1.1.3 CCV's are aliquots of 5mg/L or 50 mg/L carbon (Sections 12.2.2 and 12.3.3) analyzed at the beginning of a sequence, after every tenth sample, and at the end of each sequence.

18.1.1.3.1 Included in the count are SCV, CRDL, ULCV, BLK, BS, MS MSD, and DUP vials.

18.1.1.3.2 Acceptance limits are 85% to 115% of the expected value.

18.1.1.3.3 An unacceptable CCV may be reanalyzed one time. No instrument or other analytical parameters may be changed for the reanalysis. If reanalysis is acceptable, reanalyze all samples from the last acceptable CCV. If reanalysis is unacceptable perform system maintenance and analyze all samples from run from the last acceptable CCV on a new sequence with new Method QC.

18.1.1.4 CCB are aliquots of reagent water analyzed immediately after every CCV. Results must be less than the reporting limit.

18.1.1.5 An Upper Level Calibration Verification (ULCV) is an aliquot of 10 mg/L or 100 mg/L carbon (Sections 12.2.1 and 12.3.1) analyzed immediately after the CRDL. Acceptance limits are 85-115% of the expected value. If ULCV fails, recalibrate. An acceptable ULCV must be obtained before samples may be analyzed.

18.1.1.6 CRDL is an aliquot of 0.5 mg/L or 1.0 mg/L carbon (Sections 12.2.4 and 12.3.6). Acceptance limits are 50-150% of the expected value.

18.1.2 Matrix Quality Control:

18.1.2.1 Prepare low level calibration matrix spikes by pipetting 25.0 mL of sample and 50 µL of 1000 mg/L carbon (Section 12.1) into a vial. Use an adjustable Eppendorf pipettor for adding carbon spikes. The Spike concentration is 2.0 mg/L carbon. Other spike concentrations may be prepared depending on sample concentration and matrix as follows:

Carbon Spiked, in mg/L = (1000 mg/L* A)/(25.0+A)

Where:
A = volume of 1000 mg/L carbon (Section 12.1) added, in mL

Note: For DOC samples, filter after spiking (Section 13.2.2).

18.1.2.2 Prepare high level calibration matrix spikes by pipetting 25 mL of sample and 1000 µL of 1000 mg/L carbon. Spike concentration is 40 mg/L.

18.1.2.3 A DUP is a replicate analysis. Acceptance limits are listed on the benchsheet.

18.1.2.4 Calculate relative percent difference (RPD) for MS/MSD and SAMPLE/DUP results. Acceptance limits are listed on the benchsheet. Typically a MS and MSD are analyzed since most samples have a reportable value. Calculate RPD as follows:

$$RPD_{MS/MSD} = \frac{|MS - MSD|}{(MS + MSD)} \Big| 200$$

$$RPD_{sample/dup} = \frac{|sample - dup|}{(sample + dup)} \Big| 200$$

18.1.2.4 If matrix spike recovery or duplication fails, re-run samples with an acceptable matrix spike or narrate.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running Demonstration of Capability studies (IDC and CDC). While IDCs are not instrument dependent, one is required for each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision.

19.1.1 Initial Demonstration of Capability Studies

19.1.1.1 Spike four aliquots of reagent water so concentration is in the lower half of the calibration range. Analyze the four spikes following every step outlined in the procedure. Calculate average percent recovery and relative standard deviation (RSD) using the IDC spreadsheet, located on the intranet library. Recovery must be within benchsheet SCV control limits. Relative standard deviation must be ≤ 20%.

19.1.1.2 If either criterion fails, locate and correct the source of failure and repeat the study. Repeated failure however, will confirm a problem with the procedure or technique used. If repeated failure occurs, correct the procedure or technique used and repeat the study. Samples may not be analyzed by any analyst on any instrument, until a demonstration of capability study has been successfully completed. Copies of successful IDC spreadsheets and raw data must be given to Quality Assurance.

19.1.2 Continuing Demonstration of Capability (CDC) Studies

19.1.2.1 A successful CDC must be completed annually by all analysts. CDCs may be accomplished by repeating the IDC, by using four consecutive SCV results analyzed by a single analyst during normal sample analysis, by using the last four results from the annual MDL study or by analyzing an acceptable PT sample. CDC spreadsheets and raw data must be given to Quality Assurance for inclusion in the analyst's training file.

19.2 A Method Detection Limit (MDL) study must be performed annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of and monitor all chemicals used in the laboratory.

20.2 Never dispose of a chemical without first referencing appropriate written disposal instructions for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate material safety data sheet (MSDS) when disposing of chemicals. An MSDS library is maintained on the laboratory intranet.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 (Laboratory Waste Disposal) for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998,* 5310 Total Organic Carbon, C. Persulfate-Ultraviolet or Heated-Persulfate Oxidation Method

22.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, August, 2002, Method 9060A, "Total Organic Carbon"

23.0 ATTACHMENTS

23.1 Standards Logbook Example

23.2 Raw Data Example

23.3 Instrument Logbook Example

23.4 Preparation Batch Report Example

23.5 Analysis Sequence Report Example

Attachment 23.1
Standards Logbook Example

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By
1	IN10.7-1	Phenol	IN106.16			0.05 mg/L	10ml	DI H2O	100ml	0.005 mg/L	VAS	11/21/05	11/21/05	GCA
2	IN10.7-2	↓	IN880.9			100mg/L	↓	↓	↓	100mg/L	↓	↓	↓	GCA
3	IN10.7-3		IN10.7.2			100mg/L				10mg/L				GCA
4	IN10.7-4		IN.10.7.3			10mg/L	↓	↓	↓	LCS 10mg/L	↓	↓	↓	GCA
5	IN10.7-5	Phenol	IN10.7.4			10mg/L	10ml	DI H2O	100ml	LCS 0.1mg/L	VAS	11/22/05	11/22/05	GCA
6	IN10.7-6	TOC	IN9.63.10			1000 ppm	5 ml	di H2O	200ml	25 ppm	GCA	11-28-05	12-28-05	JLB
7	IN10.7-7	TOC	IN10.4.13			10 ppm	5 ml	di H2O	100ml	0.5 ppm	GCA	11-28-05	11-28-05	JLB
8	IN10.7-8	TOC	Potassium Biphthalate	Fisher 006862A	8/15/06	dry chem	2.128g	di H2O and phosphoric acid <2	1000 ml	1000 ppm	GCA	11-28-05	5-28-04	JLB
9	IN10.7-9	Bromide	Sodium bromide			—	0.322g	DI H2O	500mL	1000 mg/L	JLB	12-1-03	6-1-04	TAM
10	IN10.7-10	↓	↓			—	0.322g	↓	500mL	LCS 1000mg/L	JLB	12-1-03	6-1-04	TAM
11	IN10.7-11		IN10.7.9			1000 mg/L	10mL		100mL	100	↓	12-1-03	12-1-03	TAM
12	IN10.7-12		IN10.7.11			100	10		↓	10		↓	↓	TAM
13	IN10.7-13		IN10.7.11			100	5			5				TAM
14	IN10.7-14		IN10.7.12			10	10			1				TAM
15	IN10.7-15		IN10.7.13			5	10			0.5				TAM
16	IN10.7-16		IN10.7.14			1.0	10			0.1				TAM
17	IN10.7-17		IN10.7.10			1000	10	↓	↓	100 LCS	↓	↓	↓	TAM
18	IN10.7-18	Bromide	IN10.7.17			100	5	DI H2O	100mL	5 LCS mg/L	JLB	12-1-03	12-1-03	TAM

file: Standard Logbook Auto.xls — page 7 of 100 — revision 2.0

Approved By: PS 1-26-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.2
Raw Data Example

Sample Results

Spl #: 3 Sample ID : ccv 1 IN25.10-6 Type : Check_Stan Date: 10/20/2008 Status: Passed
Vial #: 2 Method : 092308low - Sep 23, 2008; 01 Dilution 1 : 1 Customer ID: 00000000

Rep #	Time	TIC Area (cts)	TIC Mass (ugC)	TIC Conc (PPM)	TOC Area (cts)	TOC Mass (ugC)	TOC Conc (PPM)
1	9:50 am	-	-	-	84,152	25.257	5.051
	Avg.	-	-	-	84,152	25.257	5.051
	Std.Dev.				0		
	% RSD.				0.00		

Spl #: 4 Sample ID : ccb 1 Type : Check_Stan Date: 10/20/2008 Status: Passed
Vial #: 3 Method : 092308low - Sep 23, 2008; 01 Dilution 1 : 1 Customer ID: 00000000

Rep #	Time	TIC Area (cts)	TIC Mass (ugC)	TIC Conc (PPM)	TOC Area (cts)	TOC Mass (ugC)	TOC Conc (PPM)
1	10:01 am	-	-	-	4,373	0.593	0.119
	Avg.	-	-	-	4,373	0.593	0.119
	Std.Dev.				0		
	% RSD.				0.00		

Spl #: 5 Sample ID : scv IN25.2-2 Type : QC #1 Date: 10/20/2008 Status: Passed
Vial #: 4 Method : 092308low - Sep 23, 2008; 01 Dilution 1 : 1 Customer ID: 00000000

Rep #	Time	TIC Area (cts)	TIC Mass (ugC)	TIC Conc (PPM)	TOC Area (cts)	TOC Mass (ugC)	TOC Conc (PPM)
1	10:14 am	-	-	-	84,896	26.247	5.249
	Avg.	-	-	-	84,896	26.247	5.249
	Std.Dev.				0		
	% RSD.				0.00		

Spl #: 6 Sample ID : ccl IN25.12-2 Type : Check_Stan Date: 10/20/2008 Status: Passed
Vial #: 5 Method : 092308low - Sep 23, 2008; 01 Dilution 1 : 1 Customer ID: 00000000

Rep #	Time	TIC Area (cts)	TIC Mass (ugC)	TIC Conc (PPM)	TOC Area (cts)	TOC Mass (ugC)	TOC Conc (PPM)
1	10:27 am	-	-	-	12,770	3.189	0.638
	Avg.	-	-	-	12,770	3.189	0.638
	Std.Dev.				0		
	% RSD.				0.00		

Approved By: 1-26-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.3
Instrument Logbook Example

TriMatrix Laboratories, Inc.

Instrument #324
TOC/DOC Run Sequence

		Calibration Standard #	Conc. (mg/L)	QC Samples and Reagents	Conc. (mg/L)	Standard Number/ Reagent Number
Date:						
Analyst:				CRDL:		
Analytical Method:				CCV:		
Analysis Type:	TOC / DOC			ULCV:		
Date of Initial Calibration:				SCV:		
Element Calibration #:				SPK:		
Inst. Calibration Method #:				Sodium Persulfate:	20%	
Calibration Range:	Low / High			Phosphoric Acid:	5%	

Laboratory Sample ID	Dilution Ratio	Date/Time Sample Removed	Comments

file: Instrument 324 Sequence Log Portrait.xls page: 1 of 50 revision: 0.0

Attachment 23.4
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0707409 Page 1 of 1

Printed: 7/26/2007 12:18:10PM

Inorganic - Wet Chemistry, Waste Water, General Inorganic Prep

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0706525	TOC 5310 C	0706527	TOC 5310 C	0706567	TOC 5310 C
0706598	TOC 5310 C	0706601	TOC 5310 C	0706609	TOC 5310 C
0706610	TOC 5310 C				

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0707409-BLK1		Jul-02-07 13:33	GEH	40	40					BLANK	
0707409-BS1		Jul-02-07 13:33	GEH	40	40			7060881	40000	LCS	
0707409-MS1		Jul-02-07 13:33	GEH	25	25		0706609-01	7060878	250	MATRIX SPIKE	
0707409-MSD1		Jul-02-07 13:33	GEH	25	25		0706609-01	7060878	250	MATRIX SPIKE DUP	
0706525-01	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706525-02	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706525-03	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706525-04	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706527-05	K	Jul-02-07 13:33	GEH	40	40					[redacted]	6.27.07
0706527-08	K	Jul-02-07 13:33	GEH	40	40					[redacted]	6.27.07
0706567-01	O	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706598-01	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706598-02	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706598-03	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706598-04	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706598-05	E	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706601-01	O	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706609-01	I	Jul-02-07 13:33	GEH	40	40					[redacted]	
0706610-01	I	Jul-02-07 13:33	GEH	40	40					[redacted]	

Comments	Analyst Initials

bch_TriMatrix.rpt

Approved By: [signature] 1-26-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.5
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. ANALYSIS SEQUENCE 7072548 Page 1 of 1 Printed: 7/26/2007 12:22:59PM

Inorganic - Wet Chemistry, Water, Jul-24-07
Instrument = 198, Calibration = 7G19014

Sequence Analyses
TOC DWTR 5310 C

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client QC Type*	*Extraction Comments*
7072548-ICV1	QC		7070679		INITIAL CAL CHECK	
7072548-ICB1	QC				INITIAL CAL BLANK	
0708395-BLK1	QC				BLANK	
7072548-SCV1	QC		7070676		SECONDARY CAL CHECK	
0708395-BS1	QC				LCS	
7072548-CRL1	QC		7070801		MRL CHECK	
0707308-10	TOC DWTR 5310 C	A			[redacted]	Organic Carbon
0708395-DUP1	QC				DUPLICATE	
7072548-CCV1	QC		7070679		CALIBRATION CHECK	
7072548-CCB1	QC				CALIBRATION BLANK	

Comments:

Analyst Initials:

seq_TriMatrix.rpt

Approved By: AD 1-26-09
QA Officer

Approved By: Heather L Brady
Area Supervisor

Pace Analytical Services, Inc.
1700 Elm Street, Suite 200
Minneapolis, MN 55414

Phone: 612.607.1700
Fax: 612.607.6444

STANDARD OPERATING PROCEDURE

Analysis of Whole Air Samples for Volatile Organic Compounds by GC/MS

Reference Method: EPA Compendium Method TO-15/ TO-14 (ppbv)

SOP NUMBER:	S-MN-A-013-Rev.08
EFFECTIVE DATE:	12 April 2010
SUPERSEDES:	S-MN-A-013-Rev. 07

APPROVALS

Laboratory General Manager — Date: 06Apr2010

Laboratory Quality Manager — Date: 12Apr2010

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL. SOP IS VALID FOR ONE YEAR FROM DATE OF LAST SIGNATURE.

Signature	Title	Date

This is **COPY#**__14__ distributed on _23July2010_ by _AMS1__and is ___**CONTROLLED or** __**X**___**UNCONTROLLED.**

FIELD KIT GUIDE
FOR
PASSIVE SOIL-GAS INVESTIGATIONS
[PLEASE READ ENTIRE GUIDE BEFORE STARTING SURVEY]

I. General Information

A. BEACON is furnishing this kit to **RMT, Inc.** (RMT) specifically for use on the **Tecumseh Products site in Tecumseh, MI.** To meet the project objectives the Samplers will be retrieved **seven (7) days after installation**. Please contact BEACON following installation of the samplers at (800) 878-5510 with anticipated date when samples will arrive at BEACON's laboratory.

B. Prior to returning the Kit to BEACON, RMT should verify that the caps are tight on the Passive Soil-Gas (PSG) Samplers and that the Samplers are sealed individually in the small Sampler Bags and also in the larger Return Shipment Bag, with an adsorbent pak.

C. ***Before going to the field*** please inventory the contents of the Kit, checking them against the enclosed list to verify item counts and to become familiar with all components. (Because the components are thoroughly cleaned prior to shipment, the inventory should be conducted without opening the plastic bags.) Note that Trip Blanks are to remain sealed throughout the Survey.

D. Upon receipt of the Field Kit, BEACON requests that RMT sign and date the enclosed Chain-of-Custody Form to document receipt of the Kit. The Field Deployment Report is to be completed during the course of the survey.

E. Following completion of the survey, fill out the Chain-of-Custody Form with the following information: (i) Field Sample IDs, (ii) the name and contact phone number of the person submitting the samples, (iii) the unique number of the custody seal that will be used, and (iv) signature and date of person relinquishing samples. The Chain-of-Custody Form and Field Deployment Report are to be returned with the Field Kit to BEACON. If possible, retain photocopies for your record. Next, pack the Samplers, tools, containers, sampling caps, and requisite documentation in the Field Kit.

Note: Place the Return Shipment Bag, which contains the individually bagged PSG Samplers, in the upper tray and place the tools in the lower compartment of the Kit so they do not damage the Samplers. One trip blank should be included with each Return Shipment Bag.

Affix the tug-tight custody seal to the latch on the Field Kit, pack it in its original cardboard shipping container, and send the shipment via overnight courier (FedEx, UPS, DHL) to:

Beacon Environmental Services, Inc.
Attn: Sample Receiving
323 Williams Street, Suite D
Bel Air, MD 21014
410-838-8780

NOTE: DO NOT PACK IN THE KIT OR SHIPPING BOX STYRENE PEANUTS, NEWSPAPER, OR OTHER MATERIALS THAT COULD CONTAMINATE THE SAMPLES. PLEASE AVOID SMOKING WHILE HANDLING SAMPLERS.

II. Contents

A. This Field Kit contains the components needed for a **148**-point soil-gas survey, plus sufficient additional cartridges for **5** trip blanks (vial labeled **Trip-1 through Trip-5**, not to be opened), and **4** extra Samplers for use in the event of breakage or accidental contamination. In addition, **3** extra transport vials are provided in case a Sampler Vial breaks during retrieval. **Do not open bags until deployment.**

Code/Item		Quantity
(1)	PASSIVE SOIL-GAS SAMPLERS	**157**
(2)	EXTRA TRANSPORT VIALS	**3**
(3)	SAMPLING CAPS (in container)	**160**
(4)	CAP STORAGE CONTAINERS	**2**
(5)	TAPPING DOWELS	**1**
(6)	12″ LENGTHS OF METAL PIPE	**152**
(7)	WIRE CUTTERS	**1**
(8)	GAUZE CLOTHS	**160**
(9)	PIPE CUTTER	**1**
(10)	SCRATCH AWL	**1**
(11)	VISE GRIPS	**1**
(12)	3” x 4” PLASTIC SAMPLER BAGS (for return shipment of samples)	**160**
(13)	12” x 12” PLASTIC RETURN SHIPMENT BAG	**1**

B. In addition to the materials found in the kit, field teams will need:

- NITRILE GLOVES
- CLEAN TOWEL
- HAMMER
- ELECTRIC ROTARY HAMMER DRILL WITH:
  ½”-DIAMETER BIT WITH AT LEAST 36 INCHES OF CUTTING LENGTH and
  1¼” to 1½” DIAMETER BIT WITH AT LEAST 12 INCHES OF CUTTING LENGTH
- PIPE WRENCH (to dislodge drill bits should they become stuck)
- BALL-POINT PEN and CLIPBOARD
- PIN FLAGS, WOODEN STAKES, or OTHER LOCATION MARKERS
- FLAGGING TAPE
- BOX OF ALUMINUM FOIL

C. Additional materials necessary only for deployment through asphalt or concrete:

- DRY CONCRETE MORTAR MIX and ASSOCIATED EQUIPMENT (for temporary patching of the sample holes) including:
  SMALL PAIL, WATER, SMALL PLASTIC PUTTY KNIFE
- CHISEL or SCREWDRIVER (to remove the temporary patch)
- ASPHALT COLD PATCH or CEMENT (for final repair of the sample holes)

III. Instructions

A. GENERAL:

Deployment and retrieval of Samplers requires only one person. Separate step-by-step procedures are detailed below for sampling through vegetation or bare soils and for sampling in areas covered by asphalt, concrete, or gravel. **Keep exposure of sample cartridges to ambient air to a minimum.**

Note: Do not deploy Samplers within 10 feet of a monitoring well, penetrometer, hydropunch shaft, or other intrusive sampling apparatus that potentially creates a preferential pathway for gases.

REMEMBER: TRIP BLANKS ARE NOT TO BE OPENED.

B. SAMPLER DEPLOYMENT:

Note: Each Sampler contains two sets of adsorbent cartridges. BEACON will analyze one set per Sampler; however, the second set in each Sampler can be analyzed as a field sample duplicate. RMT will note at which locations, if any, duplicates are to be analyzed by writing separate entries corresponding to the sample location followed by the letter "D" (*i.e*, 3, 3-D, 4, 4-D) on the Chain-of-Custody Form. It is not necessary to alter the deployment pattern to have the duplicate samples analyzed. There is an additional per sample charge for analysis of any duplicates.

Vegetation or Bare Soils:

1. At each survey point, clear vegetation as necessary and, using a hammer drill and drill bit, create a 1¼"- to 1½"-diameter hole approximately 12 inches deep. Then, using the ½" drill bit, extend the hole to a three foot depth. **Note**: In areas of very organic topsoil or landscaped areas (ie, mulched areas, gardens, etc.) it is important to get beneath the organic soil layer to the underlying soil below.

2. When the holes have been drilled, take a 12-inch length of 1"-diameter metal pipe and lower it into the sample hole, being careful not to touch the inside of the pipe. Any portion of pipe above grade is cut flush with the ground surface, using the pipe cutter. With the tapping dowel and a hammer, push or tap the pipe one inch into the base of the drilled hole (see **attached figure**).

3. Remove one of the Samplers (a glass vial containing four ***hydrophobic*** adsorbent cartridges) and unwind the retrieval wire wrapped around it. Holding the capped end of the vial in one hand, pull the wire tight (to straighten it) with the other hand. Remove the solid cap on the Sampler Vial and replace it with a Sampling Cap (a one-hole cap with a screen meshing insert). Place the solid cap in the Field Kit.

Note: At each sampling location, verify that the (black) sampling cap is on the vial before installing the Sampler.

4. Lower the Sampler, open-end down, into the metal pipe approximately four inches so that the retrieval wire sticks out of the hole. Cover the open end of the pipe with a balled up **wad** of aluminum foil, pressing it tightly on top of the pipe with the tapping dowel. Next, cover the hole to grade with local soils or sand, leaving the end of the wire exposed above the surface of the ground. Using the hammer, collapse the soils above the Sampler. **Coil the wire and lay it flat on the ground surface**. Place the solid cap in the Cap Storage Container. Clearly mark the sample location with a pin flag or wooden stake.

5. Close the Field Kit, and on the Field Deployment Report record: (a) sample-point number; (b) date/time of emplacement (to nearest minute); and (c) other relevant information (*e.g.*, soil type, vegetation, proximity to potential source areas). Mark the sample location and take detailed notes (*i.e.*, compass bearings and distances from fixed reference points).

6. Move to next location.

Concrete, Asphalt, or Gravel Covered Areas:

1. At each survey point, drill a 1¼"- to 1½"-diameter hole through the asphalt/concrete/gravel to bare soil using a rotary hammer drill or comparable equipment. This hole should be approximately 12 inches deep. **Note**: When one person is performing fieldwork, it is often more efficient to drill all sample-point holes before beginning Sampler deployment.

2. When the hole through concrete/asphalt/gravel has been completed, using the ½" drill bit, extend the hole to a three foot depth. Next, take a 12-inch length of 1"-diameter metal pipe and lower it into the sample hole, being careful not to touch the inside of the pipe. Any portion of pipe above grade is cut flush with the ground surface, using the pipe cutter. With the tapping dowel and a hammer, push or tap the pipe one inch into the base of the drilled hole (see **attached figure**).

3. Remove one of the Samplers (a glass vial containing four ***hydrophobic*** adsorbent cartridges) and unwind the retrieval wire approximately six inches from the sampler, so that a coil of wire remains at the end. Remove the solid cap on the Sampler Vial and replace it with a Sampling Cap (a one-hole cap with a screen meshing insert). Place the solid cap in the Field Kit.

Note: At each sampling location, verify that the (black) sampling cap is on the vial before installing the Sampler.

4. Lower the Sampler, open-end down, into the metal pipe approximately four inches.

   If sampling through asphalt or concrete, bend the end of the wire over the top of the pipe so that the coil of wire hangs over the top and outside of the pipe. Next, plug the top of the hole with a wad of aluminum foil. Using the tapping dowel, push down the aluminum foil so it forms a seal on the metal pipe and rests ¼" below the surfacing. Cover the hole to grade with a **¼" thick** concrete patch. [**Note**: A ¼" thick patch is all that is required. If it is thicker it will be difficult to remove during retrieval.] Next, place the solid cap in the Cap Storage Container.

   If sampling through gravel, extend the retrieval wire out of the pipe and plug the pipe with a wad of aluminum foil. Using the tapping dowel, push down the aluminum foil so it forms a seal on the metal pipe. Bend the wire over the aluminum foil plug and while the wire is extended out of the hole, cover the aluminum foil with local soil or sand. **Coil the wire and lay it flat on the ground surface.** Next, place the solid cap in the Cap Storage Container.

   If a hole deeper than 12 inches is created, it will be necessary to use more than one wad of aluminum foil. In these situations, extend the wire out of the pipe. While holding onto the wire, plug the top of the pipe and hole loosely with as many wads as needed. Before inserting the last wad of foil, bend the wire so it rests below the uppermost wad of foil. This will make it easy to retrieve the Sampler during retrieval.

5. Close the Field Kit, and on the Field Deployment Report record: (a) sample-point number; (b) date and time of emplacement (to nearest minute); (c) type of surfacing and approximate thickness; and (d) other relevant information (*e.g.*, surfacing material, proximity to potential source areas). Be sure to mark the sample location and take detailed notes (*i.e.*, compass bearings and distances from fixed reference points).

6. Move to next location.

C. SAMPLER RETRIEVAL:

Prior to retrieving samples, seal each Trip Blank in a 3"x4" Sampler Bag, and place the bagged Trip Blank in a separate larger bag marked "Return Shipment Bag." One trip blank should be included with each Return Shipment Bag. Stow the sampler blocks, with the Transport vials and extra samplers, in the lower compartment of the kit. The sampler blocks are to be returned to BEACON's lab along with the samples.

Note: Each Sampler contains two sets of adsorbent cartridges. BEACON will analyze one set per Sampler; however, the second set in each Sampler can be analyzed as a field sample duplicate. RMT will note at which locations, if any, duplicates are to be analyzed by writing separate entries corresponding to the sample location followed by the letter "D" (*i.e*, 3, 3-D, 4, 4-D) on the Chain-of-Custody Form. It is not necessary to alter the deployment pattern to have the duplicate samples analyzed. There is an additional per sample charge for analysis of any duplicates.

Vegetation or Bare Soils:

1. At each sample location open the Field Kit and place it and the wire cutters within easy reach. Remove a square of gauze cloth and place it and a clean towel on the open Kit. Remove a solid cap from the Cap Storage Container and place it on the Kit, also.

2. Remove the aluminum foil plug, using vise grips and the scratch awl, if necessary, and retrieve the Sampler from the hole.

3. Holding the Sampler upright, clean the sides of the vial with the clean towel (especially close to the Sampling Cap). Remove the Sampling Cap, cut the wire from the vial with the wire cutters, and clean the vial threads completely with the gauze cloth.
   [**Note**: **Completely remove the wire to ensure the cap fits tight on the vial and no soil is returned in the field kit**.]

4. Firmly screw the solid cap on the Sampler Vial and clean the vial completely with the gauze cloth. With a **ballpoint pen** record the sample number, corresponding to the sample location, on the cap's label. [**Note**: Do not use a Sharpie marker.]

5. Return the sampling cap to the Sampling Cap container. Place the sealed and labeled Sampler Vial in the smaller 3" x 4" plastic Sampler Bag. Then place the individually bagged and labeled sampler into the larger bag labeled "Return Shipment Bag."

   Note: Each sampler must be individually bagged and placed in a Return Shipment Bag, with approximately 40 samplers and one trip blank per Return Shipment Bag.

6. On the Field Deployment Report, record: (a) date and time of retrieval (to nearest minute); and (b) any other relevant information.

7. After all samples have been retrieved, verify that the caps on each Sampler are sealed tightly and that the seals on the Sampler Bags are closed. Verify that all Samplers are stored in the Return Shipment Bag, which contains an adsorbent pak. Seal the Return Shipment Bag and place it in the upper tray of the Field Kit, and place the provided tools and materials in the lower compartment of the Field Kit.

<u>Note</u>: Please do not return the sampling caps, used pipe, or the wire with the Field Kit as they could bias the samplers. Return *all* the other materials and equipment (blocks, extra samplers, tools, containers, *etc.*).

<u>Asphalt, Concrete, or Gravel:</u>

1. At each sample point covered by gravel, clear away the soil or sand to expose the aluminum-foil plug. For those locations covered by asphalt or concrete, use a small chisel and hammer to remove the concrete patch to expose the aluminum foil.

2. Next, open the Field Kit and place it and the wire cutters within easy reach. Remove a square of gauze cloth and place it and a clean towel on the open Kit. Remove a solid cap from the Cap Storage Container and place it on the Kit, also.

3. While securely holding onto the retrieval wire, remove the aluminum-foil plug, using the scratch awl, as necessary. Holding the Sampler upright, clean the sides of the vial with the clean towel (especially close to the Sampling Cap). Remove the Sampling Cap, cut all the wire from the vial with the wire cutters, and clean the vial threads completely with gauze cloth.
   [**Note**: **Completely remove the wire to ensure the cap fits tight on the vial and no soil is returned in the field kit**.]

4. Firmly screw the solid cap on the Sampler Vial and clean the vial completely with the gauze cloth. With a **ballpoint pen** record the sample number, corresponding to the sample location, on the cap's label. [**Note**: Do not use a Sharpie marker.]

5. Return the sampling cap to the Sampling Cap container. Place the sealed and labeled Sampler Vial in the smaller 3" x 4" plastic Sampler Bag. Then place the individually bagged and labeled sampler into the larger bag labeled "Return Shipment Bag."

   Note: Each sampler must be individually bagged and placed in a Return Shipment Bag, with approximately 40 samplers and one trip blank per Return Shipment Bag.

6. On the Field Deployment Report, record: (a) date and time of retrieval (to nearest minute); and (b) any other relevant information. Return the sampling cap to the Sampling Cap container.

7. After all samples have been retrieved, verify that the caps on each Sampler are sealed tightly and that the seals on the Sampler Bags are closed. Verify that all Samplers are stored in the Return Shipment Bag, which contains an adsorbent pak. Seal the Return Shipment Bag and place it in the upper tray of the Field Kit, and place the provided tools and materials in the lower compartment of the Field Kit.

<u>Note</u>: Please do not return the sampling caps, used pipe, or the wire with the Field Kit as they could bias the samplers. Return *all* the other materials and equipment (blocks, extra samplers, tools, containers, *etc.*).

8. Fill sampling holes to grade with an asphalt cold patch or cement.

IV. Forms

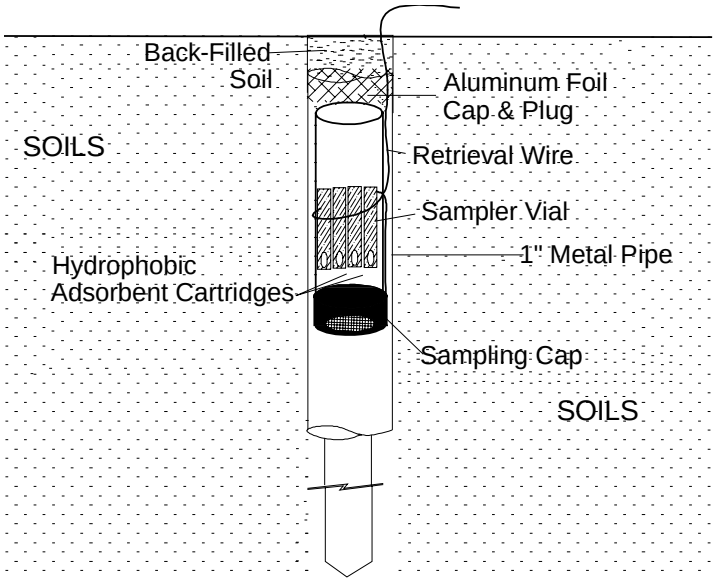
The Field Kit also contains a **Chain-of-Custody Form** and a **Field Deployment Report**.

A. The Chain-of-Custody Form is to be completed in accordance with **Section I**.

B. The Field Deployment Report is to be filled out during the Survey as indicated in **Section III**.

BEACON'S PASSIVE SOIL-GAS SAMPLER

DEPLOYMENT THROUGH SOILS



DEPLOYMENT THROUGH AN ASPHALT/CONCRETE CAP

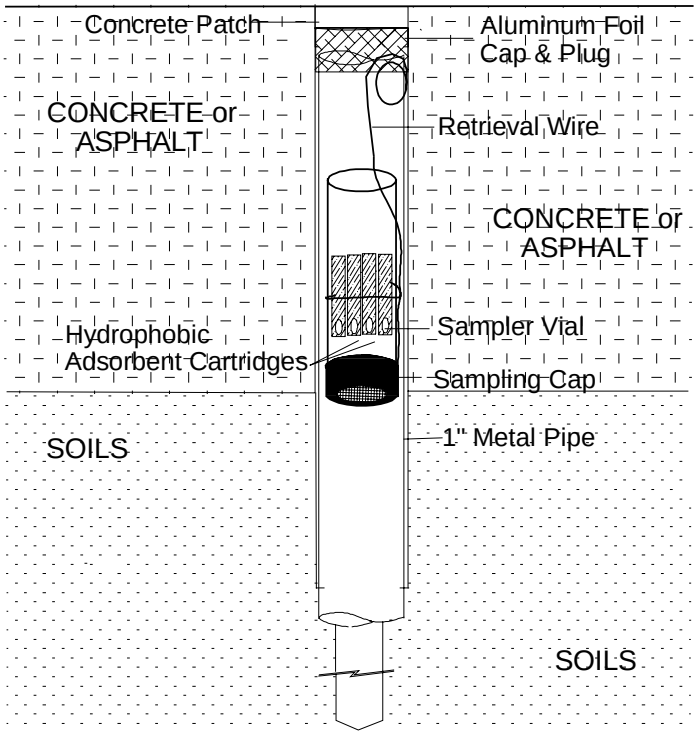


Table of Contents

1. PURPOSE

1.1 The purpose of this Standard Operating Procedure (SOP) is to provide quality control and analytical guidance for the analysis of whole air samples and soil vapor samples contained in Summa ® passivated canisters or Silco ® lined canisters (or equivalent) using gas chromatography/mass spectrometry. This SOP is based on EPA Compendium Method TO-15.

2. SCOPE AND APPLICATION

2.1 This procedure is designed to analyze whole air samples collected in Summa ® canisters or Silco ® lined canisters (or equivalent) for some of the volatile organic compounds (VOCs), or hazardous air pollutants (HAPs), found in Title III of the Clean Air Act Amendments of 1990. This SOP is related to only those VOCs that have been found to be stable when collected in Summa ® polished stainless steel canisters or Silco ® lined canisters (or equivalent). VOCs are defined as organic compounds having a vapor pressure greater than 10^{-1} Torr. Attachment I lists target VOCs applicable to this method.

2.2 This SOP is based on the EPA Compendium Method TO15 which can also be applied to TO14. As such, this SOP will serve to cover both analyses. See EPA Compendium Method TO15 Section 3 and Attachment V for compound list.

3. SUMMARY OF METHOD

3.1 Samples are received in Summa ® canisters or Silco ® lined canisters (or equivalent). The gauge pressure upon arrival is measured and recorded. The canister is then pressurized to 5 psi gauge pressure using an inert gas. The canister is connected to an autosampler tree, which concentrates the sample prior to injection into a GC/MS. The data is then analyzed for the desired volatile organic compounds.

3.2 This method addresses an extensive set of VOCs by incorporating a multisorbent, dry purge technique for water management.

3.3 An aliquot of the whole air sample is concentrated prior to gas chromatographic (GC) separation and mass spectrometry (MS) full scan detection. Samples expected to contain VOCs in a range of 0.1 parts per billion by volume (ppbv) to 500 ppbv can be analyzed by this technique.

4. INTERFERENCES

4.1 Carrier gas may contain small amounts of contaminants and is filtered prior to use in instrumentation. Other interferences are sample specific and are dealt with as they occur.

4.2 Interferences in samples can result from contamination of the canisters. To minimize this problem, processes must be implemented to ensure that the canisters are contamination free. See SOP S-MN-A-004 - Procedure for Cleaning, Certification, Leak Checking, and Preparation for Shipment of SUMMA Passivated Canisters.

4.3 Contamination of analytical equipment can also occur when samples containing high concentrations of VOCs are analyzed. The resulting “carryover” contamination will vary from system to system. The analyst needs to use best judgment when evaluating sample data following samples with large detection levels.

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical must be regarded as a potential health hazard and exposure must be as low as reasonably achievable. Cautions are included for known extremely hazardous materials

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) must be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advised.

5.3 MSDS sheets are located electronically on Groupwise and must be consulted prior to handling samples and standards.

6. DEFINITIONS

6.1 Absolute canister pressure = $P_g + P_a$, where P_g = gauge pressure in the canister (kPa, psig) and P_a= barometric pressure.

6.2 Absolute pressure - Pressure measured with reference to absolute zero as opposed to atmospheric pressure, usually expressed as kPa, mm Hg or psia.

6.3 Cryogen - A refrigerant used to obtain very low temperatures for sample concentration. A typical cryogen is liquid nitrogen (bp - 195.8°C).

6.4 Dynamic calibration - Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.

6.5 Gauge pressure - Pressure measured above ambient atmospheric pressure as opposed to absolute pressure. Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.

6.6 MS-SCAN - The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.

6.7 MS-SIM - The GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly.

6.8 Qualitative accuracy - The ability of an analytical system to correctly identify compounds.

6.9 Quantitative accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

6.10 Additional definitions may be found in the glossary of the current Pace Analytical Quality Manual.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Samples are collected into evacuated Summa ® canisters and Silco ® canisters (or equivalent). The canisters are then shipped back to Pace Analytical Services, Inc. for analysis.

7.2 Samples collected in Summa ® canisters, Silco ® canisters (or equivalent) must be analyzed within 28 days of collection.

7.3 If samples have been collected in Tedlar bags, the samples need to be transferred to a Summa Canister within 48 hours to maintain a 28 day holding time. The holding time may be extended to 72 hours per client specific QAPPS. Collection in a Tedlar bag results in higher reporting limits. See Attachments VIII-X for instructions and documentation.

7.3.1 Ohio VAP samples must be transferred to a Summa Canister within 48 hours to extend the holding time to 28 days.

8. EQUIPMENT AND SUPPLIES

8.1 Standard preparation materials for static dilution technique:

8.1.1 0.010, 0.025, 0.05, 0.1, 0.25, 0.5, 1, 5, and 10 mL gas tight syringes.

8.1.2 Neat liquid standards of at least 95%.

8.1.3 2L glass static dilution flask equipped with a Mini-inert cap.

8.1.4 Oven capable of maintaining a temperature of 65°C.

8.1.5 Summa ® passivated canisters or Silco ® lined canisters (or equivalent), six liter or fifteen liter capacity.

8.1.6 High accuracy dual pressure/vacuum gauge.

8.1.7 Nitrogen

8.1.8 Organic free water.

8.2 Analytical instrumentation.

8.2.1 Agilent Technologies 6890N gas chromatograph equipped with a split/splitless injection port and electronic pressure control (EPC) or equivalent.

8.1.2.1 Gas chromatograph.Suggested Operating Parameters:

1) Initial temp: 40°C for 2.0 min.
2) Ramp A: 8°C/min to 150°C
3) Ramp B: 15°C/min to 200°C
4) Hold 2 min
5) EPC Pressure: 9psi
6) Temp 250°C
7) Split Flow 20mL/min

8.1.2.2 Injection port parameters.

1) EPC pressure: 9 psi
2) Temperature: 250°C
3) Purge valve:Initial value On, Off time 0.0 min.
4) Split flow: 20 mL/min.

8.2.2 J & W Scientific DB-5 60m x 0.32mm capillary column or DB-624 60m x 0.32mm with a 1.8 µm film thickness or equivalent.

8.2.3 High purity grade high-pressure helium cylinder for column carrier gas equipped with a dual stage pressure regulator.

8.2.4 Hewlett Packard 5973 Mass Selective Detector, or equivalent with Chemstation operating software and WinTarget data processing software or equivalent.

8.2.4.1 Suggested Mass spectrometer parameters:

1) Electron volts: 70 nominal
2) Scan range: 29 to 300 amu
3) Scan time: At least 2 scans/peak, not to exceed 1 sec/scan
4) Interface temp: 250°C
5) The GC/MS system must be set up to meet manufacturer's specification. The mass calibration and resolution of the GC/MS are verified by the analysis of the tune standard, p-bromofluorobenzene (BFB). For more information refer to the Chemsystem User's Guide and the GC/MS User's Guide.

8.2.5 Entech 7100A pre-concentrator with 7016 canister manifold autosampler.

8.2.6 Entech Pre-Concentrator suggested settings:

8.2.5.1

During Concentration	Temperature (°C)
Module No. 1, Glass Bead Cryotrap	-150
Module No. 2, Sorbent Packed Cryotrap	-20
Focusing Trap	-160

8.2.5.2

Desorb/Transfer/Inject	Preheat (ºC)	Final Temp(ºC)
Module No. 1, Glass Bead Cryotrap	10	10
Module No. 2, Sorbent Packed Cryotrap	50	180
Focusing Trap	N/A	N/A

8.2.5.3

Media Concentrated/Transferred	Volume (cc)	Flow Rate (sccm)
Internal Standard & Surrogate	50	200
Sample	25 to 500	250
Sweep/Dry Purge	75	100
Transfer to Packed Column	40	10

8.2.5.4 Sample Transfer

Line Conditioning Sample Flush Before Trapping	20 sec
Carrier Flush Before Trapping	2 to 4 min.
Sample Transfer to Focusing Trap	2 to 4 min.
Sample Injection	2 to 5 min.

8.2.5.5

System Bakeout	Temperature (ºC)	Time (min.)
Module No. 1	150	10
Module No. 2	190	10

8.2.5.6

Regulated Zones	Temperature (ºC)
8-Port Valve	100
GC Transfer Line	110
Manifold Transfer Line	100
16-Position Select Valve	100
Sample Container	Ambient

9. REAGENTS AND STANDARDS

9.1 Calibration Standard: The calibration standard is purchased in the form of a pressurized cylinder from SPECTRA GASES, INC. This is a custom mix that includes all compounds of interest at 1ppmv.

9.1.1 2 PPBV: Using the 1000cc gas tight syringe, pull 90cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 2 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

9.1.2 20 PPBV: Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

9.1.3 Suggested Ical levels and preparation:

Ical Level	Concentration	Calibration Standard	Amount of Calibration Standard Used
Level 1	0.10 ppbv	2.0 ppbv std	25 cc
Level 2	0.20 ppbv	2.0 ppbv std	50 cc
Level 3	1.00 ppbv	20.00 ppbv std	25 cc
Level 4	5.00 ppbv	20.00 ppbv std	125 cc
Level 5	10.00 ppbv	20.00 ppbv std	250 cc
Level 6	20.00 ppbv	20.00 ppbv std	500 cc

9.2 Initial Calibration Verification (second source standard): The laboratory control standard is purchased in the form of a pressurized cylinder from a source independent of the calibration mix (Custom Gas Solutions, or equivalent). This is a custom mix that includes all compounds of interest at 1ppmv. See Attachment I for the list of compounds present in the standard.

9.2.1 20 PPBV: Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister

volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

9.2.2 The ICV is prepared by taking 250 cc from the above 20 ppbv standard and delivering it to a 15 L canister that has been humidified according to 9.2.1.

9.3 Internal Standard/ Surrogate/ BFB Standard 100ppbv: The internal, surrogate, and BFB standards are purchased as neat standards from specific vendors; such as Chem-Service, Sigma-Aldrich or equivalent.

9.3.1 To prepare a neat/cocktail standard: Using Entech Static Dilution software, enter barometric pressure, room temperature, flask temperature, flask volume, canister pressure, canister volume, flask concentration and desired final concentration (100ppbv). The software calculates approximate transfer volume 1 (vial to flask), then transfer volume 2 (flask to canister)*. Note: Standard canister (6 L or 15 L) must be cleaned, evacuated, and humidified with 50ul H2O before being used.

Example: Barometric Pressure: 29.92
Room Temperature : 24 °C
Flask Temperature : 65 °C
Flask Volume : 2000 ml
Canister Pressure : 30 psig
Canister Volume : 15,000 ml (15 L)
Flask Concentration : 520.015 PPM

*The software calculates transfer volume to the 1/10000th. The volumetric syringes are calibrated to 1/10th of a decimal place. Therefore, the analyst must adjust the volume to a measurable amount prior to standard preparation.

9.3.2 Next, pressurize the 15 L canister 30 psig with clean nitrogen. This yields a final concentration of 100 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

9.3.3 The tune standard, Bromofluorobenzene (BFB), must be 50ng or less on column.

9.3.3.1 The tune standard can be combined with the CCV standard so long as all criteria can be evaluated and met.

9.3.4 Internal standard compounds and surrogate standard compounds are used in the analysis.

9.3.4.1 Internal Standards: 1,4-Difluorobenzene and Chlorobenzene-d5

9.3.4.2 Surrogates: Hexane-d14, Toluene-d8, and 1,2-Dichlorobenzene-d

9.4 Standard Canister Preparation:

9.4.1 Static Dilution Technique

9.4.1.1 Summary: Standard preparation is accomplished by injecting an aliquot of liquid standard cocktail into a static dilution vessel. The static dilution vessel is held at a temperature of 65^{o}C. The liquid standard vaporizes and is quickly vented to come to equilibrium. An aliquot is removed and injected into a canister. The canister is then pressurized with nitrogen to a pre-established final pressure.

9.4.1.2 Procedure

9.4.1.2.1 The volume of a clean 2L round-bottom flask, modified with a threaded glass neck to accept a Mininert septum cap, is determined by weighing the amount of water required to completely fill up the flask. Assuming a density of 1 g/mL for water, the weight of the flask in grams when filled with water is taken as the volume of the flask in milliliters.

9.4.1.2.2 The dried flask is flushed with nitrogen. After a few minutes, the glass neck is immediately capped with a Mininert septum cap.

9.4.1.2.3 The flask is placed in a 65°C oven and allowed to equilibrate at that temperature for about 15 minutes. Predetermined aliquots of liquid standards are injected into the flask making sure to keep the flask temperature constant at 65°C.

9.4.1.2.4 The contents are allowed to equilibrate in the oven for at least 15 minutes. To avoid condensation, syringes must be preheated in the oven at the same temperature prior to withdrawal of aliquots.

9.4.1.2.5 Sample aliquots may then be taken from the static dilution flask for introduction into a clean, evacuated canister. The canister is then filled to a final predetermined pressure. An aliquot or aliquots totaling greater than 1 percent of the flask volume will be avoided.

9.4.1.2.6 The concentration of each component in the flask is calculated using Equation 1:

Equation 1

$$\text{Concentration}(\text{mg/L}) = \frac{(V_i)(d)}{V_f}$$

where: V_i =Volume of liquid neat standard injected into the flask in mL;
d=Density of the liquid neat standard in mg/mL;
V_f =Volume of the flask in liters.

Caution: In the preparation of standards by this technique, make sure that the volume of neat standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask.

9.4.1.2.7 The concentration in ppbv of each component in the flask is determined using Equations 2 and 3 as follows:

9.4.1.2.7.1 First determine the volume of the compound as a gas using Equation 2:

Equation 2

$$V = \frac{nRT}{P} \quad \text{where,} \qquad n = \frac{(V_i)(d)}{M}$$

where, V=Volume of injected compound at STP in liters;
n=Moles;
R=Gas constant (0.08206 L-atm/mole ^{o}K);
T=Ambient temperature in ^{o}K;
P=Ambient pressure in atm;
V_i=Volume of liquid neat standard injected into the flask in mL;
d=Density of the neat standard in g/mL;
M=Molecular weight of the compound in g/mole.

9.4.1.2.7.2 Now calculate the concentration in the flask in ppbv using Equation 3:

Equation 3

$$\text{ppbv} = \frac{V}{V_f}(10^9)$$

where: V=Gas volume of compound as determined in Eq. 8 in liters;
V_f=Volume of static dilution flask in liters.

9.4.1.2.8 The concentration in ppbv of each compound in the canister can be determined using Equation 4:

Equation 4

$$\text{ppbv} = \frac{(V_i)(C_x)}{V_c}$$

where: V_i=Volume removed from static dilution flask and injected into the canister in liters;
C_x=Concentration of compound x in the static dilution flask in ppbv;
V_c=Final canister volume in liters.

9.4.1.2.9 Entech Standards Preparation has a database of compounds and their properties. The program does the necessary conversions of units and calculations (equations 1-4) to yield the amounts of neat standard put in the standard cocktail, the amount of cocktail spiked into the 2L flask, and the aliquot taken from the 2L flask to the final canister. This program can be used to make any gas standard from neat liquid standards.

9.4.1.2.10 See Attachment VI for a single sheet summary of Air Standard Preparation.

10. CALIBRATION

10.1 INSTRUMENT TUNE

10.1.1 It is necessary to establish that the GC/MS system can produce tuning and standard mass spectral criteria prior to analyzing standards or samples. The GC/MS is set up according to the manufacturer's specifications. The MS source and mass filter are adjusted by monitoring the mass spectra of PFTBA.

10.1.2 Before any standard, blank, or sample analysis can occur using the GC/MS system, it must be demonstrated that the GC/MS is capable of producing compliant spectra when p-bromofluorobenzene (BFB) is analyzed. Attachment II lists the required spectral criteria.

10.1.3 Procedure

10.1.3.1 Prepare a standard solution of BFB at a concentration that allows the collection of 50ng or less under the optimized concentration parameters (see Section 9.3)

10.1.3.2 The BFB is introduced into the system through microscale purge and trap.

10.1.3.3 Evaluate the BFB spectrum.

10.1.3.3.1 The spectrum of BFB must be acquired by averaging three scans; the apex and the scans that immediately proceed and follow the apex.

10.1.3.3.2 Background subtraction is accomplished using a single scan taken before the BFB peak.

10.1.3.3.3 The instrument performance check must be analyzed initially and once every 24-hour period. The tune period begins at the time of injection of the BFB.

10.1.3.4 If the BFB spectrum meets the criteria listed in Attachment II, standard and sample analysis may begin.

10.1.3.5 If the BFB spectrum fails to meet the criteria listed in Attachment II, the MS must be retuned. Repeated failures may indicate the need for MS maintenance such as cleaning the ion source.

10.2 INITIAL CALIBRATION

10.2.1 All standards, blanks, spikes, and samples must be analyzed using the same conditions. A calibration curve must consist of a minimum of 5 standards (6 for quadratic) and spans the expected monitoring range established for each compound of interest to determine instrument response and linearity. The lowest level of the curve must be at or below the reporting limit for each analyte. A typical calibration curve can cover a range from 0.1 to 20 ppbv. Section 9.1.3 contains standard preparation information.

10.2.2 Calibration is performed using the internal standard technique. See Attachment III for internal standard groups. The data is evaluated using WinTarget.

10.2.3 Initial Calibration Verification (Section 9.2): a second source standard must be analyzed following an initial calibration curve which contains all the analytes of interest. The spike level of the ICV must be near the midpoint level of the calibration curve. The ICV is considered to pass if the recoveries of 95% of the analytes fall within 60-140% and no more than 5% fall within 50-150%.

10.2.4 The ENTECH 7000 Concentrator automatically adds a specified concentration of internal standards and surrogates (Section 9.3) to each analysis during trapping.

10.2.5 Using the Target data processing software, evaluate the calibration data.

10.2.6 Calculations

10.2.6.1 Relative Response Factor (RRF): Tabulate the area response of the primary ion (Attachment III) for each compound and the associated internal standard. Use the internal standard, which has a retention time nearest to the compound of interest. Calculate the relative response factors (RRF) for each compound using Equation 5:

Equation 5

$$\text{Relative Response Factor (RRF)} = \frac{(A_x)(C_i)}{(A_i)(C_x)}$$

where, A_x=Area of the primary ion for compound x to be measured;
A_i=Area of the primary ion for the internal standard associated with compound x;
C_i=Concentration of the internal standard in ppbv;
C_x =Concentration of compound x to be measured in ppbv.

10.2.6.2 Mean Relative Response Factor. Calculate the mean RRF for each compound using the RRF from the five (or six, where n=6)-point calibration using Equation 6:

Equation 6

$$\overline{R_f} = \frac{\sum_{n=5} R_f}{n}$$

where, $\overline{R_f}$ =Average relative response factor;
R_f=Relative response factor from calibration curve;
n=Number of data points.

10.2.6.3 Standard Deviation ($\sigma_{(n-1)}$).

Equation 7

$$\sigma_{(n-1)} = \sqrt{\sum_{i-1}^{n} \frac{(x_i - \bar{x})^2}{(n-1)}}$$

10.2.6.4 %Relative Standard Deviation (%RSD). Using the average RRF from Equation 6 and the standard deviation from Equation 7, calculate the %RSD using Equation 8:

Equation 8

$$\%RSD = \frac{S_{(n-1)}}{\overline{R_f}} x100$$

10.2.6.5 Mean Area response for Internal Standard

Equation 9

$$\bar{y} = \sum_{i=1}^{n} \frac{y_i}{n}$$

where $\bar{y}$ = mean area response

y = Area response for the internal standard for each initial calibration standard

10.2.6.6 If a linear regression is used, the regression will produce the slope and intercept terms for a linear equation according to Equation 10:

Equation 10

$$y = ax + b$$

where: y = instrument response (peak area or height)
a = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = the intercept, do not include the origin (0) as a calibration point

10.2.6.7 To calculate the sample concentration by the internal standard method using the linear regression equation, use Equation 11:

Equation 11:

$$C_s=[(A_sC_{is}/A_{is})-b]/a$$

where: As = Area of the peak for the target analyte in the sample
Ais = Area of the peak of the internal standard
Cs = Concentration of the target analyte in the calibration standard
Cis = Concentration of the internal standard
a = Slope of the line (also called the coefficient of Cs)
b = The intercept

10.2.6.8 To calculate the coefficient of determination (or r^2) for a quadratic curve fit, use Equation 12:

Equation 12:

$$COD = \frac{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{n}(y_{obs} - Y_i)^2}{\sum_{i=1}^{n}(y_{obs} - \bar{y})^2}$$

where: y_{obs} = Observed response for each concentration from each initial calibration standard

$\bar{y}$ = Mean observed response from the initial calibration (See equation 6)

Y_i = Calculated response at each concentration from the initial calibration (See Equation 5)

n = Total number of calibration points in the equation, 6 points for quadratic

p = Number of adjustable parameters in the polynomial equation

10.2.6.9 Calculate the sample concentration by the internal standard method using the quadratic regression by comparing peak heights to the calibration curve.

Regression equation (quadratic):

$$y = ax^2 + bx + c$$

10.2.7 Technical Acceptance Criteria.

10.2.7.1 The %RSD for all calibrated target compounds must be ±30% with no more than 2 compounds at ±40%. Alternately, linear regression may be used with an r^2 value of 0.995 or greater. A quadratic curve may be utilized if the r^2(equals COD in Equation 12) value is 0.990 or greater and six calibration points are included in the curve. Curves must not be forced through zero. *For Ohio VAP: quadratic curve fit will only be used for analytes that have historically exhibited nonlinear response.*

10.2.7.2 The area response for each internal standard in each calibration level must be within 40% of the mean area response over the calibration range. The RRT of each compound must agree within ± 0.06 RRT units of the average RRT from the initial calibration curve

10.2.8 Corrective Action

10.2.8.1 If the technical acceptance criteria fail for the initial calibration curve, inspect the system for any possible leaks. A high baseline and reduced response may be indicative of a leak.

10.2.8.2 Examine the response factors of each calibration level. If the response factors of all the compounds for one level appear to be significantly different, analyze that same level calibration standard again.

10.2.8.3 If the same results occur after reanalysis, a new standard canister must be made and analyzed.

10.2.8.4 If a leak or other system problem cannot be found, it may be necessary to clean the ion source or perform column maintenance.

10.2.8.5 No samples can be analyzed until a compliant initial calibration curve has been established and verified against a second source standard or technical justification given for the analysis to continue.

10.2.8.6 Recalibration must be performed if any major change has been made to the GC/MS system such as replacing the GC column, cleaning the MS source or repair.

10.3 Continuing Calibration Verification

10.3.1 The initial calibration curve for each compound of interest must be checked and verified before sample analysis can occur. This is accomplished by analyzing a continuing calibration verification (CCV) standard at 10 ppbv. (See Section 9.1)

10.3.2 The CCV is analyzed after a compliant tune once every 24-hour period during sample analysis.

10.3.3 Calculations

10.3.3.1 Calculate the RRF for each target compound from the daily calibration standard using Equation 5.

10.3.3.2 Percent Difference (%D). The % D in the RRF of the daily RRF of an individual compound compared to the mean RRF for that compound in the most recent calibration curve is determined as follows:

Equation 13

$$\%\mathrm{D} = \frac{\left|R_i - R_c\right|}{R_i}(100)$$

where, R_i=The average RRF from the initial calibration curve for compound *x*;
R_c=RRF for compound *x* from the daily calibration standard.

10.3.4 Technical Acceptance Criteria

10.3.4.1 The %D for each target compound in the daily calibration standard must be less than or equal to 30 percent.

10.3.4.2 The RRT of each compound must agree within ± 0.06 RRT units of the average RRT from the initial calibration curve

10.3.4.3 For TO14 only analysis, the CCV criteria must be ±10%.

10.3.5 Corrective Action

10.3.5.1 If the CCV does not meet criteria, the system and standards must be evaluated for potential problems. If a problem is isolated and corrected, attempt to run a second CCV. If the second attempt also does not meet criteria, perform further necessary troubleshooting and maintenance.

10.3.5.1.1 Check pressure on the standard canister.

10.3.5.1.2 Check system for leaks.

10.3.5.1.3 Check to see that standards were made correctly.

10.3.5.2 If corrective action attempts fail or two consecutive CCV do not meet criteria, then a new calibration curve must be analyzed.

10.3.5.3 Samples will not be analyzed until CCV criteria has been met or technical justification given for the analysis to continue.

11. PROCEDURE

11.1 ANALYTICAL SEQUENCE

The following is the GC/MS analytical sequence for samples each 24-hour period:

11.1.1 Instrument tune using (BFB) see Section 10.1

11.1.2 Initial Multi-Point Calibration or CCV see Section 10.2 or 10.3

11.1.3 ICV or Laboratory Control Sample (LCS) see Section 10.2.3 and 11.3

11.1.4 Laboratory Method Blank

11.1.5 20 field samples

11.1.6 Sample duplicate, minimum 1 in 20 samples

11.1.7 Any necessary dilutions from previously analyzed samples

11.1.8 In the event that time remains in the 24 hour tune period, an additional blank and LCS must be analyzed in order to analyze additional reportable samples.

11.2 BLANK ANALYSIS

11.2.1 A clean canister filled with humidified nitrogen is analyzed on the GC/MS system to demonstrate that the system is free of interferences.

11.2.2 The Method Blank is prepared in the same manner as any standard or sample and analyzed in the same manner.

11.2.3 A Method Blank is analyzed once every 24-hour period or every 20 samples, whichever comes first.

11.2.4 The Method Blank is analyzed after the daily calibration standard.

11.2.5 An instrument blank may need to be analyzed after any sample that has known VOCs present that exceed the upper calibration limit of the method to demonstrate that the system is free of possible carryover effects. When possible, historical data can be used to determine if there will be high levels of contaminants present that may cause carry over in the system.

11.2.6 See Equation 16 for the calculation on how to determine the concentration present.

11.2.7 See Section 12 for technical acceptance criteria and corrective actions.

11.3 LABORATORY CONTROL SAMPLE (LCS)

11.3.1 The laboratory control standard is prepared from the same standard as the calibration standard (20ppbv) as outlined in section 9.1.2. The LCS will be analyzed at a minimum of 1 in every 20 samples.

11.3.2 Calculations

11.3.2.1 Field sample calculations in Section 11.7 also apply to the LCS.

11.3.2.2 Calculate the percent recovery of the LCS using Equation 14:

Equation 14

$$\text{Percent Recovery} = \frac{C_q}{C_a}(100)$$

where: C_q=Quantitated concentration of compound *x* in ppbv;
C_a=Actual concentration of compound *x* in ppbv.

11.3.3 See section 12 for technical acceptance criteria and corrective actions.

11.4 SAMPLE ANALYSIS

11.4.1 Upon receipt, the canister pressure of each sample is measured and recorded on the canister sample tag.

11.4.1.1 If the canister pressure is less than 5 psig, the canister pressure must be increased before analysis can occur.

11.4.1.1.1 Add clean nitrogen or helium gas to the sample canister. For a six liter canister, 5 psig is the desired final pressure. A one liter canister requires a final pressure of 10 psig for adequate sample volume for analysis.

11.4.1.1.2 Record the final canister pressure on the canister sample tag noting which gas was added. Also, note the information in the final analytical results report.

11.4.1.1.3 Calculate the resultant dilution factor using Equation 15:

Equation 15

$$\text{Dilution Factor} = \frac{P_f}{P_i}$$

where: P_f=Final canister pressure in psig;
P_i=Initial canister pressure in psig.

See attachment V for the application of dilution factors for filling canisters.

11.4.1.1.4 This dilution factor is applied to Equation 16.

11.4.2 Once the GC/MS system is demonstrated to be in control, an aliquot of the air sample is removed from the canister and pre-concentrated using the Entech 7100A pre-concentrator and 7016 autosampler manifold.

11.4.3 Analyze the samples under the same operating conditions as the instrument calibration and quality control samples.

11.4.4 Analyze a duplicate sample for every 20 samples analyzed.

11.4.5 If time remains in the 24-hour tune period in which an initial calibration was performed, samples may be analyzed without the analysis of a daily calibration standard.

11.4.6 If the tune period has expired, an instrument performance check standard and daily calibration standard must be analyzed before samples can be analyzed.

11.4.7 If time remains in the tune period after a batch of no more than 20 samples and its re-runs have been analyzed, additional samples may be analyzed after a new LCS and method blank have been analyzed.

11.4.8 Technical Acceptance Criteria can be found in Section 11.9

11.5 QUALITATIVE ANALYSIS

11.5.1 The compounds listed in Attachment I are identified by an analyst competent in the interpretation of mass spectra. Sample mass spectrum is compared to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the target compound identifications: (1) elution of the sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

11.5.2 The relative retention time (RRT) of the sample component must agree within ± 0.06 RRT units of the RRT of the standard component using the continuing check standard as reference.

11.5.3 Standard and sample mass spectra are compared using reference spectra obtained on the GC/MS system being used. The mass spectra used for comparison are from the same standard as that being used for RRT comparison. Mass spectral requirements are as follows:

11.5.3.1 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

11.5.3.2 The relative intensities of ions specified above must agree within ± 20% between the standard and sample spectra.

11.5.3.3 Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process favors false positive.

11.5.4 Non-target sample components shall be library searched using the latest NIST library for the purpose of tentative identification. These components are referred to as TICs Tentatively Identified Compounds) and will be noted as such in any final report with a qualifier of "J" unless the client specifies differently. The "J" qualifier indicates an estimated value. Guidelines for identification are as follows:

11.5.4.1 Characteristic ions in the reference spectrum (ions greater than 10% of the most abundant ion) must be present in the sample.

11.5.4.2 The relative intensities of the major ions must agree within ± 20%.

11.5.4.3 Ions present in the sample spectrum but not in the reference spectrum must be reviewed for background contamination or presence of co-eluting peaks.

11.5.4.4 If in the technical judgment of the analyst, no valid identification can be made, the compound will be reported as an unknown with possible classification such as hydrocarbon.

11.5.4.5 TIC searches are reported only upon client request.

11.6 Identified target analytes shall be quantitated using the internal standard method using the EICP area of the characteristic ions of analytes listed in Attachment III. This ion is referred to as the quantitation ion.

11.7 The RRF from the continuing calibration standard analysis is used to quantitate samples and blanks. Calculate the concentration of the sample component using Equation 16:

Equation 16

$$C_x = \frac{(A_x)(C_i)(D_f)}{(A_i)(R_x)}$$

where: C_x=Concentration of compound *x* in ppbv;
A_x=EICP area of the quantitation ion for compound *x*;
C_i= Concentration of the internal standard associated with compound *x* in ppbv;
D_f=Dilution factor from Equation 12 (if no dilution was performed, D_f equals 1.)
A_i=EICP area of the quantitation ion for the internal standard associated with compound *x*;
R_f=Average RRF for compound *x* from the most recent calibration curve.

11.8 The internal standard method of quantitation is also used to determine an estimated concentration for Tentatively Identified Compounds (TIC). The nearest internal standard to the TIC is used as a reference to estimate the concentration of the TIC. If the nearest internal standard exhibits interferences, the next closest internal is used. The estimated concentration is obtained using Equation 16 with the following exceptions:

A_x = Total ion chromatogram area of the TIC,
A_i = Total ion chromatogram area of the specific internal standard;
R_f = 1.0

Estimated TIC concentrations are flagged with a qualifier of "J" which indicates that the quantitated amount is an estimate.

11.9 GENERAL TECHNICAL ACCEPTANCE CRITERIA

11.9.1 For data to be reported without qualification, the following criteria must be met for all samples, CCVs, method blanks, and quality control samples:

11.9.1.1 The EICP area response for each internal standard must be within ±40% of the EICP area response in the most recent CCV. See Attachment III for a list of analytes and assigned internal standards.

11.9.1.2 The retention time for each of the internal standards must be ±0.33 minutes of each of the IS retention times in the most recent CCV.

11.9.1.3 Recoveries for surrogate standard compounds (where required) must fall within ±30% of the true value.

11.9.2 If the technical acceptance criteria is not met, the instrument calibration, laboratory quality control samples and/or associated samples must be reanalyzed to confirm results. See Section 10 for corrective action for calibration failures and Section 12 for all other samples (including QC).

11.9.2.1 If the surrogates don't fall within laboratory generated limits, the system must be checked to determine the cause of the failures. The sample must be reanalyzed to confirm the results unless there is definitive proof of matrix interference. The data will be qualified accordingly.

12. QUALITY CONTROL

12.1 Three performance criteria are used to demonstrate method validity which are as follows: (1) method detection limit (MDL), (2) replicate precision, and (3) accuracy - % recovery of LCS.

12.1.1 The MDL is determined following the guidelines set forth in S-ALL-Q-004.

12.1.2 Replicate precision is based upon the relative difference between replicate measurements of the same sample expressed as a percentage,

[(Measurement #1 - Measurement #2) x 100%]/Average of 2 measurements.

12.2 A Method Blank analyzed once every 24-hour period or every 20 samples, whichever comes first.

12.2.1 Technical Acceptance Criteria

12.2.1.1 The blank must not contain any target analyte at a concentration greater than its reporting limit and must not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.

12.2.2 Corrective Action

12.2.2.1 If a Method Blank fails acceptance criteria, the source of the contamination must be identified and eliminated.

12.2.2.2 If a source of contamination is corrected, another Method Blank must be prepared and analyzed to verify that the problem has been resolved.

12.2.2.3 However, if the contaminant cannot be eliminated and samples are analyzed, samples containing the same artifact as that found in a blank must be flagged accordingly.

NOTE: For Ohio VAP samples, if the detection is above the reporting limit and corrective actions do not result in acceptable data, the samples must be re-analyzed undiluted. If re-analysis is not possible due to depleted sample volume, then contact the client for further instructions. The client may want to re-submit the sample or have the lab qualify the data and narrate as appropriate

12.3 A LCS must be analyzed once every 24-hour period or every 20 samples, whichever is more frequent.

12.3.1 Technical Acceptance Criteria

12.3.1.1 The percent recovery for each analyte in the LCS must be within the internally generated QC limits.

12.3.2 Corrective Action

12.3.2.1 If a LCS fails to meet the recovery limit criteria, inspect the system for the possibility of a poor sampling.

12.3.2.2 If the LCS fails and no error in sampling was found, the system must be recalibrated. It may be necessary to prepare new calibration standards.

12.3.2.3 If the samples cannot be reanalyzed, qualify the data accordingly.

12.3.2.4 For Ohio VAP samples, if the outlier is an analyte of interest and corrective actions do not result in acceptable data, the samples must be re-analyzed. If re-analysis is not possible due to depleted sample volume, then contact the client for further instructions. The client may want to re-submit the sample or have the lab qualify the data and narrate as appropriate

12.4 Duplicate sample analysis is performed once per 20 samples. See Attachment VII for exception to this criteria.

12.4.1 The RPD between the sample and the sample duplicate must be < 30% and can be calculated using Equation 17:

Equation 17

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100$$

Where: RPD = Relative Percent Difference

A = Sample Value
B = Duplicate Value

12.4.2 If the RPD fails to meet criteria, the instrument must be evaluated to determine if there was an error with the analysis. If there is not evidence of malfunction, the samples must be reanalyzed to confirm results. If the data confirms, report the original data and qualify accordingly.

12.5 Internal Standards

12.5.1 Technical Acceptance Criteria – See 11.9.1.

12.5.2 Corrective Action

12.5.2.1 Examine the instrument for possible errors or malfunctions and correct any that are discovered. Re-analyze the samples and QC and report the acceptable data

12.5.2.2 If there is no evidence of error or malfunction, re-analyze the affected QC and samples. If the data confirms, report the original data and qualify accordingly.

12.5.2.3 Unless a matrix interference was detected, Ohio VAP samples must be re-analyzed undiluted.

12.6 Surrogates

12.6.1 Technical Acceptance Criteria

12.6.1.1 Surrogates are not required by the TO15 method. Therefore they are only be analyzed upon client request.

Note: Ohio VAP samples must not include surrogates.

12.6.1.2 If surrogates are requested by the client, they must meet internally generated limits (±30%).

12.6.2 Corrective action

12.6.2.1 If surrogates do not meet the recovery limits, re-analysis is performed to confirm the outlier and data is qualified as necessary.

12.6.2.2 One exception to the above corrective action occurs if the sample is non-detect for the analytes of interest and the surrogate fails above the recovery limits. In this situation, the data is biased high and is unaffected by the high surrogate recoveries. The sample is qualified accordingly without re-analysis.

13. METHOD PERFORMANCE

13.1 There are several requirements that must be met to ensure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file within the QA office

13.1.2 An initial demonstration of capability (IDC) must be performed per SOP All-Q-020. A record of the IDC will be maintained in his/her file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual minimum detection limit (MDL) study following SOP ALL-Q-004 is completed for this method and whenever there is a major change in personnel or equipment. Results are stored in the QA Office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed to demonstrate continuing competence according to SOP S-ALL-Q-010 – Proficiency Testing Program.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes will reflect anticipated usage and reagent stability.

14.2 The Environmental Protection Agency (USEPA) requires that laboratory waste management practice be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. For further information on waste management consult SOP ALL-S-001, or equivalent replacement.

15. REFERENCES

15.1 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition; USEPA, January 1999; EPA/625/R-96/010b. Compendium Method TO15.

15.2 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition; USEPA, January 1999; EPA/625/R-96/010b. Compendium Method TO14A

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA, ETC

16.1 ATTACHMENT I: Target Compound List

16.2 ATTACHMENT II: Required BFB Key Ions and Ion Abundance Criteria

16.3 ATTACHMENT III: Characteristic Ions for Target Compounds

16.4 ATTACHMENT IV: Calibration of THC as Gas

16.5 ATTACHMENT V: Canister Dilution Factors

16.6 ATTACHMENT VI: Air Laboratory Standard Preparation Procedures

16.7 ATTACHMENT VII: Procedures for Analyzing MPCA Samples

16.8 ATTACHMENT VIII: Procedure for Tedlar Bags

16.9 ATTACHMENT IX: Tedlar Sign-off Logbook

16.10 ATTACHMENT X: Tedlar Bag Transfer Log

16.11 ATTACHMENT XI: Common Logbook Abbreviations

17. REVISIONS

Revision Number	Reason for Change	Date
MN-A-013-rev3	Sec. 9-Remove heating band. Sec. 10 Add surrogate standard compounds standard**.**Sec. 11. Add second source standard to verify calibration and preparation of the surrogate standard canister. Sec. 12 Add second source verification of calibration to analytical sequence and that surrogate standard compounds will fall within internally generated limits. Attachment IV reformatted	10/31/05
MN-A-013-rev.2	Reformat to conform to corporate model. Add reference to MDL SOP: SOP ALL-P-04 to section 14.	3/28/05
S-MN-A-013-rev.04	Reformat Section 7 to new style Re-worded multiple sections to make SOP more concise Eliminated multiple sections to reduce repetition. Combined with TO14 SOP since one SOP covered both via the TO15 Compendium Method Attachment I was edited to reflect TO14 compounds and reporting limits were removed. Added attachments V and VI.	6/08/07
S-MN-A-013-Rev.05	Complete rearrangement of sections. Deleted Section 7 Responsibilities and Distribution based on SOP of SOP Preparation Moved Instrument condition to Section 8, move all standard preparation to Section 9, moved all calibration and tuning information to Section 10, moved all QC acceptance and corrective action to Section 12. Added corrective actions throughout Added attachments VIII-X for Tedlar bags Added corrective actions for internal standards and surrogates (12.5 and 12.6).	21April2008
S-MN-A-013-Rev.06	Changed the holding time for Tedlar bag from 72 to 48 hrs for Ohio VAP, except for client specific QAPPS Added that BFB can be combined with CCV analysis CCV criteria for TO-14 is 10% Section 1 edited to include soil vapor samples Clarification about differences in Ohio VAP samples vs. other clients Section 12.5; Ohio VAP samples must be reanalyzed undiluted	02Jun2009
S-MN-A-013-Rev.07	Added Benzyl Chloride to the target compound list. Changed location of MSDS to Groupwise Added reference to Method TO-14 Formatting updates Changed heading of table in 8.2.5.3 to “Sample Transfer” Remove “LCS” from 9.2.2 LCS info updated in 11.3.1 “LCS” removed from Attachment VI	03Dec2009
S-MN-A-013 Rev.08	Updated Methyl Ethyl Ketone – primary ion: 72 secondary ion: 43	31Mar2010

ATTACHMENT I - Target Compound List

Compound	CAS RN	TO14 compounds
1,1,1-Trichloroethane	71-55-6	X
1,1,2,2-Tetrachloroethane	79-34-5	X
1,12-trichloroethane	79-00-5	
1,1-Dichloroethane	75-34-3	X
1,1-Dichloroethene	75-35-4	X
1,2,4-Trichlorobenzene	95-63-6	X
1,2,4-Trimethylbenzene	95-63-6	X
1,2-Dibromoethane	106-93-4	X
1,2-Dichlorobenzene	95-50-1	X
1,2-Dichloroethane	107-06-2	X
1,2-Dichloropropane	78-87-5	X
1,3,5-Trimethylbenzene	108-67-8	X
1,3-Butadiene	106-99-0	
1,3-Dichlorobenzene	541-73-1	X
1,4-Dichlorobenzene	106-46-7	X
4-Ethyltoluene	622-96-8	
Acetone	67-64-1	
Acrolein	107-02-8	
Acrylonitrile	107-13-1	
Benzene	71-43-2	X
Benzyl Chloride	100-44-7	
Bromodichloromethane	75-27-4	
Bromoform	75-25-2	
Bromomethane	74-83-9	X
Carbon Disulfide	75-15-0	
Carbon Tetrachloride	56-23-5	X
Chlorobenzene	108-90-7	X
Chloroethane	75-00-3	X
Chloroform	67-66-3	X
Chloromethane	74-87-3	X
Cis-1,2-Dichloroethene	156-59-2	X
Cis-1,3-Dichloropropene	10061-01-5	X

ATTACHMENT I (continued)

Compound	CAS RN	TO14 compounds
Cyclohexane	110-82-7	
Dibromochloromethane	124-48-1	
Dichlorodifluoromethane	75-71-8	X
Dichlorotetrafluoroethane	76-14-2	X
Ethanol	64-17-5	
Ethyl Acetate	141-78-6	
Ethyl Benzene	100-41-4	X
Freon 113	76-13-1	X
Heptane	142-82-5	
Hexachlorobutadiene	87-68-3	X
Hexane	110-54-3	
Isopropyl Alcohol	67-63-0	
M,P Xylene	106-42-3	X
O-Xylene	95-47-6	X
Methyl Butyl Ketone	591-78-6	
Methyl Ethyl Ketone	78-93-3	
Methyl Isobutyl Ketone	108-10-1	
Methyl Tert Butyl Ether	1634-04-4	
Methylene Chloride	75-0902	X
Napthalene	91-20-3	
Propylene	115-07-1	
Styrene	100-42-5	X
Tetrachloroethene	127-18-4	X
Tetrahydrofuran	109-99-9	
Toluene	108-88-3	X
Trans-1,2-Dichloroethene	156-60-5	
Trans-1,3-Dichloropropene	10061-02-6	X
Trichloroethene	79-01-6	X
Trichlorofluoromethane	75-69-4	X
Vinyl Acetate	108-05-4	
Vinyl Chloride	75-01-4	X

*Current reporting limits can be found in Horizon

ATTACHMENT II - Required BFB Key Ions And Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	8.0 - 40.0 percent of mass 95
75	30.0 - 66.0 percent of mass 95
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of mass 95 (See note)
173	less than 2.0 percent of mass 174
174	50.0 - 120.0 percent of mass 95
175	4.0 - 9.0 percent of mass 174
176	93.0 - 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

ATTACHMENT III - Characteristic Ions For Target Compounds

Compound	Primary Ion	Secondary Ion(s)	Internal Standard Group
Propylene	41	39	1
Dichlorodifluoromethane	85	87	1
Chloromethane	50	52	1
Dichlorotetrafluoroethane	85	135,87	1
Vinyl Chloride	62	64	1
1,3-Butadiene	54	39	1
Bromomethane	94	96	1
Chloroethane	64	66	1
Ethanol	31	45	1
Trichlorofluoromethane	101	103,105	1
Acetone	43	58	1
Isopropyl Alcohol	45	43	1
1,1-Dichloroethene	61	96	1
Freon 113	101	103,151	1
Methylene Chloride	49	84,86	1
Carbon Disulfide	76	44,78	1
Trans-1,2-Dichloroethene	96	61,98	1
Methyl Tert Butyl Ether	73	41	1
Vinyl Acetate	43	86	1
1,1-Dichloroethane	63	65	1
Methyl Ethyl Ketone	72	43	1
Hexane	57	41,43	1
Cis-1,2-Dichloroethene	96	61,98	1
Ethyl Acetate	43	61,70	1
Chloroform	83	85,47	1
Tetrahydrofuran	42	41,72	1
1,1,1-Trichloroethane	97	99,61	1
1,2-Dichloroethane	62	64	1
Benzene	78	77,50	1
Carbon Tetrachloride	117	119	1
Cyclohexane	56	84,41	1
Heptane	43	41	1
1,2-Dichloropropane	63	41,62	1
Trichloroethene	130	132,95	1

ATTACHMENT III (continued)

Compound	Primary Ion	Secondary Ion(s)	Internal Standard Group
Bromodichloromethane	83	85	1
Napthalene	128	127	1
Methyl Isobutyl Ketone	43	58,100	1
Cis-1,3-Dichloropropene	75	39,77	1
Trans-1,3-Dichloropropene	75	39,77	1
Toluene	91	92	1
1,12-trichloroethane	97	83,61	1
Methyl Butyl Ketone	43	58	2
Dibromochloromethane	129	127	2
1,2-Dibromoethane	107	109	2
Tetrachloroethene	166	164,131	2
Chlorobenzene	112	77,114	2
Ethyl Benzene	91	106	2
M,P,& O Xylene	91	106	2
Bromoform	173	171	2
Styrene	104	78,103	2
1,1,2,2-Tetrachloroethane	83	85	2
4-Ethyltoluene	105	120,79	2
1,3,5-Trimethylbenzene	105	120	2
1,2,4-Trimethylbenzene	105	120	2
1,3-Dichlorobenzene	146	111,148	2
Benzyl Chloride	91	126	2
1,4-Dichlorobenzene	146	148,111	2
1,2-Dichlorobenzene	146	111,148	2
1,2,4-Trichlorobenzene	180	182,184	2
Hexachlorobutadiene	225	227,223	2
1,4-Difluorobenzene	114	88	IS #1
Chlorobenzene	117	82	IS #2
Hexane-d14 (surr)	66	64	1
Toluene-d8 (surr)	98	100	1
1,4-Dichlorobenzene-d4 (surr)	150	152	2

ATTACHMENT IV - Calibration of THC as Gas

IV-1 THC as gas is calibrated by using the same calibration runs that are used for all other compounds, as well as using the same acceptance criteria.

IV-2 The original calibration files are copied to a target batch. This does not change the raw data in any way, it merely allows the same data to be processed against two different methods

IV-3 The area response is obtained by summing the area in the total ion chromatogram from the first eluting compound of interest till the end of the run. The internal standard is included as part of this value, the response factor is not calculated using the internal standard method. It is solely based on area response and calibration concentration

IV-4 The calibration concentration at each level is obtained by summing the values of the individual compounds present in the calibration standard.

IV-5 A response factor is obtained as detailed earlier in this SOP. Calibration criteria are the same as stated earlier in this SOP.

IV-6 Custom THC values may be obtained and are noted as such on final reports. These custom values can be based on calibrating using a select list of compounds or a select time frame for example. Requests for these custom values will be evaluated on an individual basis for analytical feasibility.

ATTACHMENT V - Canister Dilution Factors (6L)

6 Liter Canister					
Units (inches Hg or PSIG)	**Initial Pressure**	**Initial Pressure Converted to PSIA**	**Final Pressure (PSIG)**	**Pressure Converted to PSIA**	**Dilution Factor**
Hg	0	14.69	5	19.69	1.34
Hg	-1	14.22	5	19.69	1.38
Hg	-2	13.75	5	19.69	1.43
Hg	-3	13.28	5	19.69	1.48
Hg	-4	12.81	5	19.69	1.54
Hg	-5	12.35	5	19.69	1.59
Hg	-6	11.88	5	19.69	1.66
Hg	-7	11.41	5	19.69	1.73
Hg	-8	10.94	5	19.69	1.80
Hg	-9	10.47	5	19.69	1.88
Hg	-10	10	5	19.69	1.97
Hg	-11	9.53	5	19.69	2.07
Hg	-12	9.06	5	19.69	2.17
Hg	-13	8.59	5	19.69	2.29
Hg	-14	8.12	5	19.69	2.42
Hg	-15	7.66	5	19.69	2.57
Hg	-16	7.19	5	19.69	2.74
Hg	-17	6.72	5	19.69	2.93
Hg	-18	6.25	5	19.69	3.15
Hg	-19	5.78	5	19.69	3.41
Hg	-20	5.31	5	19.69	3.71
Hg	-21	4.84	5	19.69	4.07
Hg	-22	4.37	5	19.69	4.5
Hg	-23	3.9	5	19.69	5.04
Hg	-24	3.43	5	19.69	5.73
Hg	-25	2.97	5	19.69	6.64
Hg	-26	2.5	5	19.69	7.89
Hg	-27	2.03	5	19.69	9.71
Hg	-28	1.56	5	19.69	12.64
Hg	-29	1.09	5	19.69	18.08
PSIG	1	15.69	5	19.69	1.25
PSIG	2	16.69	5	19.69	1.18

ATTACHMENT V (continued) - Canister Dilution Factors (1L)

1 Liter Canister

Initial Pressure Units (inches Hg or PSIG)	Initial Pressure	Initial Pressure Converted to PSIA	Final Pressure (PSIG)	Final Pressure Converted to PSIA	Dilution Factor
Hg	0	14.69	10	24.69	1.68
Hg	-1	14.22	10	24.69	1.74
Hg	-2	13.75	10	24.69	1.80
Hg	-3	13.28	10	24.69	1.86
Hg	-4	12.81	10	24.69	1.93
Hg	-5	12.35	10	24.69	2.00
Hg	-6	11.88	10	24.69	2.08
Hg	-7	11.41	10	24.69	2.16
Hg	-8	10.94	10	24.69	2.26
Hg	-9	10.47	10	24.69	2.36
Hg	-10	10	10	24.69	2.47
Hg	-11	9.53	10	24.69	2.59
Hg	-12	9.06	10	24.69	2.73
Hg	-13	8.59	10	24.69	2.87
Hg	-14	8.12	10	24.69	3.04
Hg	-15	7.66	10	24.69	3.22
Hg	-16	7.19	10	24.69	3.43
Hg	-17	6.72	10	24.69	3.67
Hg	-18	6.25	10	24.69	3.95
Hg	-19	5.78	10	24.69	4.27
Hg	-20	5.31	10	24.69	4.65
Hg	-21	4.84	10	24.69	5.10
Hg	-22	4.37	10	24.69	5.65
Hg	-23	3.9	10	24.69	6.33
Hg	-24	3.43	10	24.69	7.20
Hg	-25	2.97	10	24.69	8.31
Hg	-26	2.5	10	24.69	9.88
Hg	-27	2.03	10	24.69	12.2
Hg	-28	1.56	10	24.69	15.8
Hg	-29	1.09	10	24.69	22.7
PSIG	1	15.69	10	24.69	1.57
PSIG	2	16.69	10	24.69	1.48

ATTACHMENT VI - Air Laboratory Standard Preparation Procedures

CALIBRATION STANDARD

The calibration standard is purchased in the form of a pressurized cylinder from SPECTRA GASES, INC. This is a custom mix that includes all compounds of interest at 1ppmv.

2 PPBV:

Using the 1000cc gas tight syringe, pull 90cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 2 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

20 PPBV:

Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

SECOND SOURCE VERIFICATION

The laboratory control standard is purchased in the form of a pressurized cylinder from a source independent of the calibration mix (Custom Gas Solutions, or equivalent). This is a custom mix that includes all compounds of interest at 1ppmv.

20 PPBV:

Using the 1000cc gas tight syringe, pull 900cc from TO15 Stock Standard Cylinder into a clean, evacuated 15L canister, that has been humidified with 50ul H2O. Pressurize the canister to 30 psig with clean nitrogen. This yields a final concentration of 20 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

Internal Standard/ Surrogate/ BFB Standard 100ppbv:

The internal, surrogate, and bfb standards are purchased as neat standards from specific vendors; such as Chem-Service, Sigma-Aldrich or equivalent.

To prepare a neat/cocktail standard: Using Entech Static Dilution software, enter barometric pressure, room temperature, flask temperature, flask volume, canister pressure, canister volume, flask concentration and desired final concentration (100ppbv). The software calculates approximate transfer volume 1 (vial to flask), then transfer volume 2 (flask to canister)*. Note: Standard canister (6 L or 15 L) must be cleaned, evacuated, and humidified with 50ul H2O before being used.

Example:
Barometric Pressure: 29.92
Room Temperature : 24 degree C
Flask Temperature : 65 degree C
Flask Volume : 2000 ml
Canister Pressure : 30 psig
Canister Volume : 15,000 ml (15 L)
Flask Concentration : 520.015 PPM

*The software calculates transfer volume to th $1/10000^{th}$. The volumetric syringes are calibrated to $1/10^{th}$ of a decimal place. Therefore, the analyst will adjust the volume to a measurable amount prior to standard preparation.

Then, pressurize the 15 L canister 30 psig with clean nitrogen. This yields a final concentration of 100 ppbv. Record the standard ID, date created, analyst initial, canister number, canister volume, stock standard ID, volume used, water volume added, final pressure (psig), final concentration, and expiration date in the Pace Standard Preparation Logbook.

ATTACHMENT VII - Procedures For Analyzing MPCA Samples

VII-1 Samples must be carefully monitored for carryover from previous samples with large detections. Analysts and data reviewers need to verify that each analysis has been evaluated for potential carryover.

- If a compound of interest has an on-column concentration that is greater than 10% of the previous sample, it will be assumed that this value is not due to carryover.
- If the compound of interest has an on-column concentration between 2 and 10% of the previous sample, then the analyst will carefully examine other factors relating to sample analysis (i.e. the concentration of related components, the overall concentration of constituents in each sample, etc.). When in doubt, the analyst must re-analyze the sample to confirm that the results are not due to carryover.
- When the compound of interest has an on-column concentration which is less than 2% of the previous sample's concentration, but greater than the method reporting limit, the sample must be analyzed to confirm or eliminate possible carryover.

VII-2 Sample duplicate analysis must be performed at a minimum of 1 in 10 samples analyzed.

VII-3 The relative detection limit for MPCA samples is 0.200 ppbv for all analytes except m&p xylene which has a relative detection limit of 0.400 ppbv.

ATTACHMENT VIII - Procedure for Tedlar Bags

Transfer of Tedlar Bags to SUMMA Canisters

In the event that a sample is collected into a tedlar bag, the client has 48 hours to get the bag to the facility for analytical testing. Pace Analytical Services recognizes a 48 hour holding time for all samples collected in tedlar bags. Upon receipt at the laboratory, the tedlar bag will be transferred into a batch certified, evacuated one liter SUMMA canister for analysis. The sample will subsequently be analyzed by the appropriate method within 28 days of transfer.

Procedure for transfer:

- Tedlar bag is received and logged for analysis by Pace Analytical Services
- The sample is delivered to the Air Lab, and the laboratory numbers assigned to the sample is recorded in a logbook (as delivered; see Attachment IX).
- The bag is connected to a clean, evacuated canister (105mTorr).
    - The tip of the bag valve is placed into tubing, connected by a ¼" nut to the sample valve of the canister, secured with a wrench to insure all sample is pulled into the can.
- The bag is opened first. Second, the can is opened.
    - By opening the canister second, the sample will be transferred into the can through vacuum (since the can is evacuated to 150mTorr, and the bag is at ambient room pressure).
- After the sample is transferred the sample data and canister number, time and date, is recorded into the transfer logbook (Attachment X).
- Sample is submitted to the laboratory for analysis.
- A data qualifier is added to the report, notifying the client of the transfer.

ATTACHMENT IX - Tedlar Sign-off Logbook (example)

Pace Analytical

Tedlar Signoff Logbook

Date	Time	Project Number	Sample Number(s)	Method	Initials

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ATTACHMENT X - Tedlar Bag Transfer Log (example)

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Tedlar Bag Transfer Log

Sample ID	Can ID	Date and Time Collected	Date and Time when Tedlar Bag was evacuated to the can	Comments

O107a(28Sep2006)

ATTACHMENT XI – Common Logbook Abbreviations

RR	**Reanalysis for previously analyzed sample**
DIL	**Dilution for over-range compounds from a previously reported sample**
CONF	**Confirms results from a previously analyzed sample**
C/O	**Possible carryover from a prior sample**
OK	**Analysis is acceptable and sample is reported**

VOLATILE ORGANIC COMPOUNDS BY DIRECT INJECT (DI)-GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) USING SELECTIVE ION MONITORING (SIM)

1 SCOPE AND APPLICATION

1.1 This method is a capillary gas chromatography/mass spectrometry (GC/MS) method used to determine part per billion concentrations of volatile organic compounds (VOC). The volatiles are introduced into the GC/MS system by the direct injection technique. This method is used to determine volatile organic compounds in soil, sediment and waste samples.

Compound	CAS No.
Benzene	71-43-2
Toluene	108-88-3
Ethylbenzene	100-41-4
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3
1,3,5-Trimethylbenzene	108-67-8
1,2,4-Trimethylbenzene	95-63-6

1.2 Instrumentation: Hewlett Packard (HP) 5972 Mass Selective Detector (MS) or equivalent capable of acquiring data in Selective Ion Monitoring (SIM) mode, HP 5890 Gas Chromatograph, Leap AS-200 auto-sampler, Agilent MS Productivity software with Enviroquant® data analysis menu.

1.3 This method is used to identify and quantitate volatile organic compounds that can be efficiently extracted with methanol.

1.4 The GC/MS provides for selective detection of the target analytes by measuring specific characteristic ions for each analyte and qualification from the selected characteristic ion using the Internal Standard method of quantitation. Compound identification is performed by comparing the unknown compounds retention time and qualifier ion ratios against reference qualifier ion ratios and retention time acquired from authentic standards.

1.5 Table 1 provides statistically determined method detection limits (MDL) for target compounds in silica sand. In general, soils are reported to the level of the lowest calibration standard. Some compounds have higher reporting limits due to instrument conditions or performance.

1.6 Typical Initial Demonstration of Capability (IDC) data for silica sand are reported in Table 2.

1.7 The Lead Chemist should be a chromatography expert capable of operating and

maintaining the GC/MS system and experienced in the analysis of VOCs. The analyst should have completed training and demonstrated capability prior to analyzing samples. Training includes generating a valid initial calibration and an acceptable IDC study in addition to understanding the use of mass spectra for identification and quantitation.

2 SUMMARY OF METHOD

2.1 Soil samples are extracted with methanol in the VOC vial.

2.2 An aliquot of the extract is injected directly onto the chromatographic column and target analytes are measured by GC/MS-SIM.

2.3 Identification of target analytes is accomplished by first comparing the retention time of measured components to the retention time of authentic standards. The measured ions and their relative intensities of components which elute within the retention time window for an analyte are then compared to the ions and ratios of the authentic standard. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using linear regression with a weighting factor of 1 over concentration.

2.4 A limitation of using GC/MS-SIM is the inability to analyze for non-target analytes because only certain mass fragments of target analytes are being detected by the MS.

3 INTERFERENCES

3.1 Major contaminant sources are materials and solvents that enter the atmosphere or diffuse into the methanol which have interfering ions or are present at large enough concentrations to induce a response on the electron multiplier generating a false response for the ion being measured. Special care must be used with solvents to avoid methylene chloride and acetone contamination. Methanol is stored apart from other solvents in the VOA annex.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the seal of the sample container into the sample during shipment and storage. Matrix interferences are minimized through SIM analysis by only scanning for mass fragments of target analytes compared to full scan mode.

3.3 High levels of petroleum hydrocarbons, such as those found in diesel fuel, can potentially generate interferences for compounds being analyzed. Peaks that elute within a retention time window for an analyte but fail ion ratio criteria must be closely evaluated for possible interferences. Analysis using SIM requires the analyst to place emphasis on the ion ratio criteria to eliminate false positives. Since less information is collected in the SIM mode than full scan, additional analysis may be required to confirm the presence of an analyte.

4 APPARATUS AND MATERIALS

4.1 Gas Chromatograph / Mass Spectrometer System

Gas Chromatograph:	HP 5890 with Split/Splitless Injector
Detector:	HP 5972 Mass Selective Detector or equivalent
Autosampler:	LEAP Technologies A200S
Column:	RTX-1, 30 m x 0.32 mm I.D., 1.5 µ film or equivalent
Data System:	Agilent MSD Productivity Chemstation with Enviroquant

4.2 GC/MS Supplies List

Seals:	Dual Vespel Ring Inlet Seal, Restek Cat.#21239
Septa:	LB-2 Septa, 11 mm, Supelco #164742
Ferrules:	1/16” x 0.5 mm Vespel/Graphite, Restek Cat# 20231/20249
Liners:	Gooseneck Splitless Liner 4 mm x 6.5 mm x 78.5 mm, Restek Cat.#20799

4.3 Vortex Mixer

4.4 Ultrasonic Bath

4.5 Centrifuge: Capable of Holding 40 mL VOA vials

4.6 Balances

4.6.1 Top loader capable of weighting to 0.01 gram

4.6.2 Analytical capable of weighting to 0.0001 gram

4.7 Micro-syringes: Gas-Tight®, 10, 25, 100, 250, 500, and 1,000 µL

4.8 Vials

4.8.1 40 mL VOA vials

4.8.2 GC Vials: 2 mL amber autosampler vials Catalog. #40811A-120 and PTFE/Silicon aluminum seals, Part #5150-11, LSDC

4.8.3 Scintillation Vials: 20 mL

4.9 “Lock and Load” Sampling System

4.9.1 Plastic syringes for collection of a nominal 10 gram soil sample, Environmental Sampling Supply Part # LL5035SRNG

4.9.2 Plastic re-usable handle, Environmental Sampling Supply Part # LL5035HNDL

4.10 5 oz. specimen container with lid for collection of soils intended for percent solids analysis, VWR Part # 15704-020 and 15704-019.

4.11 Disposable single use glass purge tubes, 19 x 150 mm glass culture tubes, VWR Part # 45060-19150

4.12 Glass disposable transfer pipettes – 5 ¾"

4.13 EMD Optifix® solvent dispenser capable of 10 mL volume

4.14 Stainless Steel spatulas

4.15 Eppendorf Air displacement pipette with 1 mL disposable tips

4.16 Drying Oven capable of maintaining 105 °C

4.17 Refrigerator capable of maintaining 4 °C

4.18 Freezer capable of maintaining temperatures below -15 °C

4.19 Plastic 250 to 500 mL squirt bottles

5 REAGENTS

5.1 Solvents

5.1.1 Methanol: Purge & Trap Grade, stored in the Annex apart from other solvents

5.1.2 VOC-free water purchased commercially. Roundy's brand distilled water is used.

5.1.3 Prepare 1 L of a 25% water/75% methanol solution to be used for working standard preparation. Slowly add 250 mL of water to approximately 700mL of methanol in a 1000mL volumetric flask. Care must be exercised because the solution will become warm. Once the solution cools, bring the flask to volume with methanol.

5.2 Solids

5.2.1 Silica Sand: Baked in a muffle furnace at 400 °C for a minimum of 4 hours

5.3 Acids/Bases – not applicable for this method

5.4 Stock Calibration Standards

5.4.1 Stock solutions are purchased from vendors as certified concentrations in methanol. The vendor is Absolute Standards. The BTEX and trimethylbenzenes (Absolute Part # 90379) are purchased at 1000 µg/mL.

5.4.2 Table 4 provides a listing of components in each stock solution. Stocks are used only once when the ampoule is opened and then diluted to prepare the working solution.

5.5 Intermediate Standard Solutions:

5.5.1 10,000 ng/mL BTEX and Trimethylbenzene Standard

5.5.1.1 The intermediate standard solution (10,000 ng/mL) is prepared by diluting the stock standard solutions (See Section 5.4.1) and surrogate (See Section 5.7.1.1) in a 50 mL volumetric flask with methanol.

5.5.1.2 Fill a 50 mL volumetric flask ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that a syringe needle tip can be submerged in the methanol.

5.5.1.3 Aliquots of the stock are added below the level of methanol in the flask from the syringe needle.

NOTE: Table 5 summarizes the preparation of these standards.

5.5.1.4 Once all components are added to the 50 mL volumetric, make to volume with purge and trap grade methanol.

5.5.1.5 The standard is stored in 40 mL VOA vials. Store frozen and assign a six-month expiration date.

5.6 Calibration Standards:

5.6.1 The working standards are prepared using a 25% water/ 75% methanol solution at levels of 5, 10, 25, 100, 200, 500, and 2,000 ng/mL for all targets and surrogates.

5.6.2 ICAL Level 1 (5 ng/mL) Preparation

5.6.2.1 Fill a 50 mL volumetric flask ¾ full of the 25% water/75% methanol solution known to be free of volatiles. Make sure the volumetric has enough solution added so that a syringe needle tip can be submerged.

5.6.2.2 Using a Gas-Tight® syringe, add 25 µL of the Intermediate Standard Solution (See Section 5.5.1) to a 50 mL volumetric flask by inserting the syringe below the surface of the water/methanol in the flask.

5.6.2.3 Make to volume with the water/methanol solution.

5.6.2.4 Transfer to LIMS labeled amber VOA vials, store refrigerated and assign a one month expiration date.

5.6.3 Refer to Table 6 for the preparation of the remaining 6 ICAL levels.

5.7 Surrogate

5.7.1 Stock Surrogate Solution

5.7.1.1 The surrogate standard, toluene-d_8 is purchased from Absolute standards at 1000 µg/mL (Part # 70282). See Table 4.

5.7.2 Working Surrogate Standard (Toluene-d_8 at 20,000 ng/mL)

5.7.2.1 Fill a 25 mL volumetric flask ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that a syringe needle tip can be submerged in the methanol.

5.7.2.2 Using a Gas-Tight® syringe, aliquot 500 µL of the stock surrogate (See Section 5.7.1.1) in a 25 mL volumetric flask by inserting the syringe below the surface of the methanol in the flask.

5.7.2.3 Make to volume with methanol.

5.7.2.4 Once prepared, the working surrogate solution is then portioned into 2 mL GC vials for daily use. Each vial is filled to its neck and capped.

5.7.2.5 Assign an expiration date of three months; enter information into LIMS system and store refrigerated.

5.8 Laboratory Control Sample (LCS) Spike Mix

5.8.1 The LCS spike mix is also the Intermediate Standard Mix detailed in Section 5.5.1.

5.9 MS/MSD Spike Mix

5.9.1 The MS/MSD spike mix is also the Intermediate Standard Mix detailed in Section 5.5.1.

5.10 Second Source

5.10.1 Stock Second Source Standard Solution (1,000 µg/mL)

5.10.1.1 The second source stock standard solutions are purchased from vendors as certified concentrations in methanol. The vendor is Restek. The BTEX, trimethylbenzene standard (Restek Part # 30095) is purchased at 1,000 µg/mL. Table 4 provides a listing of components in each stock solution.

5.10.2 Intermediate Second Source Standard Solution (10,000 ng/mL)

5.10.2.1 The intermediate standard solution (10,000 ng/mL) is prepared by diluting the stock standard solutions (See Section 5.10.1.1) and surrogate (See Section 5.7.1.1) in a 50 mL volumetric flask with methanol.

5.10.2.2 Fill a 50 mL volumetric flask ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that a syringe needle tip can be submerged in the methanol.

5.10.2.3 Aliquots of the stock are added below the level of methanol in the flask from the syringe needle.

NOTE: Table 5 summarizes the preparation of these standards.

5.10.2.4 Once all components are added to the 50 mL volumetric, make to volume with purge and trap grade methanol.

5.10.2.5 The standard is stored in 40 mL VOA vials. Store frozen and assign a six-month expiration date.

5.10.3 Working Second Source Standard (200 ng/mL)

5.10.3.1 Fill a 50 mL volumetric flask ¾ full of water/methanol solution (See Section 5.1.3) known to be free of volatiles. Make sure the volumetric has enough water/methanol solution so that a syringe needle tip can be submerged.

5.10.3.2 Using a Gas-Tight® syringe, aliquot 1000 µL of the Intermediate Standard Solution (See Section 5.10.2) into a 50 mL volumetric flask by dispensing the standard below the surface of the water/methanol solution in the flask.

5.10.3.3 Make to volume with methanol.

5.10.3.4 Transfer to LIMS labeled 40 mL amber VOA vials, store refrigerated and assign a one month expiration date.

5.11 Internal Standards (1,4-difluorobenzene, chlorobenzene-d_5 and 1,4-dichlorobenze-d_4)

5.11.1 Stock Internal Standard Solutions

5.11.1.1 Stock solutions are purchased as certified solutions in methanol from Restek. The internal standards (Part#30074) are purchased at 2,500 µg/mL. Table 4 provides a listing of the components in each stock solution.

5.11.2 Working Internal Standard Solution

5.11.2.1 The working internal standard solution is prepared at 10,000 ng/mL by diluting the stock standard solution (See Section 5.11.1) in a 50 mL

volumetric flask. Table 5 summarizes preparation of this standard.

5.11.2.2 Fill a 50 mL volumetric ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that the syringe needle tip can be submerged below the level of the methanol.

5.11.2.3 Using a 250 µL Gastight syringe aliquot 200 µL of internal standard mix (See Section 5.11.1) and add to the 50 mL volumetric by inserting the syringe below the surface of the methanol.

5.11.2.4 Make to volume with purge and trap methanol.

5.11.2.5 Once prepared, the working internal standard solution is then portioned into 2 mL GC vials for daily use. Each vial is filled to its neck and capped.

5.11.2.6 Assign an expiration date of three months, enter information into LIMS system and store frozen.

6 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sample Containers, Custody and Preservation: Containers are provided by the laboratory to the client prior to sampling whenever possible. Samples are collected by the client and delivered to the laboratory with a chain of custody (COC). Upon receipt of samples, the sample custodian notes any deviations on the chain of custody.

NOTE: If a COC is not received with the samples, ECCS personnel will fill out a COC per GEN-003 Sample Receipt and Login SOP.

6.2 Soil Samples

6.2.1 ECCS typically supplies clients with a pre-weighed 40 mL VOA vial containing 10 mL of methanol for soil collection. In addition, ECCS supplies a Lock and Load T handle (See Section 4.9) and disposable sample container for collection of a pre-measured 10 g (approximately) of soil.

NOTE: Need to encourage clients to collect MS/MSD samples for VOAs. Collect 2 additional methanol vials for 1 sample in every 20.

NOTE: Methanol used needs to be purge and trap grade methanol.

6.2.2 The client is responsible to put about 10 g of soil into the VOA vial containing purge and trap methanol either by use of a Lock and Load or using a balance in the field.

6.2.3 A common alternative approach is used by some clients. The client will weigh 25 grams of sample into a 2-oz jar containing 25 mL of methanol.

6.2.4 All samples collected in the field need to be placed in a cooler on ice for transport

to a lab.

6.2.5 Soil samples received at an ECCS lab are stored in a refrigerator at 4 °C.

NOTE: Once received, soil and water samples are stored in a refrigerator in the Annex if at the fixed-base laboratory.

6.2.6 Soil samples received for percent solids analysis are stored in a separate refrigerator in the fixed base lab or on the bench in field labs.

NOTE: Field lab moisture analysis is usually started within 2 to 4 hours of receipt. Therefore, storage in a refrigerator or on ice is not necessary.

6.3 Methanol preserved soil samples have a hold time of 14 days from time of collection.

6.4 Soil samples received without preservation in methanol are extracted as soon as possible by weighing 10 g of soil into a 40 mL VOA vial containing 10 mL of methanol.

NOTE: Make sure to note on the COC that the sample was received without preservation.

6.5 Safety

6.5.1 Recommend use of latex/rubber/nitrile gloves during collection of any and all samples

6.5.2 Recommend use of safety glasses in case methanol splashes out of VOA vial when transferring sample from a “lock and load” sampling device into the VOA vial.

6.5.3 Methanol is a flammable solvent. Smoking is not permitted in the vicinity of methanol, nor should sample collection vials be stored near an open flame. An additional hazard exists because when methanol burns, the flame is almost colorless.

7 PROCEDURE

7.1 Water extraction is not applicable to this method.

7.2 Soil Blanks/LCS/MS Preparation

7.2.1 Blank: The blank for soil is prepared by weighing 10 grams of silica sand (See Section 5.2) into a VOA vial spiking with 100 µL of the working surrogate solution then adding 10 mL of purge and trap methanol. The soil blank is sonicated and processed along with actual samples. A 0.5 mL aliquot of the methanol extract from the soil blank is transferred to a 2.0 mL auto-sampler vial and 20 µL of the working internal standard added prior GC/MS analysis.

NOTE: Blanks once prepared are sonicated along with unknown samples and stored with unknown samples.

7.2.2 LCS and MS/MSD Samples: The laboratory control sample is prepared from silica sand and methanol in a VOA vial similar to the soil blank except the target compounds are also spiked onto the sand by adding 200 µL of the 10,000 ng/mL Intermediate Stock standard prior to adding 9.8 mL of methanol to the vial. Add 200 µL of the Intermediate Standard Solution (See Section 5.5.1) to the 9.8 mL of methanol and10 grams of sand in the vial. The LCS soil is then sonicated and processed along with actual samples. Transfer 0.5 mL of the methanol extract from the soil LCS to a 2.0 mL auto-sampler vial and spike with 20 µL of the working internal standard solution prior to GC/MS analysis. The Matrix Spike/Matrix Spike duplicate samples are prepared using the same spike solution and volumes as the LCS. The MS and MSD, prepared and analyzed separately, are prepared using two separate sample containers containing sample from the same sampling point.

7.2.3 Soil Weight Determination

7.2.3.1 Client supplied soils in non-ECCS containers, preserved with methanol: Use the weight of soil and volume of methanol supplied with the sample by the client.

7.2.3.2 Client supplied soil in a jar: Accurately weigh 10 g of soil into a 40 mL VOC vial, record weight and immediately add 10 mL of methanol.

7.2.3.3 Encore sampler: Dispense the contents of the sampler into a tared jar containing 10.0 ml of methanol. Determine the sample weight by difference and record the weight.

7.2.3.4 VOA vials supplied by ECCS with the Lock and Load System (Preferred Method)

7.2.3.4.1 Each VOA vial contains 10.0 mL of methanol. The entire container including the vial, label, and methanol was pre-weighed to 0.XX g prior to shipment to the client. The total weight is recorded on the label with permanent ink.

7.2.3.4.2 Upon receipt at ECCS the vial will be reweighted to 0.XX g. The weight will be recorded on the COC and the information entered into the LIMS system either by sample entry or by the chemist prior to analysis.

7.2.3.4.3 The soil weight obtained is the wet weight to be used for sample analysis.

7.2.4 Extraction by Sonication:

7.2.4.1 Add 100 µL of the surrogate standard to the samples and blank.

NOTE: The LCS/MS/MSD do not need surrogate spiked separately as the intermediate spike mix (See Section 5.1.1) already contains surrogate.

7.2.4.2 Vortex the soil sample contained in the VOA vial for about 2 minutes or until the soil breaks up.

7.2.4.3 Place enough water in the ultrasonic bath to be level with the top of the methanol in the soil vial. The water in the bath needs to be clean, free of dust and debris, in order to effectively transfer energy in the bath.

7.2.4.4 Sonicate the sample for 20 minutes.

NOTE: Water bath needs to be kept cool either by using ice or changing the water.

NOTE: During sonication if clumps of soil do not break up, cool the sample and using mechanical means break up the clumps (i.e. vortex, use stainless steel spatula).

7.2.4.5 After sonication, vortex the samples again for approximately 30 seconds.

7.2.5 Aliquoting Extracts and Archiving:

Allow the soil to settle from the upper layer of clear methanol extract - usually ½ hour. Alternatively, the vial may be centrifuged at a low speed (<1500 rpm) to separate the soils from the extract.

NOTE: Care must be taken while centrifuging not to break the spinning vial from too high of a speed.

7.3 Concentration and Transfer: Not applicable to this method.

7.4 Clean-Up and/or Derivatization: Not applicable to this method.

7.5 Instrument Conditions

7.5.1 GC/MS Pump Down: Allow the GC/MS system to stabilize under high vacuum when starting from a vented state. Before analysis, allow at least 4 hours for the MS to stabilize after pump-down.

7.5.2 GC Conditions:

Column:	RTX-1, 30 m x0.32 mm, 1.5 µm film or DB-624, 30 m x 0.32 mm, 1.8 µm

film

Injector Temp:	200 °C
Column Head Pressure:	5 PSI
Column Flow:	2 mL/min
Purge Valve Flow:	1 mL/min
Split Flow:	14 mL/min
Split Ratio:	7:1
Oven Temperature Program	
Initial Temperature:	50 °C
Initial Hold:	2 minutes
Initial Ramp:	12 °C/ minute
Final Temperature:	12 °C/ minute
2nd Ramp	22 °C/ minute
Final Temperature:	320 °C
Final Hold:	1.0 minutes

7.5.2.1 MS Conditions

Tune:	Maximum Sensitivity Autotune
EM Voltage:	300 above autotune
Mode:	Selective Ion Monitoring

7.5.2.2 SIM Scan Parameters

Group	Start Time	Ion	Dwell Time (msec)
1	2.4 min	78	50
		77	50
		91	50
		92	50
		106	50
		98	50
		100	50
		114	50
		63	50
		117	50
		82	50
2	8.5 min	105	50
		120	50
		94	50
		66	50
		108	50
		107	50
		122	50

99	50
71	50
150	50
115	50

NOTE: It may be necessary to adjust Group times as columns are maintained.

7.6 Preventive Maintenance

7.6.1 The tune report for the MS serves as a useful diagnostic tool. It can be used to indicate when maintenance on the MS is required.

7.6.1.1 Detection of Leaks in the GC/MS System: The presence of a base peak at m/z 28 (N_2) that is higher than m/z 69 (base peak of PFTBA) in the tune report indicates the presence of a gross leak in the GC/MS vacuum system. A common source of the leak is a loose transfer line nut sealing the GC capillary column to the MS transfer line. Tightening this nut often eliminates a leak.

7.6.1.2 Water in the GC/MS System: The presence of a base peak at m/z 18 (H_2O) that is higher than m/z 69 in the tune report indicates excessive water remains in the MS manifold. The MS vacuum system does not efficiently remove water and this condition indicates that a longer equilibration time is needed prior to initial calibration.

7.6.1.3 Electron Multiplier Voltage: An electron multiplier voltage higher than 2700 in the tune report indicates that the multiplier needs replacement and/or the MS source needs cleaning.

7.6.1.4 Peak Shape/Resolution of PFTBA Calibration Peaks: The appearance of the PFTBA peaks (m/z 69, 219, 502) used to calibrate the MS should be symmetric without any shoulders. Isotope masses of these same peaks (m/z 70, 220, 503) should be present and indicated in the auto tune report. Non-symmetric peak shape or the non-detection of isotope masses usually indicates that the MS source needs to be cleaned.

7.7 Calibration

7.7.1 Maximum Sensitivity Autotune: The GC/MS system is software-tuned by successfully completing a maximum sensitivity autotune. Perfluorotributylamine (PFTBA) is used as the tuning compound. A hardcopy report is generated from the tune (See Figure 1). See Section 7.6.1 for interpretation of the tune report.

7.7.1.1 Tune calibration check 4-bromofluorobenzene (BFB) is required to be injected in full scan mode once every 12 hours and must pass acceptance criteria (See Figure 2).

7.7.2 Calibration Standards

7.7.2.1 Initial Calibration (ICAL) Curve Preparation: ICALs are prepared in 2.0 mL GC vials at seven calibration levels (See Table 6). A 0.5 mL aliquot of each standard is transferred to 2.0 mL auto-sampler vials and 20 µL of the working internal standard is added prior GC/MS analysis.

7.7.2.2 Prepare a second source verification standard from the intermediate second source standard (See Section 5.10.2) at 200 ng/mL and analyze with each initial calibration.

7.7.3 Accurate Use of Micro-Syringes: Care must be exercised when making a measurement with a micro-syringe that air bubbles are not included and that the syringe barrel does not partially drop during handling thus ejecting a portion of the aliquot. Air bubbles can be eliminated from the syringe by lifting and pumping the syringe plunger quickly a few times prior to measuring the aliquot. Draw the aliquot up slowly for the final measurement. Do not invert the vial containing a standard while filling the syringe as excess standard will run down the outside needle of the syringe resulting in a high bias. When handling an aliquot contained in a syringe, hold the syringe in a manner that a finger is slightly pushing against the extended plunger of the syringe to prevent its movement.

7.7.4 Analyze each of the calibration standards listed in Table 6, collect the data and tabulate the area response of the characteristic ions against the concentration for each target compound using internal standard method. Characteristic ions are listed in Table 3. The internal standard selected for the calculation of these ratios should be the internal standard that has a retention time closest to the compound being measured. The internal standard used for each target compound is provided in Table 7. Calculate amount ratios and response ratios for each target compound and at each calibration level relative to the appropriate internal standard.

The amount ratio and response ratios are calculated as follows:

$$Amount\ Ratio = \frac{C(s)}{C(is)} \qquad Response\ Ratio = \frac{A(s)}{A(is)}$$

Where:

A(s) = Peak area of the target compound or surrogate
A(is) = Peak area of the internal standard
C(s) = Concentration of the target compound or surrogate in µg/L
C(is) = Concentration of the internal standard in µg/L

7.7.4.1 Use of Linear Curves: Linear curves are used for target compounds.

Plot the response ratio against the amount ratio for each of these target compounds. The curve fit is linear regression with the weight of the inverse

of the concentration. The linear regression equation is:

$$Y = m \times x + b$$

Where:
Y = Response Ratio
m = Slope
x = Amount Ratio
b = intercept
1/x = Weighting factor, where x=concentration

The correlation coefficient for these target compounds must be greater than 0.995.

7.7.5 Updating Qualifier Ion Abundances

The relative responses of qualifier ions for target compounds are updated through the data system with each initial calibration. The 200 ng/mL initial calibration standard is used for this purpose. Qualifier ion responses of peaks found in samples are compared to these reference qualifier ion responses for identification. These same qualifier ion ratios are included as a unique window in the Qedit data analysis menu and can further assist the analyst during data review.

7.8 Retention Time Windows

The retention time window for extracted ion chromatograms in peak identification is 0.6 minutes wide. The GC/MS software then identifies the peak in this window with the relative retention time closest to the actual relative retention time determined from the authentic standard. The window must be this wide in order for the software's integration algorithm to have sufficient time to accurately calculate baselines as part of peak integration. The absolute retention time of target compounds should not vary by more than ± 0.05 minutes when compared to the authentic standard. However, retention times are used, only in part, to qualitatively identify compounds as in conventional GC analysis. Rather, this GC/MS method also utilizes the ratio of the qualifier ions for qualitative identification of target compounds.

7.9 Sample Analysis

7.9.1 Samples are analyzed in a group referred to as a GC run which is obtained from the GC Run Log Book. The run number is a 1 up numbering system and is unique for every analytical run. The GC run sequence begins with instrument calibration followed by sample extracts interspersed with CCV standards. The sequence ends when the entire sequence has been injected or when qualitative and/or quantitative QC criteria are exceeded.

7.9.2 Prior to sample analysis the GC/MS system must be tuned and initially calibrated as described in Section 7.7 above. After calibrating the quantitation method, the

same calibration standards are reprocessed by this calibrated method and reported. A second source standard (See Section 7.7.2.2) is included with each initial calibration. It is common to perform the initial calibration in a sequence and data package that is separate from the actual samples. Without an initial calibration, the sequence would start with a continuing calibration verification (CCV) standard followed by a soil blank. Matrix spike samples should be analyzed at the end of the sequence.

7.9.3 Continuing Calibration Verification: A calibrated system requires analysis of CCVs to remain valid. A continuing calibration check (CCV) standard must be injected after every ten samples and at the end of the analysis sequence. Concentration of the CCVs is alternated between 200 and 1000 ng/mL. The response for each compound to be quantitated must not exceed a 20% difference when compared to the theoretical value of the calibration standard. When this criterion is exceeded, inspect the GC/MS system to determine the cause and perform whatever maintenance is necessary before re-calibrating and proceeding with sample analysis. All samples that were injected after the last acceptable CCV should be re-injected, or the data may be appropriately qualified back to the last acceptable CCV. An acceptable calibration curve has a coefficient of determination (R^2) of 0.995 or greater.

$$Percent\ Difference = \frac{R2 - R1}{R1} \times 100$$

Where: R1 = Theoretical Concentration.
R2 = Calculated Concentration from succeeding analyses.

7.9.4 Qualitative analysis

The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample qualifier ion ratios with reference qualifier ion ratios. The reference qualifier ion ratios must be generated using the conditions of this method. Compounds are identified as present when the following criteria are met.

7.9.4.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.9.4.2 The absolute retention time of the sample component is within ± 0.05 minutes of the absolute retention time of the standard component.

7.9.4.3 The relative intensities of the qualifier ions agree within 30% of the relative intensities of reference qualifier ions. (Example: For an ion with an

abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20 % and 80 %.)

7.10 Calculations

Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the extracted ion current profile of the primary characteristic ion. The concentration in the sample analysis is determined using linear regression as described in Section 7.7.4.1.

7.10.1 Soil Sample Calculations: Concentrations of VOCs in soil are determined on a dry-weight basis by the following formula:

$$\mu g/kg\ \ soil\ sample = (ng/mL\ \ measured) \times \left(\frac{Vm}{W_s}\right) \times \left(\frac{1\ \mu g}{1000\ ng}\right) \div FS$$

Where:
V_m = Volume of methanol used to extract samples in mL, normally 10 mL
W_s = Wet weight of soil extracted in kg, nominally 0.010 kg
FS = Fraction of dry solids of in the soil sample, approximately 0.85

8 QUALITY CONTROL

Quality control criteria are based upon information found in Methods 8260B from EPA publication SW-846. Additional criteria may be found in Method 8000 when not specified in Method 8260B.

8.1 The MSD must successfully complete a maximum sensitivity autotune (See Section 7.7.1 and Figure 1).

8.2 There must be an initial 7 point calibration of the GC/MS system. The correlation coefficient must be greater than 0.995.

8.3 A continuing calibration check (CCV) standard must be injected after every ten samples and at the end of the analysis sequence. Concentration of the CCV is alternated between 200 and 1000 ng/mL. The percent difference between the measured response and the theoretical response for analytes in the CCV standard must be within 20% (See Section 7.9.3). Corrective action may include, reanalyzing the CCV (See Section 7.9.3), preparing a new intermediate standard (See Section 5.5), preparing a new calibration curve (See Section 7.7), reanalyzing the affected samples or qualifying the data.

8.4 The response of the internal standards must not vary by < 50% or > 200% from the midpoint calibration standard response generated during the initial 7 point calibration.

8.5 A method blank is prepared and analyzed on each analysis day and after every twenty samples. The method blank includes silica sand and is carried through all the steps of sample preparation and analysis. In the event that VOCs are measured in the blank

above the reporting limit the sample data must be qualified and the value measured in the blank reported with the samples.

8.6 Matrix spike and matrix spike duplicate (MS/MSD) samples are analyzed on each analysis day and after every twenty samples. Acceptable recoveries are 70-130%. Any indication of a potential matrix effect should be communicated to the client and the sample data is appropriately qualified.

8.7 Surrogate standards are added to all samples as system monitoring compounds for each analysis. Acceptable recoveries for surrogates are 60-140%. Samples that have surrogate recoveries outside of control limits are reanalyzed or the sample data is qualified.

9 PERFORMANCE DATA

9.1 The method detection limit (MDL) for applicable compounds has been determined for soils from the analysis of seven replicates fortified at 10 µg/ kg. The MDLs are presented in Table 1.

9.2 An initial demonstration of capability (IDC) will be performed prior to sample receipt. The IDC summary is listed in Table 2.

10 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

10.1 Contingencies for out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and filed with the Quality Manager. Analytical results shall be qualified as necessary.

11 WASTE MANAGEMENT / POLLUTION PREVENTION

11.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

12 REFERENCES

12.1 The technical elements and procedural requirements of the methods cited below were considered in preparation of this SOP.

12.1.1 Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260B, SW-846, Test Methods for Evaluating Solid Wastes, Update III Revision 2, December 1996

12.1.2 Determinative Chromatographic Separations, Method 8000B, SW-846, Test Methods for Evaluating Solid wastes, Update III, Revision 2, December 1996

12.1.3 Analytical Detection Limit Guidance & Laboratory Guide for Determining Method Detection Limits, Wisconsin Department of Natural Resources Laboratory Certification Program, April 1996, PUBL-TS-056-96

TABLE 1

MINIMUM DETECTION LIMITS (MDL) AND REPORT LIMITS FOR SOIL SAMPLES

Compound	Spike Level (µg/kg)	Mean Recovery	Percent Recovery	Percent RSD	MDL (µg/kg)	Report Limit (µg/kg)
Benzene	10	9.92	99	10.45	3.1	25
Toluene	10	8.78	88	11.03	2.90	25
Ethylbenzene	10	10.15	101	5.10	1.6	25
m+p-Xylene	20	19.32	97	4.68	2.7	10
o-Xylene	10	9.84	98	18.10	5.3	5.3
1,3,5-Trimethylbenzene	10	9.56	96	11.39	3.3	25
1,2,4-Trimethylbenzene	10	10.01	100	6.97	2.1	25

TABLE 2

INITIAL DEMONSTRATION OF CAPABILITY (IDC) FOR SOIL SAMPLES

Compound	Spike Level (µg/kg)	Mean Recovery	Percent Recovery	Percent RSD
Benzene	200	228	114	6.3
Toluene	200	267	134	5.3
Ethylbenzene	200	221	110	5.8
m+p-Xylene	400	442	110	4.1
o-Xylene	200	202	101	4.5
1,3,5-Trimethylbenzene	200	247	123	6.5
1,2,4-Trimethylbenzene	200	235	118	5.8

TABLE 3

ABSOLUTE RETENTION TIME ORDER

Compound	**Absolute RT (min)**
Benzene	3.32
1,4-difluorobenzene (IS)	3.46
Toluene-d_8 (Surrogate)	5.27
Toluene	5.35
Chlorobenzene-d_5 (IS)	6.68
Ethylbenzene	6.97
m,p-Xylene	7.10
o-Xylene	7.45
1,3,5-Trimethylbenzene	8.47
1,2,4-Trimethylbenzene	8.79
1,4-Dichlorobenzene-d_4 (IS)	8.96

NOTE: (1) RTX-1, 30 m x 0.32 mm, 1.5 µm

TABLE 3

CHARACTERISTIC MASSES (m/z)

Compound	**Primary Characteristic Ion**	**Secondary Characteristic Ion(s)**
Benzene	78	77,
Toluene	92	91
Toluene-d_8	98	100
Ethylbenzene	91	106
o, m, p – Xylenes	91	106
1,3,5-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,4-Difluorobenzene	114	63
Chlorobenzene-d_5	117	82
1,4-Dichlorobenzene-d_4	150	115

TABLE 4

STOCK STANDARD CONCENTRATIONS

Compound	Solution	Concentration (µg/mL)
Benzene	Absolute, Part #90379	1000
Toluene	Absolute, Part #90379	1000
Ethylbenzene	Absolute, Part #90379	1000
o – Xylene	Absolute, Part #90379	1000
m – Xylene	Absolute, Part #90379	1000
p – Xylene	Absolute, Part #90379	1000
1,3,5-Trimethylbenzene	Absolute, Part #90379	1000
1,2,4-Trimethylbenzene	Absolute, Part #90379	1000
Internal Standards		
1,4-Difluorobenzene	Restek, Part #30074	2500
Chlorobenzene-d_5	Restek, Part #30074	2500
1,4-Dichlorobenzene-d_4	Restek, Part #30074	2500
Surrogate		
Toluene-d_8	Absolute, Part #70282	1000
Second Source		
Benzene	Restek, Part #30095	1000
Toluene	Restek, Part #30095	1000
Ethylbenzene	Restek, Part #30095	1000
o – Xylene	Restek, Part #30095	1000
m – Xylene	Restek, Part #30095	1000
p – Xylene	Restek, Part #30095	1000
1,3,5-Trimethylbenzene	Restek, Part #30095	1000
1,2,4-Trimethylbenzene	Restek, Part #30095	1000

TABLE 5

INTERMEDIATE STANDARD SOLUTION PREPARATION (10,000 ng/mL)

Description	Absolute Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (ng/mL)
BTEX, TMBs	90379	1000	500	50	10000
Toluene-d_8	70282	1000	500	50	10000

NOTES: (1) Solvent: Methanol

TABLE 6

INITIAL CALIBRATION STANDARDS

Calibration Level	**L-1**	**L-2**	**L-3**	**L-4**	**L-5**	**L-6**	**L-7**
Volume of Intermediate Standard (Table 5) Aliquoted (µL)	25	50	125	500	1000	2500	5000
Final Volume (mL)	50	50	50	50	50	50	50
Final Concentration (ng/mL)	5	10	25	100	200	500	1000

NOTE: (1) Solvent: Methanol

TABLE 7

INTERMEDIATE SECOND SOURCE STANDARD SOLUTION PREPARATION

Description	Restek Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (ng/mL)
BTEX, TMBs	30095	1000	500	50	10000

TABLE 8

WORKING INTERNAL STANDARD SOLUTION PREPARATION (50 µg/mL)

Description	Restek Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
8260 Internal Standards	30074	2500	200	50	10

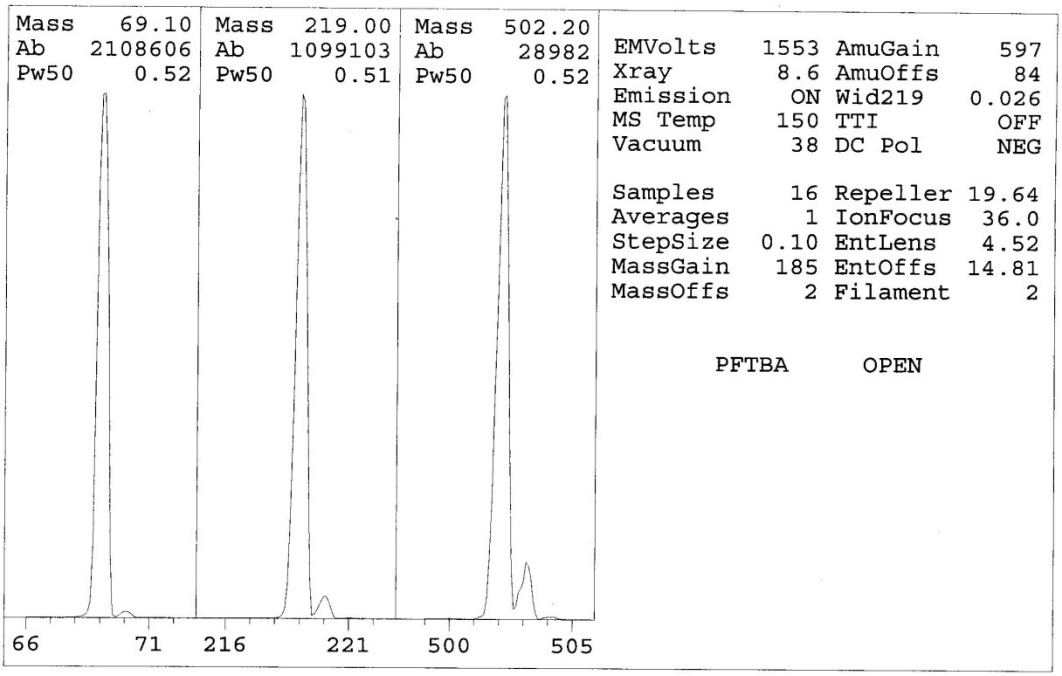
TABLE 9

WORKING SURROGATE STANDARD SOLUTION PREPARATION (100 µg/mL)

Description	Absolute Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
Toluene-d_8	70282	1000	500	25	20

FIGURE 1
MAXIMUM SENSITIVITY AUTOTUNE REPORT

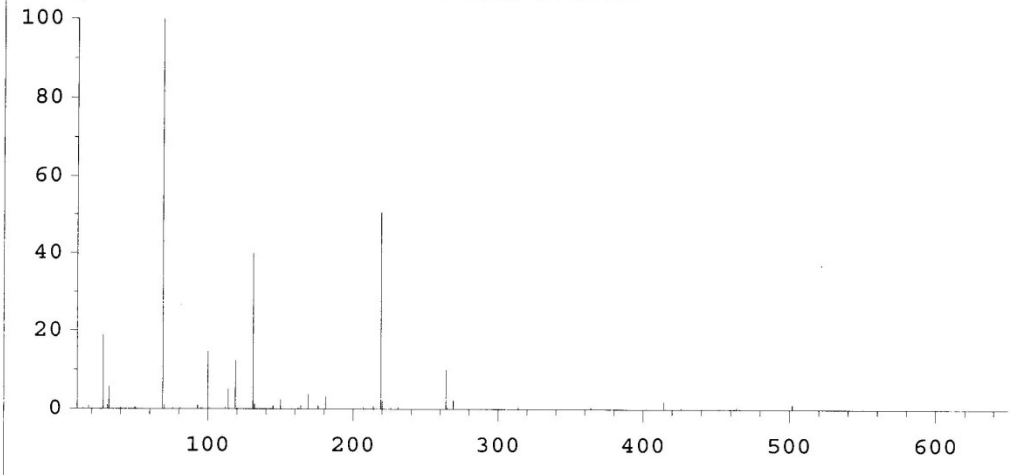
HP5971 Maximum Sensitivity Autotune
Instrument: GC/MS Instrument #1
Tue Feb 12 12:26:47 2008 D:\HPCHEM\1\5971\ATUNE.U



EMVolts	1553	AmuGain	597
Xray	8.6	AmuOffs	84
Emission	ON	Wid219	0.026
MS Temp	150	TTI	OFF
Vacuum	38	DC Pol	NEG
Samples	16	Repeller	19.64
Averages	1	IonFocus	36.0
StepSize	0.10	EntLens	4.52
MassGain	185	EntOffs	14.81
MassOffs	2	Filament	2

PFTBA OPEN

Scan: 10.00 - 650.00 Samples: 16 Thresh: 150
227 peaks Base: 69.00 Abundance: 1781248

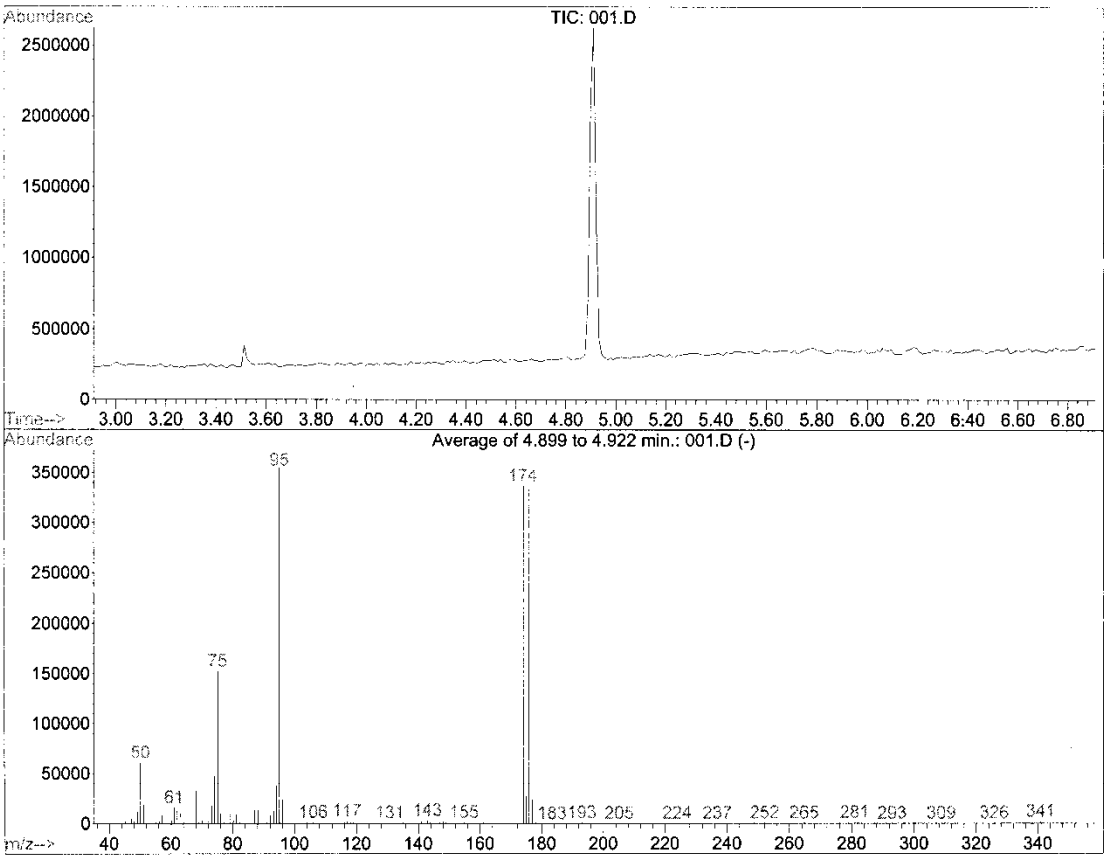


Mass	Abund	Rel Abund	Iso Mass	Iso Abund	Iso Ratio
69.00	1781248	100.00	70.00	19560	1.10
218.95	904128	50.76	219.95	39224	4.34
502.05	23968	1.35	503.05	2416	10.08

FIGURE 2
BFB TUNE REPORT

BFB

```
Data File : D:\HPCHEM\1\DATA\GC-2053\001.D              Vial: 1
Acq On    : 27 Jun 2008    3:06 pm                  Operator: cps
Sample    : bfb,50ug/ml.1ul                         Inst    : 33329A008
Misc      :                                         Multiplr: 1.00
MS Integration Params: RTEINT.P
Method    : D:\HPCHEM\1\METHODS\NRT0630.M (RTE Integrator)
Title     :
```



AutoFind: Scans 218, 219, 220; Background Corrected with Scan 214

Target Mass	Rel. to Mass	Lower Limit%	Upper Limit%	Rel. Abn%	Raw Abn	Result Pass/Fail
50	95	15	40	17.2	61180	PASS
75	95	30	60	43.1	153014	PASS
95	95	100	100	100.0	355301	PASS
96	95	5	9	6.6	23493	PASS
173	174	0.00	2	0.0	0	PASS
174	95	50	100	95.0	337515	PASS
175	174	5	9	7.9	26656	PASS
176	174	95	101	99.9	337271	PASS
177	176	5	9	7.1	24033	PASS

001.D NRT0630.M Wed Jul 09 15:46:29 2008

FIGURE 3
TOTAL ION CHROMATOGRAM OF A 200 ng/mL CCV AND DETAILED REPORT

Quantitation Report (Not Reviewed)

Data File : D:\HPCHEM\1\DATA\GC-2053\011.D Vial: 11
Acq On : 27 Jun 2008 7:04 pm Operator: cps
Sample : ccs,200ng/ml Inst : 33329A008
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Jun 30 10:43 2008 Quant Results File: NRT0627.RES

Quant Method : D:\HPCHEM\1\METHODS\NRT0627.M (RTE Integrator)
Title :
Last Update : Mon Jun 30 10:35:54 2008
Response via : Initial Calibration
DataAcq Meth : NRT

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) 1,4-Difluorobenzene	3.84	114	154525	400.00	ng/ml	0.00
3) Chlorobenzene-d5	7.01	117	101845	400.00	ng/ml	0.01
9) 1,4-Dichlorobenzene-d4	9.84	150	71071	400.00	ng/ml	0.00
System Monitoring Compounds						
4) Toluene-d8	5.49	98	76631	223.28	ng/ml	0.00
Spiked Amount 200.000			Recovery =	111.64%		
11) Phenol-d8	9.07	99	11581	146.74	ng/ml	0.00
Spiked Amount 200.000			Recovery =	73.37%		
Target Compounds						Qvalue
2) Benzene	3.64	78	98567	225.53	ng/ml	100
5) Toluene	5.56	92	54085	216.98	ng/ml	99
6) Ethylbenzene	7.35	91	88082	219.67	ng/ml	99
7) m+p-Xylene	7.50	91	136592	434.38	ng/ml	100
8) o-Xylene	7.94	91	67721	214.34	ng/ml	99
10) Phenol	9.10	94	12917	122.43	ng/ml	97
12) 1,3,5-Trimethylbenzene	9.27	105	62158	229.06	ng/ml	96
13) 1,2,4-Trimethylbenzene	9.66	105	61042	227.13	ng/ml	99
14) 2-Methylphenol	10.18	108	14259	176.87	ng/ml	100
15) 4-Methylphenol	10.41	108	11393	154.03	ng/ml	94
16) 2,4-Dimethylphenol	11.27	122	13969	177.29	ng/ml	95

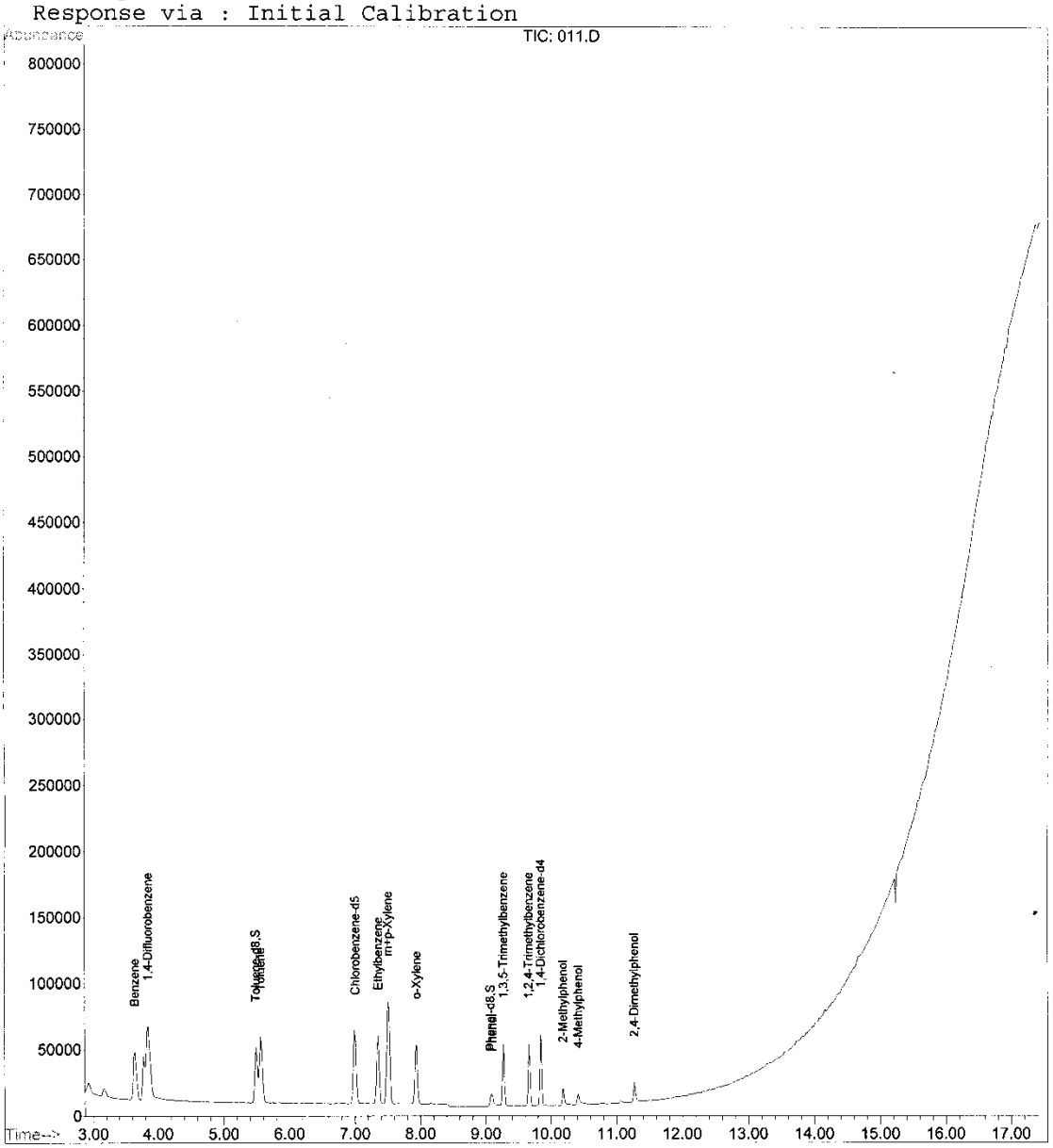
(#) = qualifier out of range (m) = manual integration
011.D NRT0630.M Wed Jul 09 15:46:40 2008 Page 1

FIGURE 3
TOTAL ION CHROMATOGRAM OF A 200 ng/mL CCV AND DETAILED REPORT

Quantitation Report

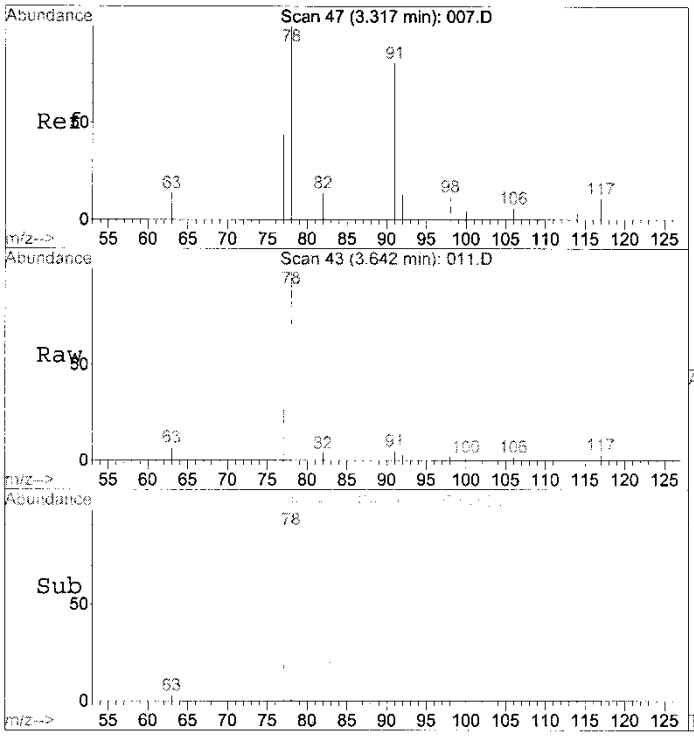
Data File : D:\HPCHEM\1\DATA\GC-2053\011.D Vial: 11
Acq On : 27 Jun 2008 7:04 pm Operator: cps
Sample : ccs,200ng/ml Inst : 33329A008
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Jun 30 10:43 2008 Quant Results File: NRT0627.RES

Method : D:\HPCHEM\1\METHODS\NRT0630.M (RTE Integrator)
Title :
Last Update : Tue Jul 01 10:39:43 2008
Response via : Initial Calibration



011.D NRT0630.M Wed Jul 09 15:46:41 2008 Page 2

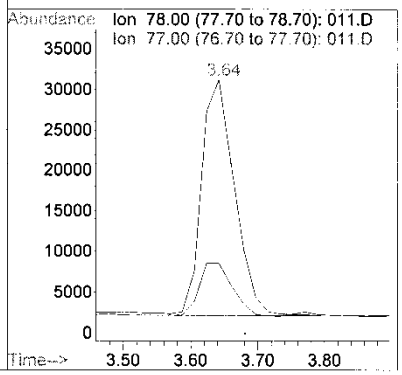
FIGURE 3
TOTAL ION CHROMATOGRAM OF A 200 ng/mL CCV AND DETAILED REPORT

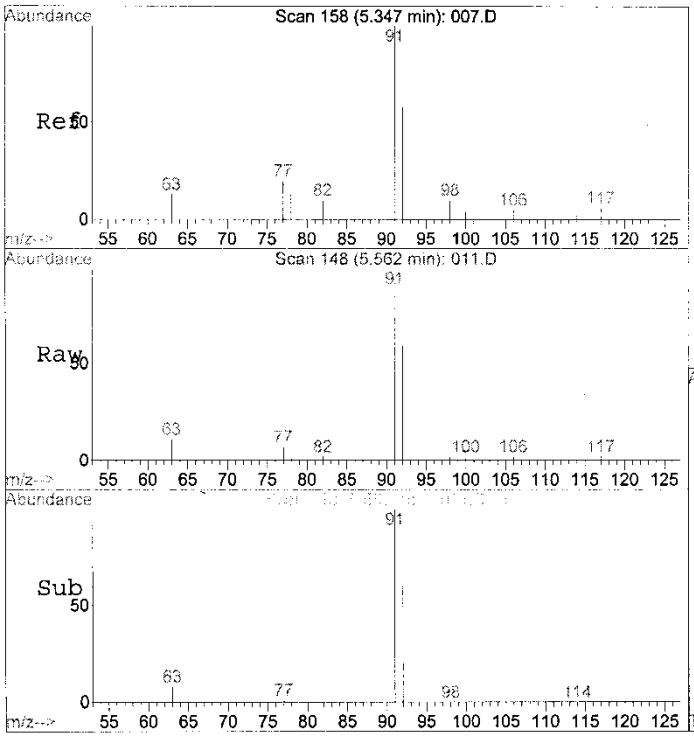


#2
Benzene
Concen: 225.53 ng/ml
RT: 3.64 min Scan# 43
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion: 78 Resp: 98567

Ion	Ratio	Lower	Upper
78	100		
77	22.6	18.1	27.1





#5
Toluene
Concen: 216.98 ng/ml
RT: 5.56 min Scan# 148
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion: 92 Resp: 54085

Ion	Ratio	Lower	Upper
92	100		
91	168.5	133.4	200.0

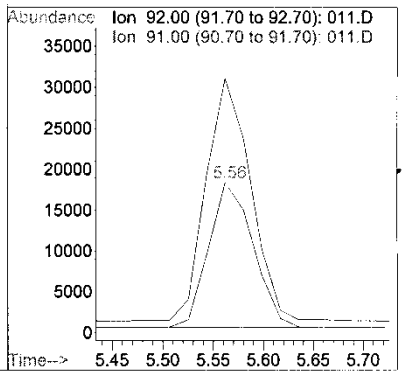
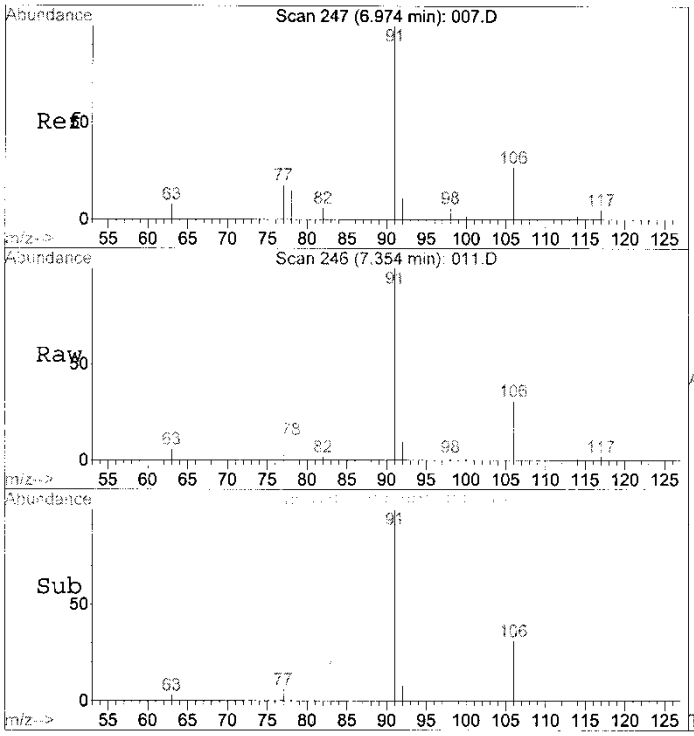


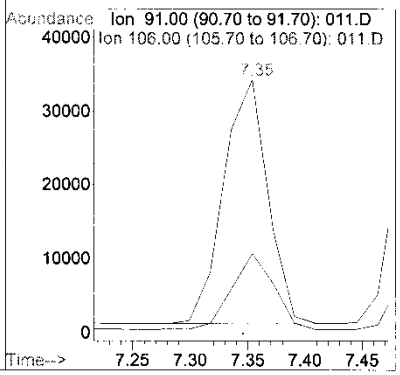
FIGURE 3
TOTAL ION CHROMATOGRAM OF A 200 ng/mL CCV AND DETAILED REPORT

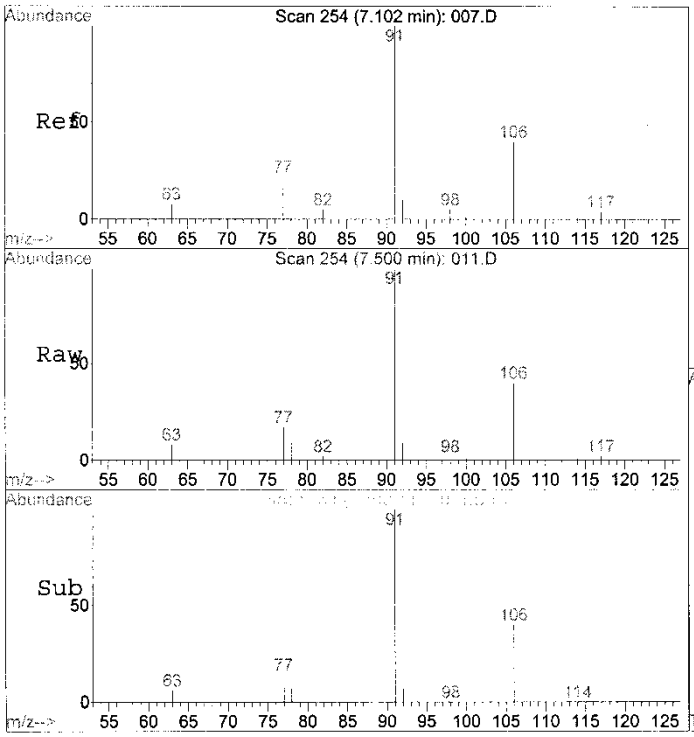


#6
Ethylbenzene
Concen: 219.67 ng/ml
RT: 7.35 min Scan# 246
Delta R.T. 0.01 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion: 91 Resp: 88082

Ion	Ratio	Lower	Upper
91	100		
106	29.4	23.3	34.9





#7
m+p-Xylene
Concen: 434.38 ng/ml
RT: 7.50 min Scan# 254
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion: 91 Resp: 136592

Ion	Ratio	Lower	Upper
91	100		
106	46.1	37.1	55.7

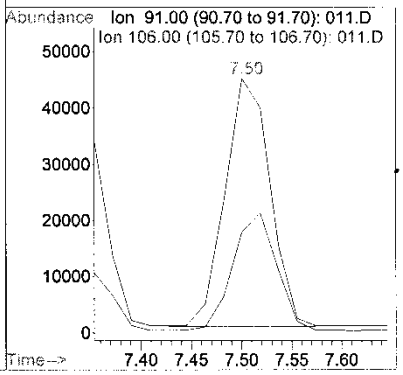
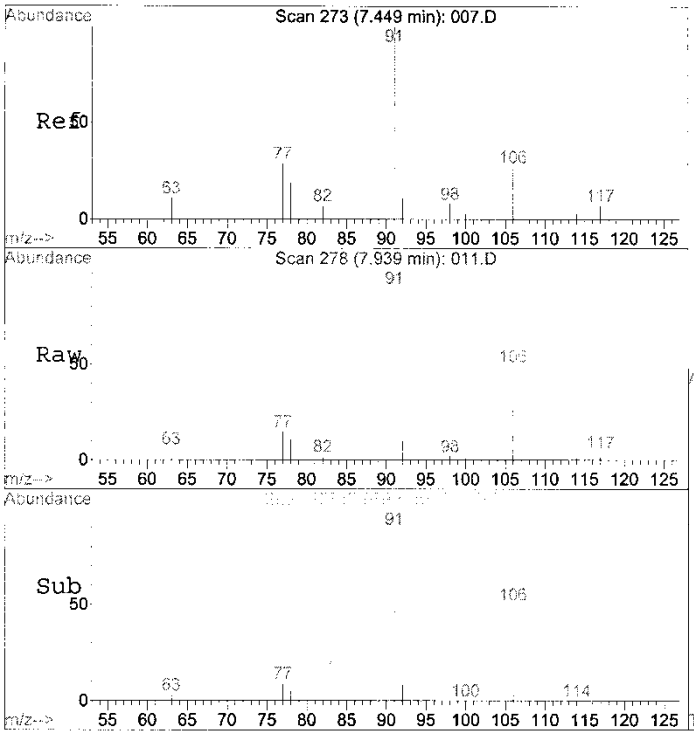


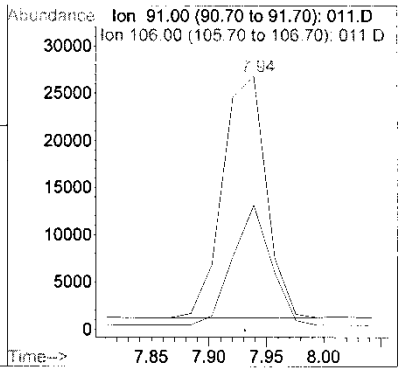
FIGURE 3
TOTAL ION CHROMATOGRAM OF A 200 ng/mL CCV AND DETAILED REPORT

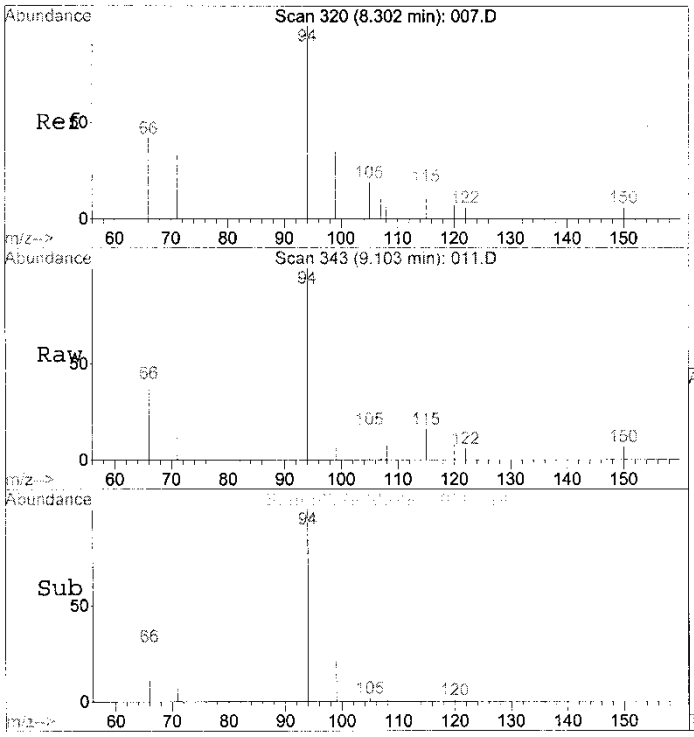


#8
o-Xylene
Concen: 214.34 ng/ml
RT: 7.94 min Scan# 278
Delta R.T. 0.01 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion: 91 Resp: 67721

Ion	Ratio	Lower	Upper
91	100		
106	43.7	35.4	53.2





#10
Phenol
Concen: 122.43 ng/ml
RT: 9.10 min Scan# 343
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion: 94 Resp: 12917

Ion	Ratio	Lower	Upper
94	100		
66	32.4	27.4	41.2

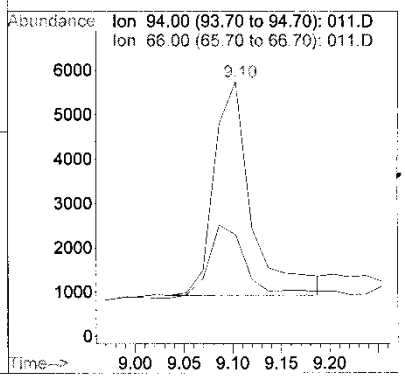
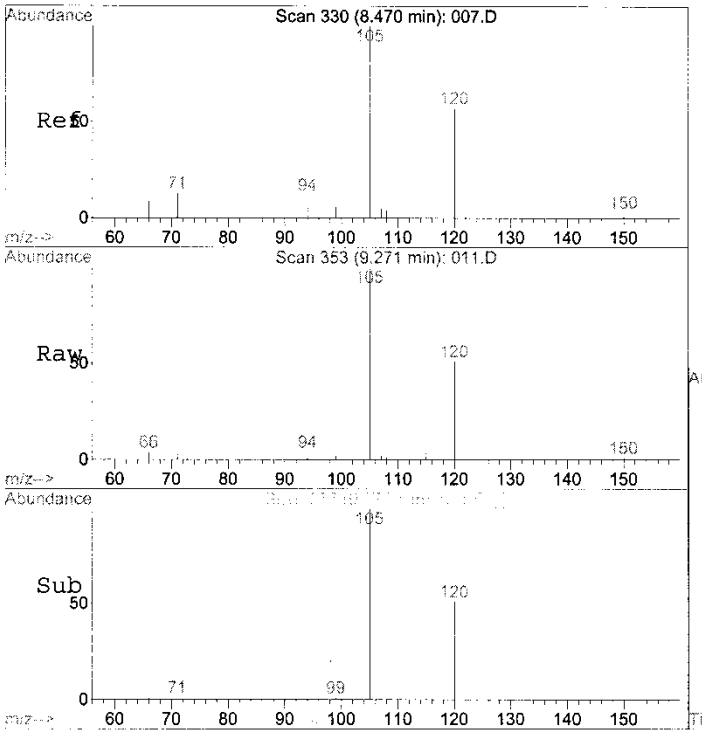


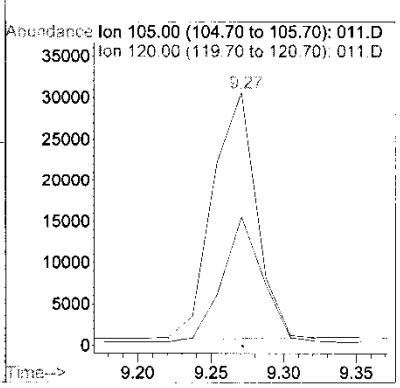
FIGURE 3
TOTAL ION CHROMATOGRAM OF A 200 ng/mL CCV AND DETAILED REPORT

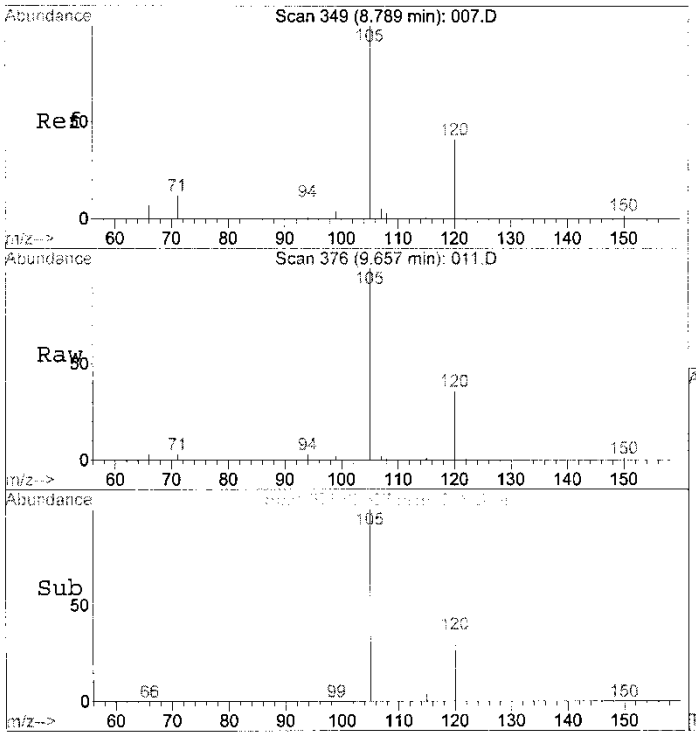


#12
1,3,5-Trimethylbenzene
Concen: 229.06 ng/ml
RT: 9.27 min Scan# 353
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion:105 Resp: 62158

Ion	Ratio	Lower	Upper
105	100		
120	46.6	39.3	58.9





#13
1,2,4-Trimethylbenzene
Concen: 227.13 ng/ml
RT: 9.66 min Scan# 376
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion:105 Resp: 61042

Ion	Ratio	Lower	Upper
105	100		
120	44.3	34.9	52.3

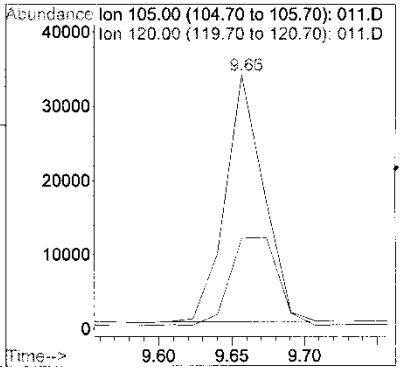
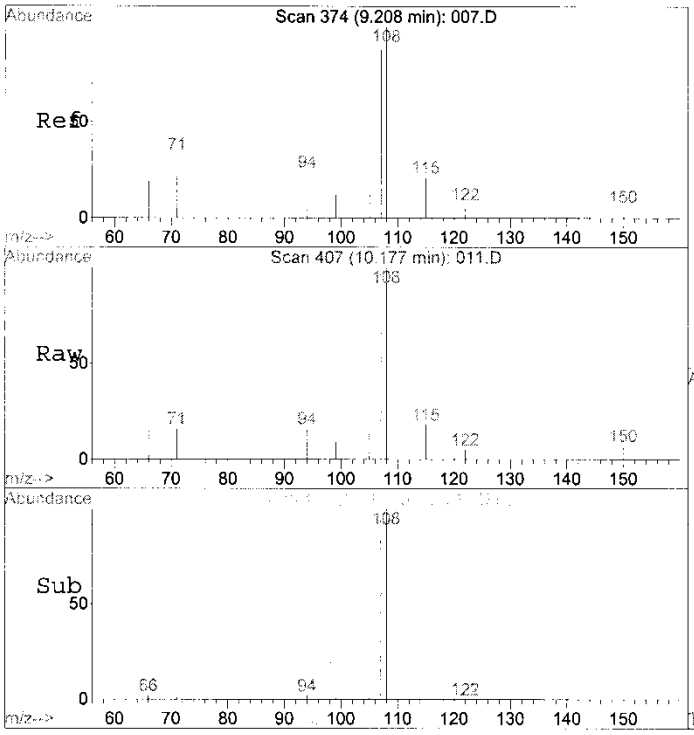


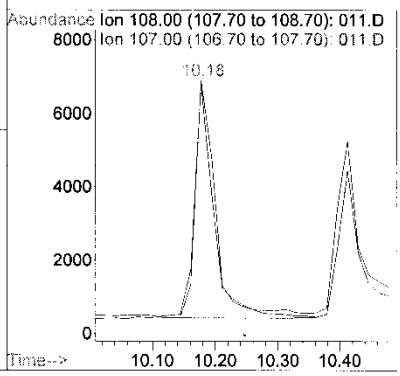
FIGURE 3
TOTAL ION CHROMATOGRAM OF A 200 ng/mL CCV AND DETAILED REPORT

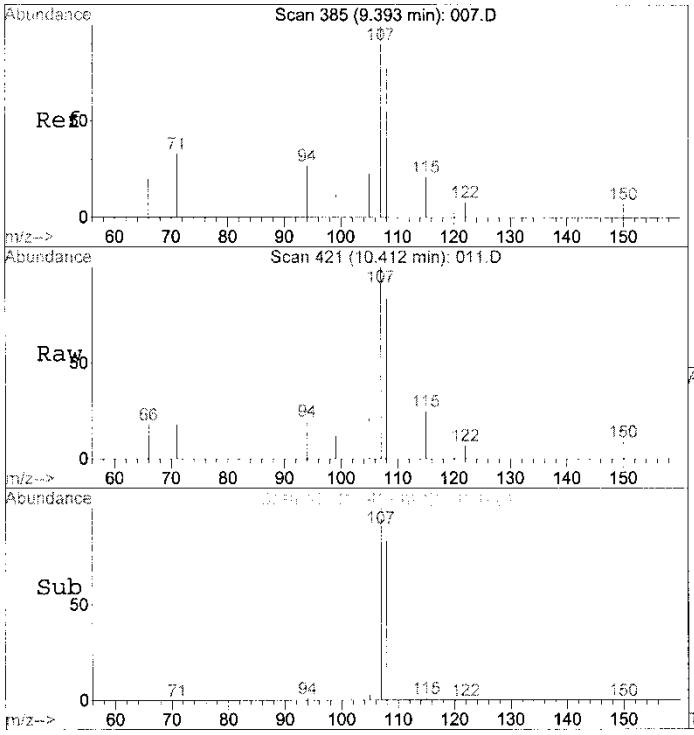


#14
2-Methylphenol
Concen: 176.87 ng/ml
RT: 10.18 min Scan# 407
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion:108 Resp: 14259

Ion	Ratio	Lower	Upper
108	100		
107	89.9	71.7	107.5





#15
4-Methylphenol
Concen: 154.03 ng/ml
RT: 10.41 min Scan# 421
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion:108 Resp: 11393

Ion	Ratio	Lower	Upper
108	100		
107	124.2	93.8	140.8

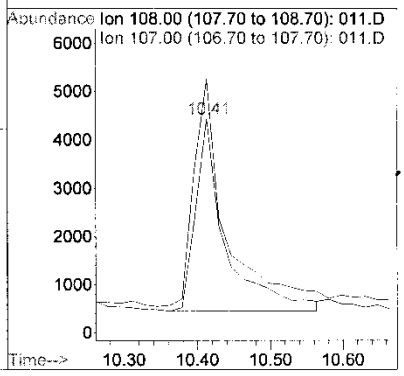
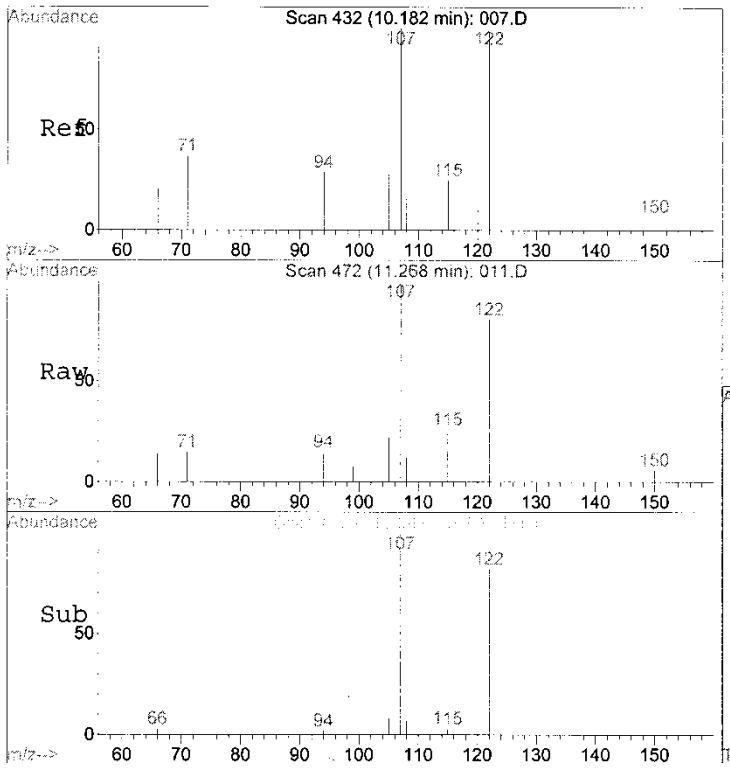


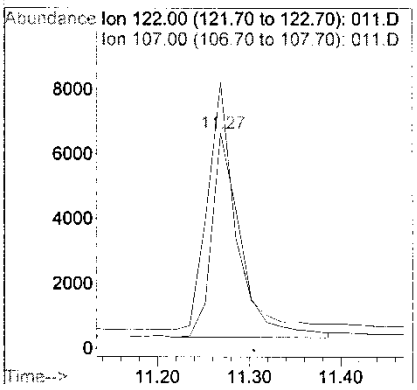
FIGURE 3
TOTAL ION CHROMATOGRAM OF A 200 ng/mL CCV AND DETAILED REPORT



#16
2,4-Dimethylphenol
Concen: 177.29 ng/ml
RT: 11.27 min Scan# 472
Delta R.T. -0.00 min
Lab File: 011.D
Acq: 27 Jun 2008 7:04 pm

Tgt Ion:122 Resp: 13969

Ion	Ratio	Lower	Upper
122	100		
107	114.5	87.4	131.0



The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

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VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP/GAS CHROMATOGRAPHY/MASS SPECTROMETRY

1 SCOPE AND APPLICATION

1.1 This method is a capillary gas chromatography/mass spectrometry (GC/MS) method used to determine part per billion concentrations of volatile organic compounds (VOC). The volatiles are introduced into the GC/MS system by the purge and trap technique. This method is based on Method 5030B and 8260B, and is used to determine volatile organic compounds in a variety of matrices. This method is applicable to soil, water, and waste samples.

Compound	CAS No.
1,1,1,2-Tetrachloroethane	630-20-6
1,1,1-Trichloroethane (1,1,1-TCA)	71-55-6
1,1,2,2-Tetrachloroethane	79-34-5
1,1,2-Trichloroethane (1,1,2-TCA)	79-00-5
1,1,2-Trichlorotrifluoroethane	76-13-1
1,1-Dichloroethane (1,1-DCA)	75-34-3
1,1-Dichloroethene (1,1-DCE)	75-35-4
1,1-Dichloropropene	563-58-6
1,2,3-Trichlorobenzene	87-61-6
1,2,3-Trichloropropane	96-18-4
1,2,4-Trichlorobenzene	120-82-1
1,2,4-Trimethylbenzene	95-63-6
1,2-Dibromo-3-chloro-propane	96-12-8
1,2-Dibromoethane	106-93-4
1,2-Dichlorobenzene	95-50-1
1,2-Dichloroethane (1,2-DCA)	107-06-2
1,2-Dichloropropane	78-87-5
1,3,5-Trimethylbenzene	108-67-8
1,3-Dichlorobenzene	541-73-1
1,3-Dichloropropane	142-28-9
1,4-Dichlorobenzene	106-46-7
2,2-Dichloropropane	594-20-7
2-Butanone (MEK)	78-93-3
2-Chlorotoluene	95-49-8
2-Hexanone	591-78-6
2-Pentanone,4-methyl (MIBK)	108-10-1
4-Chlorotoluene	106-43-4
Acetone	67-64-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5

Compound	CAS No.
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
Carbon Disulfide	75-15-0
Carbon Tetrachloride	56-23-5
Chlorobenzene	08-90-7
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
cis-1,2-Dichloroethene (cis-1,2-DCE)	156-59-2
cis-1,3-Dichloropropene	10061-01-5
Dibromochloromethane	124-48-1
Dibromomethane	74-95-3
Dichlordifluoromethane	75-71-8
Di-isopropylether	108-20-3
Ethylbenzene	100-41-4
Hexachlorobutadiene	87-68-3
Isopropylbenzene	98-82-8
Methylene chloride	75-09-2
Methyl-t-butylether (MTBE)	1634-04-4
m-Xylene	108-38-3
Naphthalene	91-20-3
n-Butylbenzene	104-51-8
n-Hexane	110-54-3
n-Propylbenzene	103-65-1
o-Xylene	95-47-6
p-Isopropyltoluene	99-87-6
p-Xylene	106-42-3
sec-Butylbenzene	135-98-8
Styrene	100-42-5
tert-Butylbenzene	98-06-6

Compound	CAS No.	Compound	CAS No.
Tetrachloroethene (PCE)	127-18-4	trans-1,3-Dichloropropene	10061-02-6
Tetrahydrofuran	109-99-9	Trichloroethene (TCE)	79-01-6
Toluene	08-88-3	Trichlorofluoromethane	75-69-4
trans-1,2-Dichloroethene	156-60-5	Vinyl chloride	75-01-4

1.2 Instrumentation: Hewlett Packard (HP) 5971 Mass Selective Detector (MSD), HP 5890 Gas Chromatograph, two Tekmar LSC 2000 Controller and ALS 2016 Autosampler purge and trap systems configured in series with a Tekmar Duet Interface Controller, Agilent MSD Productivity software with Enviroquant data analysis menu

1.3 This method is used to identify and quantitate volatile organic compounds that have boiling points below 200 °C.

1.4 The GC/MSD provides for selective detection of the VOCs by extracting specific ions for quantitation and qualification from the total ion chromatogram. Compound identification is performed by comparing the unknown compounds retention time and spectra against reference mass spectrum and retention time acquired from authentic standards.

1.5 Table 1 provides statistically determined method detection limits (MDL) and report limits for target compounds in reagent water and silica sand. In general, water samples are reported to 0.5 µg/L whereas soils are reported to 25 µg/kg on a wet weight basis. Reporting limits for soils on a dry weight basis are adjusted for the percent moisture in the sample. Some compounds have higher reporting limits due to instrument conditions or performance.

1.6 Typical demonstration of capability (DOC) data for reagent water and silica sand are reported in Table 2.

1.7 The Chemist should be a chromatography expert capable of operating and maintaining the purge and trap GC/MS system and experienced in the analysis of VOCs. Training includes generating a valid initial calibration and an acceptable initial demonstration of capability study in addition to understanding the use of mass spectra for identification and quantitation.

2 SUMMARY OF METHOD

2.1 Waters are analyzed directly without any sample preparation by GC/MS purge and trap. Soils require extraction with methanol. An aliquot is then added to reagent water and analyzed by purge and trap GC/MS.

2.2 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. This method purges the VOCs from the water matrix into a stream of inert helium gas. The VOCs are trapped from the gas stream onto an organic polymer at room temperature. When the polymer is rapidly heated, the VOCs desorb back into

the gas stream and enter the GC. The analytes are introduced directly to a narrow-bore capillary column from the purge and trap using a direct split-injector interface at the gas chromatograph (GC). The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) directly interfaced to the GC. A total ion chromatogram of a mid-range calibration standard is provided in Figure 2.

2.3 Identification of target analytes is accomplished by comparing their mass spectra with the mass spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using an average response factor regression type analysis.

3 INTERFERENCES

3.1 Major contaminant sources are volatile materials and solvents that enter the atmosphere or diffuse into the de-ionized water which will be concentrated on the trap during the purge and trap operation. Special care must be used with solvents to avoid methylene chloride and acetone contamination. The VOC laboratory is isolated from the remainder of the laboratory across the parking lot in the annex. No solvents other than methanol must enter this laboratory. Methanol is stored apart from other solvents in the VOA annex.

3.1.1 Chloromethane can form as a by-product with hydrochloric acid preservation of water samples. Chloromethane can also be generated on a used trap contaminated with salts in the purge and trap system. Corrective action should be taken whenever chloromethane levels exceed 0.5 µg/L. Corrective action could include, but not be limited to, replacing the trap or using an alternative chemical preservative for waters.

3.1.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free water or methanol and carried through the sampling, handling, and storage protocols serves as a check on such contamination. Trip blanks are issued to clients with each bottle order.

3.2 Contamination of the purge and trap system will occur when a sample is analyzed containing VOCs that exceed 100 µg/L. Erroneous positive results will occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing very high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations of volatile organic compounds, a calibration blank should be analyzed to check for cross contamination. This carry-over is minimized by application of single use glassware but not eliminated.

3.2.1 The purge and trap should be programmed to run through a “clean” cycle prior to the analysis of actual samples. This clean cycle is a two step process. In the first

step, no purge tube is attached and for 1.5 minutes excess water is blown out of the purge lines. In the second step, a purge tube is attached and for 5 minutes clean helium gas is purged through the system. Finally, the sample is loaded into the purge tube through the 3-way valve without breaking the tube's inert seal (See Section 7.6.1, Table 8).

3.2.2 Screening of unfamiliar samples prior to purge-and-trap GC/MS analysis is recommended to prevent contamination of the system. Screening of soil samples is accomplished by analyzing an aliquot of the soil methanol extract first by GC with either electron capture detection (ECD) or flame ionization detection (FID). Screening of water samples is accomplished with the purge and trap GC/MS system by performing a minimum 100 fold dilution on the sample. Alternatively, water samples may be screened by GC/ECD for common chlorinated VOCs – 1,1-DCE, 1,1-DCA, cis-1,2-DCE, PCE, TCE, and 1,1,1-TCA.

3.3 High levels of petroleum hydrocarbons, such as those found in diesel fuel, will generate interferences for 1,1,1,2-tetrachloroethane, 2-chlorotoluene, 4-chlorotoluene and 1,2,3-trichloropropane. Noisy quantitation ion profiles and poor qualifier ion matches associated with these compounds due to the petroleum interference will be evident during data analysis and will interfere with the accurate analysis of these compounds.

3.4 The internal standard, pentafluorobenzene, co-elutes and interferes with the qualifier ion for 1,1,1-TCA at m/z 61. Poor "Q Value" matches will result for 1,1,1-TCA from this interference at concentrations below 5 µg/L. Pentafluorobenzene elutes within the extraction windows for carbon tetrachloride at m/z 117 and 1,1-dichloropropene at m/z 75. These compounds may be detected by the contribution from pentafluorobenzene; however the retention time will not be accurate. Similarly, the surrogate, bromofluorobenzene, elutes within the extraction window of 1,2,3-trichloropropane at m/z 75.

3.5 Methanol from soil extracts and added to water samples interferes with bromomethane and chloroethane. The methanol has a quenching effect upon the operation of the MSD for the period that it coelutes from the column with these compounds.

3.6 The response of the ketones (acetone, MEK, MIBK, 2-hexanone) and tetrahydrofuran is controlled by their purging efficiency. Their purging efficiency is affected by the ambient temperature of the sample in the purge tube. Every attempt should be made to maintain a stable temperature in the laboratory.

4 APPARATUS AND MATERIALS

4.1 Gas Chromatograph / Mass Spectrometer System

Gas Chromatograph:	HP 5890 with Split/Splitless Injector
Detector:	HP 5971 Mass Selective Detector
Purge and Trap:	2 Tekmar LSC 2000s and ALS 2016s with

	Duet interface
Trap:	Supelco Trap K
Column:	RTX-624, 30 m x 0.32 mm I.D., 1.8 µ film or equivalent
Data System:	Agilent MSD Productivity Chemstation with Enviroquant

4.1.1 GC/MS Supplies List

Seals:	Dual Vespel Ring Inlet Seal, Restek Cat.#21239
Septa:	LB-2 Septa, 11mm, Supelco #164742
Ferrules:	1/16" x 0.5 mm Vespel/Graphite, Restek Cat# 20231/20249
Liners:	Gooseneck Splitless Liner 4 mm x 6.5 mm x 78.5 mm, Restek Cat.#20799

4.2 Gas Chromatograph (Screening Instrument)

Gas Chromatograph:	HP 5890
Detector:	Electron Capture (ECD)
Injector:	Split/Splitless
Column:	RTX-502.2, 105 m x 0.53 mm I.D., 3.0 µ film or equivalent, Restek Cat.#10910
Autosampler:	LEAP Technologies A200S
Data System:	Agilent Chemstation Rev. A.10.01

4.3 Gas Chromatograph (Screening Instrument)

Gas Chromatograph:	HP 5890
Detector:	Flame Ionization (FID)
Injector:	Split/Splitless
Column:	RTX-1, 30 m x 0.53 mm I.D., 0.25 µ film or equivalent, Restek Cat.#10125
Autosampler:	LEAP Technologies A200S
Data System:	Agilent Chemstation Rev. A.10.01

4.4 Gas Chromatograph (Screening Instrument)

Gas Chromatograph:	HP 5890 with Split/Splitless Injector
Detector:	HP 5971 Mass Selective Detector
Column:	RTX-624, 30 m x 0.32 mm I.D., 1.8 µ film or equivalent
Autosampler:	LEAP Technologies A200S
Data System:	Agilent MSD Productivity Chemstation with Enviroquant

4.5 Vortex mixer

4.6 Ultrasonic bath

4.7 Centrifuge: Capable of holding 40 ml VOA vials

4.8 Balances

4.8.1 Top loader capable of weighting to 0.01 gram

4.8.2 Analytical capable of weighting to 0.0001 gram

4.9 Sample syringe: 10 mL Gastight with a Teflon® Luer-lock tip

4.10 Micro-syringes: Gastight, 10, 25, 100, 250, 500, and 1,000 µL

4.11 Vials:

4.11.1 40 mL VOA vials

4.11.2 40 mL VOA vials preserved with 0.5 mL of 1:1 HCL

4.11.3 40 mL VOA vials containing 10 mL of purge and trap grade methanol, labeled and pre-weighed. Used for sampling of soils using Lock and Load system. See SOP "GEN-003, Chain-of-Custody, Log In, Tracking Procedures, and Sample Tracking" for more details.

4.11.4 GC Vials: 2 mL amber autosampler vials Catalog. #40811A-120 and PTFE/silicon aluminum seals, Part #5150-11, LSDC

4.12 "Lock and Load" sampling supplies

4.12.1 Plastic syringes for collection of a nominal 10 gram soil sample, Environmental Sampling Supply Part # LL5035SRNG

4.12.2 Plastic re-usable handle, Environmental Sampling Supply Part # LL5035HNDL

4.13 5 oz. specimen container with lid for collection of soils intended for percent solids analysis, VWR Part # 15704-020 and 15704-019.

4.14 Disposable single use glass purge tubes, 19 x 150 mm glass culture tubes, VWR Part # 45060-19150

4.15 Glass disposable transfer pipettes – 5 ¾"

4.16 Metal hub needles: 18 gauge, 1.27 mm O.D., 0.84 mm I.D. Hamilton part # 90018 or equivalent for attachment to the 10 mL Luer lock syringe when adding makeup methanol to samples

4.17 Optifix solvent dispenser capable of 10 mL volume (EM Science or equivalent)

4.18 Stainless steel spatulas

4.19 Drying oven capable of maintaining 105 °C

4.20 Refrigerator capable of maintaining 4 °C

4.21 Freezer capable of maintaining temperatures below -15 °C

4.22 Plastic 250 to 500 mL squirt bottles

5 REAGENTS

5.1 Solvents

5.1.1 VOC-free water purchased commercially. Roundy's brand distilled water is used.

NOTE: Store in the Annex away from contact with other solvents except methanol.

5.1.2 Methanol: purge & trap grade, stored in the annex apart from other solvents

5.2 Solids

5.2.1 Silica sSand: Baked in a muffle furnace at 400 °C for a minimum of 4 hours

5.3 Acids/bases – not applicable for this method

5.4 Stock Standards

5.4.1 Stock initial calibration standard solutions - Stock solutions are purchased from vendors as certified concentrations in methanol. The vendor is Absolute Standards. The 54 liquids (Part # 32001), 6 gases (Part#30058), and 4 ketones (Part#82402) are purchased at 2000 µg/mL. Additional compounds to complete the target list, MTBE, carbon disulfide, di-isopropyl ether, hexane, tetrahydrofuran, and 1,1,2-trichlorotrifluoroethane are purchased individually at 1000 µg/mL. Their respective part numbers are 70209, 70060, 70987, 70962, 70380, and 70474. Table 4 provides a listing of components in each stock solution. Stocks are used only once when the ampoule is opened and then diluted to prepare the working solution.

5.5 Intermediate Standard Solutions:

5.5.1 Method A+B+C (5/25/50 µg/mL)

5.5.1.1 The working intermediate standard solution (5/25/50 µg/mL) is prepared by diluting the stock standard solutions (Section 5.4.1) in a 50 mL volumetric flask with methanol.

5.5.1.2 The working standard is prepared at 5 µg/mL for all targets except the ketones (50 µg/mL) and tetrahydrofuran (25 µg/mL).

5.5.1.3 Fill a 50 mL volumetric flask ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that a syringe needle tip can be submerged in the methanol.

5.5.1.4 Aliquots of the stock are added below the level of methanol in the flask from the syringe needle.

NOTE: Table 5 summarizes the preparation of this standard.

5.5.1.4.1 Using a 250 µL Gastight syringe, aliquot 125 µL of the 2000 µg/mL liquid (32001) and gas stock (30058) to the 50 mL volumetric.

5.5.1.4.2 Using a 250 µL Gastight syringe, aliquot 250 µL of the individual standards (70209, 70060, 70987, 70962, and 70474) to the 50 mL volumetric.

5.5.1.4.3 Using a 1 mL and a 250 µL Gastight syringe, aliquot 1250 µL of ketones (82402) and tetrahydrofuran (70380) to the 50 mL volumetric.

5.5.1.5 Once all components are added to the 50 mL volumetric, make to volume with purge and trap grade methanol.

5.5.1.6 The standard is subdivided into 2 mL GC vials. Make sure to fill each vial full and cap. Store frozen and assign a one month expiration date.

5.5.2 Method C (50/250/500 µg/mL)

5.5.2.1 The working intermediate standard solution (50/250/500 µg/mL) is prepared by diluting the stock standards (Section 5.4.1) into a 10 mL volumetric flask.

5.5.2.2 Working as quickly as possible aliquot each of the stocks as listed in Table 5 Method C into the 10 mL volumetric.

NOTE: Addition of a small amount of methanol to the 10 mL volumetric flask prior to addition of each standard is OK but remember the total volume of standards used is 7 mL.

5.5.2.3 Make to volume with methanol, transfer to 2 mL GC vials, cap tightly, store frozen, and assign a one month expiration date.

5.6 Calibration Standards: Prepare calibration standards directly in the 10 mL Luer-lock syringe at eight levels immediately prior to loading them onto the purge and trap autosampler (See Step 7.7.3).

5.7 Surrogate

5.7.1 See Section 5.11, internal standard/surrogate solution

5.8 Laboratory Control Sample (LCS) Stock Solution

5.8.1 The LCS spike mix is also the intermediate standard mix detailed in Section 5.5.

5.9 MS/MSD Spike Mix

5.9.1 The MS/MSD spike mix is also the intermediate standard mix detailed in Section 5.5.

5.10 Second Source

5.10.1 Stock second source standard solutions: The second source stock standards are acquired from a different commercial vendor than that used for the initial calibration standard. This second source is Restek Corporation. A mega mix (Part#30633) contains the vast majority of the compounds at 2,000 µg/mL. The gases (Part#30042), and oxygenates (MTBE, di-isopropyl ether, Part#30465) are also acquired at 2000 µg/mL. The ketones (Part#30006) are acquired at 5,000 µg/mL. Table 4 provides a listing of components in each stock solution. A hexane stock is not available from Restek and therefore is not included in the second source standard.

5.10.2 Intermediate second source standard (5/25/50 µg/mL)

5.10.2.1 Similar to the intermediate calibration standard, the second source standard is prepared at 5 µg/mL for all targets, except the ketones (50 µg/mL) and tetrahydrofuran (25 µg/mL).

5.10.2.2 Fill a 50 mL volumetric flask ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that a syringe needle tip can be submerged.

5.10.2.2.1 Using a 250 µL Gastight syringe aliquot 125 µL of Mega Mix (36033) to the 50 mL volumetric.

5.10.2.2.2 Using a 500 µL Gastight syringe aliquot 500 µL of the ketone mix (30006) to the 50 mL volumetric.

5.10.2.2.3 Using a 250 µL Gastight syringe aliquot 125 µL of the six gases (30042) and 125 µL of the oxygenates (30465) into the 50 mL volumetric.

NOTE: Once vials are opened and aliquots taken, vials are discarded.

NOTE: See Table 5 as a reference for preparation of the second

source.

5.10.2.3 Make to volume with methanol, transfer to 2 mL GC vials and cap tightly.

5.10.2.4 Store frozen at all times and assign a one month expiration date.

5.11 Internal Standard/Surrogate

5.11.1 Stock internal standard and surrogate solutions: Stock solutions are acquired commercially as certified solutions in methanol from Restek. The internal standards (Part#30074) and surrogates (Part#30073) are purchased at 2,500 µg/mL. Table 4 provides a listing of the components in each stock solution.

5.11.2 Intermediate internal standard/surrogate solution

5.11.2.1 The working internal standard/surrogate solution is prepared at 25 µg/mL by diluting the stock standard solutions (Section 5.11.1) in a 50 mL volumetric flask. With a 2500 µg/mL stock internal or surrogate standard, 500 µL would be added to the flask. Table 5 summarizes preparation of this standard.

5.11.2.2 Fill a 50 mL volumetric ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that the syringe needle tip can be submerged below the level of the methanol.

5.11.2.3 Using a 500 µL Gastight syringe aliquot 500 µL of Internal standard Mix (#30074) and add to the 50 mL volumetric.

5.11.2.4 Using a 500 µL Gastight syringe aliquot 500 µL of Surrogate Mix (#30073) and add to the 50 mL volumetric.

NOTE: Discard unused ampoules once aliquot has been taken.

5.11.2.5 Make to volume with purge and trap methanol.

5.11.2.6 Once prepared, the working internal standard/surrogate solution is then portioned into 2 mL GC vials for daily use. Each vial is filled to its neck and capped.

5.11.2.7 Assign an expiration date of three months, enter information into LIMS system and store frozen

5.11.3 BFB tuning standard (50ug/ml)

5.11.3.1 The BFB tuning standard is prepared at 50 ug/ml by diluting the stock surrogate standard (section 5.11) (Restek part # 30073) in methanol.

5.11.3.2 Fill a 50 mL volumetric ¾ full of purge and trap grade methanol known to

be free of volatiles. Make sure the volumetric has enough methanol so that the syringe needle tip can be submerged below the level of the methanol.

5.11.3.3 Using a 1000 µL Gastight syringe aliquot 1000 µL of the Surrogate Mix (#30073) and add to the 50 mL volumetric.

NOTE: Discard unused ampoules once aliquot has been taken.

5.11.3.4 Make to volume with purge and trap methanol.

5.11.3.5 Once prepared, the BFB tuning standard is transferred to a 45 ml VOA vial.

5.11.3.6 Assign an expiration date of three months, enter information into LIMS system and store frozen

5.11.3.7 1ul of the BFB tuning standard is injected into the GC/MSD system for tune evaluation.

6 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sample Containers, Custody and Preservation

6.1.1 Containers are provided by the laboratory to the client prior to sampling whenever possible. Samples are collected by the field consultant and delivered to the laboratory with a chain of custody (COC). Upon receipt of samples, the sample custodian notes any deviations on the chain of custody.

6.1.2 If a COC is not received with the samples, ECCS personnel will fill out a COC per SOP "GEN-003, Chain-of-Custody, Log In, Tracking Procedures, and Sample Tracking".

6.2 Water Samples

6.2.1 Glass 40-mL VOA vials, containing 0.5 mL of 1:1 HCl with Teflon® lined septa are provided by ECCS for water samples.

6.2.1.1 Hydrochloric acid preserved VOA vials are purchased pre-made from a variety of vendors.

NOTE: HCl is used to eliminate/retard degradation of compounds by bacteria.

NOTE: Water samples are typically preserved with 1:1 HCl contained in the 40 mL VOA vial at the time of collection by the client.

NOTE: Client may use unpreserved VOA vials but hold time changes to 7 days.

6.2.2 Samples once taken in the field should immediately be stored on ice or refrigerated.

NOTE: A temperature blank may be used if temperature reporting is critical.

6.2.3 Once VOA vials are received at ECCS laboratories they are stored in a refrigerator at 4 °C.

6.2.4 Water samples preserved with 1:1 HCl have a hold time of 14 days. Water samples not preserved with 1:1 HCl have a hold time of 7 days.

6.3 Soil Samples

6.3.1 ECCS supplies a pre-weighed 40 mL VOA vial containing 10 mL of methanol for soil collection. In addition, ECCS supplies a Lock and Load T handle (Section 4.12) and disposable sample container for collection of a pre-measured approximately 10 g of soil.

6.3.2 The client is responsible to put about 10 g of soil into the VOA vial containing purge and trap methanol either by use of a Lock and Load or using a balance and weighting in the field.

6.3.3 An alternative approach is the client is supplied 2-oz sample jars with 25 mL of methanol and weighs 25 g into the jar.

6.3.4 Soil sample once in VOA vials/jars need to be placed in a cooler on ice for transport to a lab.

6.3.5 Soil samples received stored in a refrigerator at 4 °.

6.3.6 Methanol preserved soil samples have a hold time of 14 days from time of collection.

6.3.7 Soil samples not preserved in methanol are noted in the comments of the VOA extraction log and on the COC. These soil samples are extracted as soon as practical.

7 PROCEDURE

7.1 Water Samples

7.1.1 Screening water aamples: Water samples should be initially screened by one of the four following methods for potentially high levels of VOCs that might otherwise contaminate the purge and trap system.

7.1.1.1 Screening by purge and trap

7.1.1.1.1 A 100 fold dilution is a good starting point when using the purge and

trap to screen water samples.

7.1.1.1.2 Pull up 10 mL of clean water into the 10 mL Gastight syringe.

7.1.1.1.3 Using a 100 µL Gastight syringe, aliquot 100 µL of the unknown water sample and add to the 10 mL of clean water in the 10 mL syringe.

7.1.1.1.4 Proceed to Step 7.1.5.4 for spiking and analysis by purge and trap.

7.1.1.2 GC/ECD Screening: GC/ECD Screening: The rapid, GC/ECD screening method is applicable for chlorinated VOCs in soil and water. The chlorinated VOCs typically screened are the degreasing solvents – PCE, TCE, 1,1,1-TCA, and c-1,2-DCE. Chlorinated fumigants such as carbon tetrachloride, chloroform, and 1,2-dibromoethane may also be screened. The methanol extract from the soil or water sample is injected into the GC/ECD with an autosampler. The method uses the external standard approach with three levels of calibration standard.

GC/ECD Conditions:	
Column:	RTX-502.2, 60 m x 0.53 mm ID, 3 µm film
Injector Temp.:	225 °C
Detector Temp.:	300 °C
Column Flow:	10 mL/min
Head pressure:	15 PSI
Nitrogen Makeup:	40 mL/min
Split Flow:	150 mL/min
Septum Purge:	1.5 mL/min
Split Ratio:	15:1
Injection Volume:	Soils 0.5µL split Waters 6 µL split

Temperature Program:	
Initial Temperature:	85 °C
Initial Hold:	0.1 min
Initial Ramp:	22 °C/min
Final Temp.:	190 °C
Final Hold:	0 min
Oven Equilibration:	0.10 min
Cycle Time:	5.5 min

Stock Standards:	
Absolute Part #92950:	8 compounds chlorinated, 1000 µg/mL
Absolute Part #93305:	6 compounds (fumigants) 1000 µg/mL

Calibration Standards:

Soils:	0.5, 2.5, 25 µg/mL in methanol
Waters:	11, 48, 222 µg/L in VOA vial

NOTE: See Figure 5 for typical chromatograms.

7.1.1.3 GC/FID screening: GC/FID screening: The rapid, GC/FID screening method is applicable for BTEX in soil. Chlorinated VOCs may also be screened but they are not nearly as sensitive. The methanol extract from the soil is injected into the GC/FID system with an autosampler. The method uses the external standard approach calibrated with three levels of calibration standard.

GC/FID Conditions:	
Column:	RTX-502.2, 60 m x 0.53 mm ID, 3 µm film
Injector Temp.:	225 °C
Detector Temp.:	300 °C
Column Flow:	15 mL/min
Head pressure:	25 PSI
Column Makeup:	15 mL/min
Hydrogen Flow:	30 mL/min
Air Flow:	300 mL/min.
Split Flow:	15 mL/min
Septum Purge:	1.5 mL/min
Split Ratio:	1:1
Injection Volume:	3µL split

Temperature Program:	
Initial Temp.	100 °C
Initial Hold:	0 min
Initial Ramp:	22 °C/min
Final Temp.:	190C
Final Hold:	0 min
Oven Equilibration:	0.20 min
Cycle Time:	6.5 min

Stock Standards:	
Absolute Part # 20001	Hexadecane Extraction Volatiles (BTEX), 2,000ug/ml
Absolute Part # 92950	8 Compound Chlorinated Mix, 1000 µg/mL
Calibration Standards:	0.5, 2.5, 25 µg/mL in methanol

7.1.1.4 Direct inject GC/MS screening: The GC/MS screening method is applicable for chlorinated and aromatic VOCs in soil. The methanol extract from the

soil is injected into the GC/MS with an autosampler. The method uses the internal standard approach with seven levels of calibration standard.

GC/MS Conditions:	
Column:	RTX-624, 30 m x 0.32 mm ID, 1.8 µm film
Injector Temp.:	200 °C
Transfer Line Temp.:	280 °C
Column Flow:	1.5 mL/min
Head pressure:	2 PSI
Split Flow:	55 mL/min
Septum Purge:	1.5 mL/min
Split Ratio:	37:1
Injection Volume:	1 µL split
Temperature Program:	
Initial Temp.:	55 °C
Initial Hold:	1 min
Initial Ramp:	22 °C/min
Final Temp.:	225 °C
Final Hold:	2.5 min
Oven Equilibration:	0.50 min
Cycle Time:	20 min
Stock Standards:	
Absolute Part # 32001:	8260 Liquids, 2000 µg/mL
Restek Part #30074:	8260 Internal Standard Mix, 2500 µg/mL
Calibration Standards:	0.5, 1.0, 5.0, 10, 20, 50, 100 µg/mL in methanol

7.1.1.5 If no detects are encountered from the screening analysis the sample can be reanalyzed without any required dilution.

7.1.1.6 Based upon the screening analysis, the following table can be used as a guide to determine the final dilution required for a sample.

Screening Result	Dilution Factor	mL purged
< 0.5 µg/L	1	10
0.5 to 2 µg/L	5	2.0
2 to 5 µg/L	10	1.0
5 to 25 µg/L	50	0.200
25 to 100 µg/L	200	0.050
100 to 500 µg/L	1000	0.010

7.1.2 Rinsing/cleaning the 10 mL Luer Lock syringe between samples

7.1.2.1 Fill two specimen containers with reagent water. Label one container as

"rinse water" and label the other as "reagent water".

7.1.2.2 Fill a plastic squirt bottle with reagent water.

7.1.2.3 Rinse the tip of the 10 mL Luer lock syringe with the squirt bottle into a waste container.

7.1.2.4 From the specimen container labeled "rinse water," draw up 10 mL of rinse water with the Luer lock syringe and discard to a waste bucket.

7.1.2.5 Repeat the rinse two more times.

7.1.3 Water sample dilutions

7.1.3.1 Ideal dilutions should be prepared such that the highest concentration of any VOC measured in the diluted sample is in the 25 µg/L range.

7.1.3.2 Dilution between 2 and 10 fold

7.1.3.2.1 Aliquot between 1 mL and 5 mL and put in the 10 mL Luer Lock syringe.

7.1.3.2.2 Make to 10 mL in the syringe with clean reagent water.

7.1.3.3 Dilution greater than 10 fold

7.1.3.3.1 Fill the 10 mL Luer Lock syringe with clean reagent water.

7.1.3.3.2 Using appropriate size Gastight syringe add between 10 µL and 500 µL of unknown water.

7.1.3.3.3 Then add the unknown aliquot though the Luer tip to the reagent water contained in the 10 mL syringe.

7.1.4 Standard curve preparation

7.1.4.1 The volume of makeup methanol added to standards and samples can be varied depending upon project objectives; however, the volume of methanol must remain constant between calibration standards and the samples quantified with those same standards. The total volume of methanol can be 210, 110, or 30 µL depending upon project objectives. For example, if only waters are to be analyzed with a project, it is advantageous to reduce the total volume of methanol to 30 µL and thus increase the response and accuracy of the bromomethane and chloroethane analysis. On the other hand, if waters and low-level soils are to be analyzed simultaneously with a project, it is advantageous to use 210 µL of methanol and allow the same calibration method to be used for both waters and soils.

7.1.4.2 ICALs are prepared in 10 mL Luer lock syringes containing 10 mL of VOC

free water.

7.1.4.3 Method A (total methanol added - 210 µL)

7.1.4.3.1 ICAL (0.5 µg/L water curve) Using a 250 µL Gastight syringe add 199 µL of methanol through the Luer tip into the 10 mL of water to obtain a total of 210 µL methanol (all samples /ICALs contain a total volume of 210 µL of methanol). Using a 10 µL syringe add 10 µL of surrogate/IS (See Section 5.11.2) through the Luer tip into the 10 mL of water. Using a 10 µL syringe add 1.0 µL of spike mix (See Section 5.5) through the Luer tip into the 10 mL of water.

7.1.4.3.2 Immediately transfer the standard from its 10 mL syringe into the Tekmar ALS 2016 tube assigned to that ICAL level.

7.1.4.3.3 ICAL levels L-2 through L-8 follow the ICAL grid as listed in Table 6 Method A.

7.1.4.4 Method B (total methanol added - 110 µL)

7.1.4.4.1 ICAL (0.5 µg/L water curve) Using a 100 µL Gastight syringe add 89 µL of methanol through the Luer tip into the 10 mL of water in the 10 mL syringe to obtain a total methanol volume of 110 µL. Using a 10 µL syringe add 10 µL of Surrogate/IS (See Section 5.11.2) through the Luer tip in to the 10 mL of water. Using a 10 µL syringe add 1.0 µL of spike mix (See Section 5.5) through the Luer tip into the 10 mL of water.

7.1.4.4.2 Immediately transfer the standard into the Tekmar ALS 2016 tube assigned to that ICAL level.

7.1.4.4.3 ICAL Levels L-2 through L-8 follow the ICAL grid as listed in Table 6 Method B.

7.1.4.5 Method C (No additional methanol added)

7.1.4.5.1 Refer to Table 6 Method C for volumes used as techniques are the same as Method A+B except no additional methanol is added to the 10 mL water sample.

7.1.5 Sample Preparation

7.1.5.1 Withdraw 10 mL or an appropriate aliquot (based upon the screening analysis) of the water sample from the VOA vial into the 10 mL Luer lock syringe.

7.1.5.2 Tilt the VOA vial while withdrawing the sample to prevent inclusion of air bubbles.

7.1.5.3 Using a 250 µL syringe, either add no methanol, 100 µL methanol, or 200 µL of methanol through the Luer tip of the 10 mL syringe containing the unknown sample, depending on ICAL preparation method A, B, or C.

7.1.5.4 Then add 10 µL of the internal standard/surrogate solution (Section 5.11.2) with a 25 µL syringe.

7.1.5.5 Immediately transfer the sample from the 10 mL syringe into the Tekmar ALS 2016 purge tube assigned to that sample.

NOTE: See Section 7.3.1 for purge and trap loading information.

7.1.6 Preparation of blanks

7.1.6.1 Fill a 10 mL Luer lock syringe with 10 mL of clean water.

7.1.6.2 Add either no methanol/100 µL of methanol/or 200 µL of methanol depending on ICAL preparation method A, B, or C.

7.1.6.3 Add 10 µL of Surrogate/IS (Section 5.11.2) to the 10 mL of water.

7.1.6.4 Lock the syringe to the appropriate position on the Purge and Trap autosampler and transfer the Blank into the purge tube.

7.1.6.5 Frequency is 1 per Purge and Trap side/20 samples or less.

7.1.7 CCV/LCS analysis frequency

7.1.7.1 CCV/LCS are prepared the same.

7.1.7.2 Fill a 10 mL Luer lock syringe with 10 mL of clean water.

7.1.7.3 Add either no methanol/100 µL of methanol/or 200 µL depending on ICAL preparation method A, B, or C.

7.1.7.4 Add 10 µL of Surrogate/IS (Section 5.11.2) to the 10 mL of water.

7.1.7.5 Add 10 µL of the 5/25/50 µg/mL intermediate standard (See Section 5.5.1) to the 10 mL in the syringe.

7.1.7.6 Lock the syringe to the appropriate position on the Purge and Trap autosampler and transfer the CCV/LCS into the purge tube.

7.1.7.7 Frequency is 1 per purge and trap side/10 unknowns or tubes.

7.1.8 Matrix spike samples: Load a duplicate volume of sample into the 10 mL Luer lock syringe as described in Section 7.1.5. Add methanol per ICAL method A, B, or C, followed by 10 µL of the internal/standard solution (Section 5.11.2). To fortify the sample, add 10 µL of the 5/25/50 µg/mL intermediate standard

(Section 5.5) resulting in an addition of 5.0 µg/L. The ketones and tetrahydrofuran are spiked at 50 and 25 µg/L, respectively. Lock the syringe to the appropriate position on the purge and trap auto-sampler and transfer the sample into the purge tube. Repeat the process for the matrix spike duplicate sample.

7.1.9 Blank samples: Withdraw 10 mL reagent water into the 10 mL Luer lock syringe. Add methanol per ICAL method A, B, or C, followed by 10 µL of the internal standard/surrogate solution (Section 5.11.2).

7.2 Soil Samples

7.2.1 Soil blanks/LCS preparation

7.2.1.1 Blank: The blank for soil is prepared by weighing 10 grams of silica sand (Section 5.2) into a VOA vial and adding 10 mL of purge and trap methanol. The soil blank is sonicated and processed along with actual samples. 200 µL of the methanol extract from the soil blank is added to 10 mL of reagent water in the 10 mL syringe along with 10 µL of the internal standard/surrogate standard for purge and trap GC/MS analysis. The soil blank may be reused on another analysis day if stored in the sample refrigerator.

7.2.1.2 LCS: The laboratory control sample is prepared from silica sand and methanol in a VOA vial similar to the soil blank except the target compounds are also spiked into the methanol and 9.5 mL of methanol is added to the vial. The Intermediate standard solution (Section 5.5.2) is used to spike the target compounds into the LCS soil. 500 µL of the intermediate standard solution (Section 5.5.2) is added to the 9.5 mL of methanol and10 grams of sand in the vial. The LCS soil is then sonicated and processed along with actual samples. 200 µL of the methanol extract from the soil LCS is added to 10 mL of reagent water in the 10 mL syringe along with 10 µL of the internal standard/surrogate standard for purge and trap GC/MS analysis. It may be reused on another analysis day if stored in the sample refrigerator. Several LCS soils may be prepared in a day and stored in the sample refrigerator until used. It is suggested that several LCS soils be included whenever a new intermediate standard solution is prepared. The LCS soil expires at the same time as the intermediate standard solution used to prepare it.

7.2.2 Soil Weight

7.2.2.1 Client supplied soils in non-ECCS containers, preserved with methanol – use weight of soil and volume of methanol supplied with the sample.

7.2.2.2 Client supplied soil in a jar, Encore sampler

7.2.2.2.1 Accurately weigh about 10 g of soil into a 40 mL VOA vial, record

weight and add 10 mL of methanol.

7.2.2.3 VOA vials are supplied by ECCS with the Lock and Load supplies.

7.2.2.3.1 Each VOA vial contains 10 mL of methanol and the entire container was pre-weighed prior to shipment to the client. The total container weight to 0.XX g (Vial, label, and methanol) is recorded on the label with indelible marker.

7.2.2.3.2 Upon receipt at ECCS the vial is reweighted to 0.XX g. The weight is recorded on the COC and the information is entered into the laboratory information management system (LIMS) either by sample entry or by the chemist prior to analysis.

7.2.2.3.3 The soil weight obtained is the wet weight to be used for sample analysis.

7.2.3 Alternative soil weight/methanol volume evaluation

NOTE: The following evaluation is a necessary exercise for the state of Wisconsin, but is not to be used except in rare instances where the 1:1 ratio is mandated.

7.2.3.1 A true weight of soil in the jar is measured by determining the difference from the final weight of the jar minus the initial weight and minus the weight of 10 mL of methanol. 10 mL of methanol with a density of 0.7913 g/mL at 20 °C will contribute 7.91 grams to the final weight of the vial.

7.2.3.2 Methanol is added to samples with soil wet weights between 11 and 20 grams to achieve a 1:1 ratio of mL of methanol to g of wet soil. When adding methanol, it is measured with the 10 mL Luer lock syringe with an 18 gauge needle attached to the tip. The table below describes the action taken based upon the wet weight of soil preserved in the 40 mL VOA vial.

7.2.3.3 Evaluation Table

Target Soil Weight	Actual Weight	Initial Volume of Methanol	Action
10 g	<8 g	10 mL	Flag
10 g	8-11 g	10 mL	None
10 g	11-20 g	10 mL	Add Methanol
10 g	>20 g	10 mL	Reject

7.2.3.4 A paper record of the accurate determination of soil weight and addition of methanol is kept in the VOA Extraction Log.

7.2.4 Extraction by shaker table/sonication:

7.2.4.1 Position the ultrasonic bath on the shaker table platform so the long side of the bath is oriented in the same direction as the horizontal movement of the shaker table..

7.2.4.2 Place two foam VOA vial shipping blocks on each end the 45 ml VOA vial containing the methanol preserved soil samples. Place the blocks and VOA vials into the sonicator lengthwise so the VOA vials are positioned on their sides. Place enough water in the bath to cover the top vials but keep the water level below 2 inches from the top of the sonicator. The water in the bath needs to be clean, free of dust and debris, in order to effectively transfer energy in the bath. It may be necessary to place weight on top of the vials and blocks to keep them submerged. Plastic ice blocks can be used for this purpose.

7.2.4.3 Adjust the variable speed of the shaker table to provide maximum horizontal shaking without spilling water over the sides of the sonicator.

7.2.4.4 Sonicate the samples for 20 minutes.

NOTE: The sonicator bath needs to be kept cool either by using ice or changing the water.

NOTE: During shaking/sonication if clumps of soil do not break up, cool the sample and using mechanical means break up the clumps (*i.e.*, use stainless steel spatula).

7.2.4.5 Allow the samples to equilibrate for approximately 30 minutes after sonication prior to sampling.

7.2.5 Aliquoting extracts and archiving:

7.2.5.1 The volume of makeup methanol added to standards and samples can be varied depending upon project objectives; however, the volume of methanol must remain constant between calibration standards and the samples quantified with those same standards. The total volume of methanol can between 210, 110 and 30 µL depending upon project objectives. For example, if only waters are to be analyzed with a project, it is advantageous to reduce the total volume of methanol to 30 µL and thus increase the response and accuracy of the bromomethane and chloroethane analysis. On the other hand, if waters and low-level soils are to be analyzed simultaneously with a project, it is advantageous to use 210 µL of methanol and allow the same calibration method to be used for both waters and soils.

7.2.5.2 Allow the soil to settle from the upper layer of clear methanol extract - usually ½ hour. Alternatively, the vial may be centrifuged at a low speed (<1500 rpm) to separate the clay soils from the extract. If the vial is centrifuged, still allow ½ hour before opening the vial.

7.2.5.3 VOA vials containing soil extracts may be stored in a refrigerator or a portion may be transferred to a crimp top vial for storage in a freezer.

7.2.6 Screening soil extracts:

7.2.6.1 Transfer methanol extract to a GC vial with a 300 µL insert.

7.2.6.2 Sample is now ready to direct inject into GC/ECD, GC/FID, Direct inject GC/MS and/or dilution and inject on purge and trap system.

7.2.6.3 Refer to Step 7.1.1 for detailed screening methods.

7.2.6.4 Based upon results from the screening procedure, the following table is a guide that can be used to determine the dilution factor used for soil extracts by this purge and trap GC/MS procedure.

Screening Result	Dilution Factor	µL Extract Purged
< 2 µg/mL	1	200
2 to 10 µg/mL	5	40
10 to 25 µg/mL	20	10
25 to 50 µg/mL	50	4
50 to 200 µg/mL	100	2
> 200 µg/mL	perform initial dilution of extract in methanol	

7.2.7 Soil aliquot and analysis

7.2.7.1 Rinse the 10 mL Luer lock syringe as described in Section 7.1.2.

7.2.7.2 After syringe is cleaned, withdraw 10 mL of reagent water into the 10 mL Luer lock syringe.

7.2.7.3 Add 10 µL of the internal standard/surrogate solution (Section 5.11.2) directly into the reagent water in the 10 mL syringe.

7.2.7.4 Using a 250 µL Gastight syringe, remove 200 µL of the soil extract from the 40 mL soil sample VOA vial and add directly into the 10 mL syringe containing the IS/Surrogate and 10 mL reagent water.

7.2.7.5 In all cases, the total volume of methanol in the 10 mL of reagent water prior to loading into the purge and trap from sample extract and IS/surrogate volume is 210 µL.

7.2.7.6 If high levels of VOCs are encountered during the screening process than add the appropriate volume of soil extract as determined in Section 7.2.5 followed by addition of methanol so the total volume of methanol equals 210 µL.

7.2.7.7 Immediately transfer the 10 mL of reagent water containing the soil extract

in the 10 mL Luer lock syringe into the purge and trap.

7.2.7.8 Screw the Luer tip of the syringe to the Luer tip adapter fixed to the 3 way valve mounted to the appropriate position of the ALS 2016 autosampler.

7.2.7.9 Open the valve and slowly inject the sample into the labeled purge tube.

7.2.7.10 Close the valve before removing the syringe from the Leur tip adapter.

7.2.7.11 Repeat the process for all unknown samples to be analyzed.

NOTE: Make sure the samples have been screened by one of the techniques in the method prior to loading on the ALS 2016.

7.2.8 Matrix spike (MS), matrix spike duplicate (MSD) samples:

7.2.8.1 Select a soil sample extract for MS/MSD analysis.

NOTE: The client may supply triplicate soil aliquots for MS/MSD.

7.2.8.2 Prepare 10 mL syringe as in Section 7.2.7.

7.2.8.2.1 Load a duplicate (200 µL) volume of soil extract from the 40 mL VOA vial into the 10 mL Leur Lock syringe.

7.2.8.2.2 Add 10 µL of the internal standard/surrogate standard solution (See 5.11.2) directly into the 10 mL syringe through the Leur lock end.

7.2.8.2.3 To fortify the sample, add 10 µL of the 5/25/50 µg/mL intermediate standard (Section 5.5) directly into the 10 mL syringe through the Leur lock end.

NOTE: Resulting concentrations are 5/25/50 µg/L versus the universal water ICAL.

7.2.8.2.4 Repeat process 7.2.18.2.1 to 7.2.18.2.3 for the MSD.

7.2.9 LCS Spike

7.2.9.1 Prepare a 10 mL syringe in steps 7.2.7 and 7.2.8.

7.2.9.2 Add 200 µL of LCS sample (See Section 7.2.1.2) to the 10 mL syringe through the Luer Lock tip.

NOTE: Resulting concentration is 5, 25, 50 µg/L versus the universal water ICAL.

7.2.9.3 Add 10 µL of the internal standard/surrogate standard solution (See 5.11.2)

directly into the 10 mL syringe through the Luer Lock end.

7.2.10 Soil Blank Sample

7.2.10.1 Prepare a soil blank by weighing 10 g of heated silica sand into a 40 mL VOA vial with 10 mL of methanol (See Step 7.2.1.1).

7.2.10.2 Extract, vortex and sonicate the blank with the soil samples starting with Step 7.2.4. Analysis requires one blank/20 unknowns or less per day.

7.2.10.3 The blank is carried through the procedure as though it were a sample.

7.2.11 Continuing calibration verification (CCV) is required after every 10 unknown samples and at the end of every run..

7.2.11.1 CCVs are prepared the same as water CCVs (Section 7.1.7.1) except 200 µL of methanol is always added to the 10 mL of water in the syringe.

7.2.11.2 Purge and trap 2016 auto-sampler systems are operated with a Tekmar Duet system (Side A and Side B) as a 32 position autosampler. CCVs must be run on both sides.

7.3 Concentration and Transfer

7.3.1 Sample concentration and transfer is automatically performed during operation of the Tekmar ALS 2000 and 2016 purge and trap system prior to introduction into the GC/MS system.

7.4 Clean-Up and/or Derivatization: Not applicable to this method.

7.5 Instrument Conditions

7.5.1 GC/MSD pump down: Allow the GC/MS system to stabilize under high vacuum when starting from a vented state. Before analysis, allow at least 4 hours for the MS to stabilize after pump-down.

7.5.2 Purge and trap & GC/MS operating conditions

7.5.2.1 Purge and trap: Method "1"

Purge Flow:	Helium, 35 mL/min.
Purge Time:	11 minutes
Dry Purge:	2 minutes
Desorb:	1 minute at 250 °C
Bake Time:	8 minutes at 260 °C

7.5.2.2 GC conditions:

Column:	DB-624, 30 m x 0.32 mm, 1.8 µm film
Injector Temp:	200 °C
Transfer Line Temp:	280 °C
Column Head Pressure:	2 PSI
Column Flow:	1.7 mL/min
Split Flow:	140 mL/min
Split Ratio:	82:1

Oven Temperature Program	
Initial Temperature:	45 °C
Initial Hold:	4 minutes
Initial Ramp:	23 °C/ minute
Final Temperature:	240 °C
Final Hold:	2.5 minutes

7.5.2.3 MS conditions

Tune:	Maximum Sensitivity Autotune
EM Voltage:	2000 (200 above Autotune)
Scan Range:	45-260 amu
Scan Time:	2 scans/sec

7.6 Preventive Maintenance

7.6.1 Cleaning the purge and trap system: To minimize the possibility of carryover from occurring between analyses the purge and trap should be cleaned and programmed to run through a "clean cycle" prior to the analysis of actual samples, especially whenever high concentrations of VOCs, above 50 µg/L, have been previously run on the system. Cleaning the purge and trap involves swabbing the outside of the Luer lock threads and purge needle on the purge and trap with a clean "Kimwipe" moistened with reagent water. Reagent water is then used to rinse the 3-way valve and purge needle. The actual purge and trap "clean cycle" is a two step process. In the first step, no purge tube is attached and for 1.5 minutes excess water is blown out of the purge lines. This step uses Method 2 on the purge and trap. In the second step, a new purge tube is attached and for 5 minutes clean helium gas is purged through the system. This step uses Method 3 on the purge and trap. The tube is never removed after the second step and the sample is loaded to the tube through the 3-way valve. The very short, dummy GCMS acquisition is performed during these two steps so the duet box will switch and alternate between the two purge and trap systems. Table 8 summarizes the purge and trap and GCMS conditions used for this "clean cycle."

7.6.2 The tune report for the MSD serves as a useful diagnostic tool. It can be used to indicate when maintenance on the MSD is required.

7.6.2.1 Detection of leaks in the GC/MS system: The presence of a base peak at m/z 28 (N_2) that is higher than m/z 69 (base peak of PFTBA) in the tune report indicates the presence of a gross leak in the GC/MS vacuum system. A common source of the leak is a loose transfer line nut sealing the GC capillary column to the MSD transfer line. Tightening this nut often eliminates a leak.

7.6.2.2 Water in the GC/MS system: The presence of a base peak at m/z 18 (H_20) that is higher than m/z 69 in the tune report indicates excessive water remains in the MSD manifold. The MSD vacuum system does not efficiently remove water and this condition indicates that a longer equilibration time is needed prior to initial calibration.

7.6.2.3 Electron multiplier voltage: An electron multiplier voltage higher than 2700 in the tune report indicates that the multiplier needs replacement and/or the MSD source needs cleaning.

7.6.2.4 Peak shape/resolution of PFTBA calibration peaks: The appearance of the PFTBA peaks (m/z 69, 219, 502) used to calibrate the MSD should be symmetric without any shoulders. Isotope masses of these same peaks (m/z 70, 220, 503) should be present and indicated in the auto tune report. Non-symmetric peak shape or the non-detection of isotope masses usually indicates that the MSD source needs to be cleaned.

7.6.3 The response factors of system performance check compounds (SPCCs) calculated during the initial calibration also serve as useful diagnostic tool. SPCCs are used to check that purge and trap and GC conditions are appropriate and that compound degradation is acceptable. Chloromethane will be low if the purge flow is too fast while bromoform will be low if the flow is too slow. 1,1-DCA and especially 1,1,2,2-tetrachloroethane will be low due to degradation from active sites on contaminated transfer lines or valves in the purge and trap system. Clipping the front end of the GC column can be the first action taken to eliminate active sites. Flushing the lines and valves of the purge and trap system with 1:1 HNO_3: water, de-ionized water, and methanol may further remove the active sites. Finally, it is often necessary to totally replace contaminated lines or valves in the purge and trap system when active sites are encountered.

7.7 Purge and Trap GC/MS Calibration

7.7.1 MSD tuning verification: The GC/MS system is software-tuned by successfully completing a maximum sensitivity autotune. Perflurotributylamine (PFTBA) is the tuning compound used by the MSD during the maximum sensitivity autotune. A hardcopy report is generated from the tune (See Figure 1). See Section 7.6.2 for interpretation of the tune report. Further manual adjustments to the MSD optics settings may be also be necessary in order to successfully pass the following BFB tuning evaluation.

7.7.1.1 BFB tuning evaluation: Inject 1ul of the BFB tuning standard (Section 5.11.3) into the GC/MSD.

7.7.1.2 The mass spectra acquired from the BFB tuning standard must pass the acceptance criteria provided in Table 9

7.7.1.3 The BFB mass spectra used for tune evaluation may be a single scan or an average using any combination of the three individual MSD scans for BFB including the peak apex and the two scans to the right and/or left of the peak apex. Using a background subtracted BFB mass spectra is preferred using an appropriate background region adjacent to the BFB peak.

7.7.2 Calibration

A separate series of calibration standards must be analyzed on each of the two Tekmar 2016 autosamplers used with the Tekmar Duet purge and trap configuration. Similarly, separate quantitation methods are used to calculate VOC concentrations for samples analyzed on each of the two autosamplers.

7.7.3 Calibration standards

7.7.3.1 Prepare calibration standard as outlined in Section 7.1.4 for waters and 7.1.4.2 for soils.

7.7.3.2 Prepare a second source verification standard from the intermediate second source standard (Section 5.10.2) at 5 µg/L and analyze with each initial calibration on Side A and Side B of the Purge and Trap.

7.7.4 Accurate use of micro-syringes: Care must be exercised when making a measurement with a micro-syringe that air bubbles are not included and that the syringe barrel does not partially drop during handling thus ejecting a portion of the aliquot. Air bubbles can be eliminated from the syringe by lifting and pumping the syringe plunger quickly a few times prior to measuring the aliquot. Draw the aliquot up slowly for the final measurement. Do not invert the vial containing a standard while filling the syringe as excess standard will run down the outside needle of the syringe resulting in a high bias. When handling an aliquot contained in a syringe, hold the syringe in a manner that a finger is slightly pushing against the extended plunger of the syringe to prevent its movement.

7.7.5 Using the results of a few test analyses, adjust the electron multiplier voltage for the MSD tune file accordingly to achieve approximately 1.5 million area counts for the internal standard, 1,4-difluorobenzene, from its extracted ion profile. Analyze each of the calibration standards listed in Section 7.7.3, collect the data and tabulate the area response of the characteristic ions against the concentration for each target compound using internal standard method. Characteristic ions are listed in Table 3. The internal standard selected for the calculation of these ratios should be the internal standard that has a retention time closest to the compound being measured. The internal standard used for each target compound is provided

in Table 7. Calculate amount ratios and response ratios for each target compound and at each calibration level relative to the appropriate internal standard.

The amount ratio and response ratios are calculated as follows:

$$Amount\ Ratio = \frac{C(s)}{C(is)} \qquad Response\ Ratio = \frac{A(s)}{A(is)}$$

Where:

A(s) = Peak area of the target compound or surrogate
A(is) = Peak area of the internal standard
C(s) = Concentration of the target compound or surrogate in µg/L
C(is) = Concentration of the internal standard in µg/L

7.7.5.1 Calculation of response factors (RF) and percent relative standard deviation (% RSD): Calculation of response factors are used to evaluate the initial calibration against specific criteria listed in method 8260 and are also used to quantitate sample concentrations. Individual response factors are calculated by the following formula for each calibration level:

$$Response\ Factor\ (RF) = \frac{Response\ Ratio}{Amount\ Ratio}$$

The % RSD is calculated by the following formula:

$$\%\ RSD = \left(\frac{RF_{STDEV}}{RF_{AVE}}\right) \times 100$$

Where:
RF_{STDDEV} = Standard deviation of individual response factors
RF_{AVE} = Average of individual response factors

NOTE: Chemstation has a section to do this in the software for every ICAL generated. See Figure 4 for example.

7.7.5.2 Calibration check compounds (CCCs): The % RSD of the response factors must be < 30% for individual CCCs in order for the initial calibration to be valid. The CCCs are:

vinyl chloride	1,1-dichloroethene
chloroform,	1,2-dichloropropane
toluene	ethyl benzene

7.7.5.3 The % RSD for other target compounds must also be less than 30%, except for 1,2-dibromo-3-chloropropane and naphthalene which must be less than 40%. In addition, bromomethane, chloroethane, acetone, 2-butanone, tetrahydrofuran, methyl isobutyl ketone, and 2-hexanone are exempt from a % RSD requirement. These compounds must meet a linear calibration

criteria (Section 7.7.5.4). A data system report summarizing the % RSD of the initial calibration is provided in Figure 4.

7.7.5.3.1 If the correlation coefficient or % RSD does not meet the acceptance criteria for any compound then the integrity of the system is failing and corrective action is needed to eliminate a system leak and/or active sites (See Section 7.6). Visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites, or poor chromatographic behavior. Polar compounds, like acetone, purge poorly and will always exhibit an erratic response.

7.7.5.4 System performance check compounds (SPCCs) are monitored for a minimum average response factor (RF_{AVE}) from the initial calibration data. Figure 4 provides a data system report listing a summary of response factors. The SPCCs are:

chloromethane	1,1-dichloroethane
chlorobenzene	bromoform
1,1,2,2-tetrachloroethane	

7.7.5.5 The average response factor is determined by the data system from the mean of individual response factors by the formula:

$$RF_{AVE} = \frac{\sum\left(\frac{Response\ Ratio}{Amount\ Ratio}\right)}{N}$$

Where: N = Number of standards used in ICAL.

The minimum average response factors for SPCCs are:

chloromethane	0.10
1,1-dichloroethane	0.10
chlorobenzene	0.30
bromoform	0.10
1,1,2,2-tetrachloroethane	0.30

7.7.5.6 The average RF calculated from the calibration data must be greater than these minimum average response factors in order for the initial calibration to be valid.

7.7.5.7 SPCCs are used to check that purge and trap and GC conditions are appropriate and that compound degradation is acceptable. Chloromethane will be low if the purge flow is too fast while bromoform will be low if the flow is too slow. 1,1-Dichloroethane and especially 1,1,2,2-tetrachloroethane will be low due to degradation from active sites on contaminated transfer lines or valves in the purge and trap system.

NOTE: If system fails usually the first compound to fail is the 1,1,2,2-TCA. If failure occurs system may need to be cleaned, non use of specific port and/or at a minimum the 1,1,2,2-TCA data must be qualified. If problems persist the unit should be removed from service and maintenance performed as noted in other sections of this method.

7.7.5.8 Use of linear calibration curves: Linear calibration curves are used for select target compounds because they typically have high % RSDs. These compounds include:

bromomethane	chloroethane	acetone
2-butanone	tetrahydrofuran	methyl isobutyl ketone
2-hexanone		

7.7.5.8.1 Bromomethane and chloroethane exhibit erratic response due to methanol quenching of the MSD. The ketones and tetrahydrofuran exhibit erratic purging efficiencies. Plot the response ratio against the amount ratio for each of these target compounds. The curve fit is linear regression with the weight of the inverse of the concentration. The linear regression equation is:

$$Y = m \times x + b$$

Where:
Y = response ratio
m = slope
x = amount ratio
b = intercept
1/x = weighting factor

7.7.5.8.2 The correlation coefficient for these target compounds must be greater than 0.99.

7.7.6 Updating qualifier ion abundances: The relative responses of qualifier ions for target compounds are updated through the data system with each initial calibration. The 5 µg/L initial calibration standard is used for this purpose. Mass spectra of peaks found in samples are compared to these qualifier ion responses and the purity of the match is included in the report as a Q value. A Q value above 90 typically indicates the qualitative identification of a target compound in samples. These same qualifier ion ratios are included as a unique window in the QEdit data analysis menu and can further assist the mass spectrometrist during data review.

7.8 Retention time windows: The retention time window for extracted ion chromatograms in peak identification is 0.6 minutes wide. The GCMS software then identifies the peak in this window with the relative retention time closest to the actual

relative retention time determined from the authentic standard. The window must be this wide in order for the software's integration algorithm to have sufficient time to accurately calculate baselines as part of peak integration. The absolute retention time of target compounds should not vary by more than ± 0.05 minutes when compared to the authentic standard. However, retention times are used, only in part, to qualitatively identify compounds as in conventional GC analysis. Rather, this GC/MS method also utilizes the mass spectra of peaks for qualitative identification of target compounds.

7.9 Sample Analysis

7.9.1 Prior to sample analysis the purge and trap GC/MS system must be tuned and initially calibrated as described in Section 7.7 above. After calibrating the quantitation method, the same calibration standards are reprocessed by this calibrated method and reported. A second source standard (Section 7.7.3.2) is included with each initial calibration. It is common to perform the initial calibration in a sequence and data package that is separate from the actual samples. Without an initial calibration, the sequence would start with a CCV followed by a water and/or soil blank. A CCV must be analyzed on each of the two Tekmar 2016 autosamplers in the purge and trap Duet configuration. If the time of sample analysis is going to extend beyond 12 hours from the first CCV standard, a second CCV standard must be analyzed after the first 12 hours of analysis and later in the sequence. Matrix spike samples should be analyzed at the end of the sequence.

7.9.2 Continuing Calibration Verification:

7.9.2.1 The working calibration curve must be verified on each analysis day and once every 12 hours by running a CCV. The concentration of the CCV is 5 µg/L, except for the ketones (50 µg/L) and tetrahydrofuran (25 µg/L). The percent difference between the response factor for the CCCs in the CCV standard and the average response factor determined from the initial calibration data must not vary by more than 20% in order for the calibration to remain valid. If the response factor of any CCC varies by more than 20%, then a new calibration curve must be prepared and affected samples reanalyzed or the data must be appropriately qualified.

$$Percent\ Difference = \left| \frac{(RF_{AVE} - RF_{CCV})}{RF_{AVE}} \times 100 \right|$$

Where:
RF_{AVE} = average response factor from the initial calibration
RF_{CCV} = response factor for the CCV

7.9.2.2 The percent difference must not vary by more than 30% for other target compounds. Initiate corrective action only if a non CCC compound exceeds the 30% limit for any two sequential CCVs. Bromomethane, chloroethane,

acetone, 2-butanaone, MEK, tetrahydrofuran, 2-hexanaone and naphthalene are allowed a 40% percent difference.

In addition, the SPCCS in the CCV must meet their minimum response factor specified in Section 7.7.5.3 in order for the initial calibration to remain valid.

Figure 4 provides a data system report of the % difference between continuing calibration response factors and the initial calibration average response factors.

7.9.3 Qualitative analysis

7.9.3.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

7.9.3.2 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.9.3.3 The absolute retention time of the sample component is within ± 0.05 minutes of the absolute retention time of the standard component.

7.9.3.4 The relative intensities of the characteristic ions agree with 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20 % and 80 %.)

7.9.3.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.9.3.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with

shoulder(s) or a valley between two or more maxima), appropriate selection of analyte background spectra is important.

7.9.3.7 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

7.10 Calculations

Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the extracted ion current profile of the primary characteristic ion. The concentration in the sample analysis is determined using the average response factor as described in Section 7.7.5.1. Linear regression analysis is used for the select compounds described in Section 7.7.5.4.

7.10.1 Water Sample Calculations: Concentrations of VOCs in water are determined directly from the average response factor and multiplied by any dilution factor:

$$\mu g/L\ water\ sample = \mu g/L\ measured \times DF$$

Where: DF = Dilution factor, based on 10 mL of sample in the purge tube (e.g. If 10 mL of sample is used, the DF =1; likewise, if 2 mL of sample was used with 8 mL water, the DF = 5.)

7.10.2 Soil sample calculations: Concentrations of VOCs in soil are determined on a dry-weight basis by the following formula:

$$\mu g/kg\ \ soil\ sample = (\mu g/L\ measured \times 0.01L) \times \left(\frac{V_m/V_p}{W_s}\right) \div FS$$

Where:
V_m = Volume of methanol used to extract samples in mL, normally 10 mL
V_p = Volume of methanol extract purged in mL, normally 0.200 mL
W_s = Wet weight of soil extracted in kg, nominally 0.010 kg
FS = Fraction of dry solids of in the soil sample, approximately 0.85

7.10.3 Soil sample correction factor by LIMS: The correction factor for calculating soils from uploaded GC/MS data is established in the bench sheet by setting the initial sample volume at 10 mL and the final sample volume to 500 mL. This multiplier of 50 converts µg/L results from the uploaded GC/MS data to µg/kg results for methanol extracted soils

Where applicable, the concentration of any non-target analytes identified in the sample should be estimated by the same formula, except the areas A(x) and A(is) should be from the total ion chromatograms, and the response ratio for the compound

should be assumed to be 1. The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

8 QUALITY CONTROL

8.1 Quality control criteria are based upon information found in Methods 8260B and 5030B from EPA publication SW-846. Additional criteria may be found in Method 8000 when not specified in Method 8260B or 5030B.

8.2 The MSD must successfully complete both a maximum sensitivity autotune (Section 7.7.1 and Figure 1) and a BFB tune evaluation (Section 7.7.1.1).

8.3 Initial calibration must consist of at least 5 points from the available 7 calibration standards. The % RSD of response factors must be less than 30% for individual CCCs (See Section 7.7.5.2). The % RSD of the remaining target compound must be less than 30% except as noted in Section 7.5.5.2.

8.4 A mid-point standard (5 µg/L) is used for continuing calibration verification (CCV) and analyzed on each analysis day or every 12 hours. The percent difference between the response factors for the CCCs in the CCV standard must be within 20% of the average response factor calculated from the initial calibration (Section 7.9.1). The percent difference of the remaining target compounds in the CCV must not vary by more than 30% for each of any two sequential CCVs. The response factor determined for SPCCs in the CCV must exceed their minimum response factor (Section 7.7.5.3). If any CCC or SPCC in a CCV fails, then corrective action must be initiated. Corrective action may include, but not be limited to, reanalyzing the CCV (Section 7.9.1), preparing a new intermediate standard (Section 5.5), preparing a new calibration curve (Section 7.7), reanalyzing the affected samples or qualifying the data.

8.5 The response of the internal standards must not vary by < 50% or > 200% from the responses of the mid-point standard (5ug/L) used in during the initial 7 point calibration.

8.6 A method blank is prepared and analyzed on each analysis day and after every twenty samples. For soils, the method blank includes silica sand and is carried through all the steps of sample preparation and analysis. In the event that VOCs are measured in the blank, the results cannot be subtracted from sample results. The sample data must be qualified and the value measured in the blank reported with the samples.

8.7 MS/MSD samples are analyzed on each analysis day and after every twenty samples. Typical acceptable recoveries are 70-130%. Any indication of a potential matrix effect should be discussed with the client and the sample data is appropriately qualified.

8.8 Surrogate standards are added to all samples as system monitoring compounds for each analysis. Acceptable recoveries for surrogates are 70-130%. Samples that have

surrogate recoveries outside of control limits are reanalyzed or the sample data is qualified.

9 PERFORMANCE DATA

9.1 MDL have been determined for waters and soils from the analysis of seven replicates fortified at 0.50 µg/L and 25 µg/kg, respectively. The MDLs are presented in Table 1.

9.2 DOC were performed for waters by analyzing 4 replicate samples spiked at 5 µg/L. The DOC summary for waters is listed in Table 2. DOCs were also performed for soils at 250 µg/kg. The DOC summary for soils is provided in Table 2.

10 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

10.1 Contingencies for out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and filed with the Quality Manager. Analytical results shall be qualified as necessary.

11 WASTE MANAGEMENT / POLLUTION PREVENTION

11.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

12 REFERENCES

12.1 Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260B, SW-846, Test Methods for Evaluating Solid Wastes, Update III Revision 2, December 1996

12.1.1 Purge and Trap of Aqueous Samples, Method 5030B, SW-846, Test Methods for Evaluating Solid Wastes, Update III Revision 2, December 1996

12.1.2 Determinative Chromatographic Separations, Method 8000B, SW-846, Test Methods for Evaluating Solid wastes, Update III, Revision 2, December 1996

12.2 Analytical Detection Limit Guidance & Laboratory Guide for Determining Method Detection Limits, Wisconsin Department of Natural Resources Laboratory Certification Program, April 1996, PUBL-TS-056-96

TABLE 1
MDLS AND REPORTING LIMITS
WATER SAMPLES

Compound	MDL (µg/L)	Reporting Limit (µg/L)
Dichlorodifluromethane	0.092	0.50
Chloromethane	0.12	1.0
Vinyl Chloride	0.078	0.50
Bromomethane	2.2	5.0
Chloroethane	0.75	5.0
Trichlorofluoromethane	0.12	0.50
1,1,2-Trichlorotrifluoroethane	0.099	0.50
1,1-Dichloroethene	0.15	0.50
Acetone	7.7	20
Carbon Disulfide	0.073	0.50
Methylene Chloride	0.049	0.50
Methyl-t-butyl ether	0.10	0.50
trans-1,2-Dichloroethene	0.13	0.50
n-Hexane	0.095	0.50
1,1-Dichloroethane	0.15	0.50
Diisopropyl Ether	0.12	0.50
2,2-Dichloropropane	0.092	0.50
cis-1,2-Dichloroethene	0.15	0.50
2-Butanone (MEK)	6.6	20
Tetrahydrofuran	3.4	10
Bromochloromethane	0.12	0.50
Chloroform	0.11	0.50
1,1,1-Trichloroethane	0.085	0.50
Carbon Tetrachloride	0.087	0.50
1,1-Dichloropropene	0.10	0.50
Benzene	0.064	0.50
1,2-Dichloroethane	0.13	0.50
Trichloroethene	0.096	0.50
1,2-Dichloropropane	0.11	0.50
Dibromomethane	0.11	0.50
Bromodichloromethane	0.13	0.50
c-1,3-Dichloropropene	0.087	0.50
Methyl Isobutyl Ketone (MIBK)	6.0	20
Toluene	0.12	0.50
t-1,3-Dichloropropene	0.18	0.50
1,1,2-Trichloroethane	0.072	0.50
Tetrachloroethene	0.10	0.50
1,3-Dichloropropane	0.10	0.50

TABLE 1
MDLS AND REPORTING LIMITS
WATER SAMPLES

Compound	MDL (µg/L)	Report Limit (µg/L)
2-Hexanone	7.5	20
Dibromochloromethane	0.076	0.50
1,2-Dibromoethane	0.087	0.50
Chlorobenzene	0.071	0.50
1,1,1,2-Tetrachloroethane	0.14	0.50
Ethyl Benzene	0.10	0.50
m-+ p-Xylenes	0.10	0.50
o-Xylene	0.097	0.50
Styrene	0.075	0.50
Bromoform	0.12	0.50
Isopropylbenzene	0.066	0.50
1,1,2,2-Tetrachloroethane	0.092	0.50
Bromobenzene	0.092	0.50
1,2,3-Trichloropropane	0.17	1.0
n-Propylbenzene	0.083	0.50
2-Chlorotoluene	0.093	0.50
1,3,5-Trimethylbenzene	0.072	0.50
4-Chlorotoluene	0.068	0.50
t-Butylbenzene	0.098	0.50
1,2,4-Trimethylbenzene	0.096	0.50
s-Butylbenzene	0.066	0.50
1,3-Dichlorobenzene	0.081	0.50
p-Isopropyltoluene	0.082	0.50
1,4-Dichlorobenzene	0.061	0.50
n-Butylbenzene	0.071	0.50
1,2-Dichlorobenzene	0.087	0.50
1,2-Dibromo-3-chloropropane	0.19	0.50
1,2,4-Trichlorobenzene	0.076	1.0
Hexachlorobutadiene	0.12	1.0
Naphthalene	0.53	5.0
1,2,3-Trichlorobenzene	0.067	1.0

Waters analyzed in GC-1629 and GC-1636, March 2007

TABLE 1
MDL AND REPORTING LIMITS
SOIL SAMPLES

Compound	Soil MDL (µg/kg)	Soil Report Limit (µg/kg)
Dichlorodifluromethane	4.2	25
Chloromethane	8.4	25
Vinyl Chloride	5.0	25
Bromomethane	890	1000
Chloroethane	160	500
Trichlorofluoromethane	5.8	25
1,1,2-Trichlorotrifluoroethane	3.9	25
1,1-Dichloroethene	6.2	25
Acetone	1300	1500
Carbon Disulfide	3.4	25
Methylene Chloride	4.4	25
Methyl-t-butyl ether	9.6	25
trans-1,2-Dichloroethene	5.2	25
n-Hexane	4.4	25
1,1-Dichloroethane	2.4	25
Diisopropyl Ether	7.8	25
2,2-Dichloropropane	5.7	25
cis-1,2-Dichloroethene	3.9	25
2-Butanone (MEK)	1000	1500
Tetrahydrofuran	600	1500
Bromochloromethane	4.8	25
Chloroform	5.8	25
1,1,1-Trichloroethane	4.2	25
Carbon Tetrachloride	4.3	25
1,1-Dichloropropene	3.4	25
Benzene	2.5	25
1,2-Dichloroethane	6.0	25
Trichloroethene	4.6	25
1,2-Dichloropropane	5.0	25
Dibromomethane	11	25
Bromodichloromethane	4.8	25
c-1,3-Dichloropropene	4.8	25
Methyl Isobutyl Ketone (MIBK)	840	1500
Toluene	4.1	25
t-1,3-Dichloropropene	5.7	25
1,1,2-Trichloroethane	5.0	25
Tetrachloroethene	4.9	25
1,3-Dichloropropane	8.0	25

TABLE 1
MDLS AND REPORT LIMITS
SOIL SAMPLES

Compound	Soil MDL (µg/kg)	Soil Report Limit(µg/kg)
2-Hexanone	950	1500
Dibromochloromethane	6.0	25
1,2-Dibromoethane	6.0	25
Chlorobenzene	2.1	25
1,1,1,2-Tetrachloroethane	4.8	25
Ethyl Benzene	1.2	25
m-+ p-Xylenes	4.5	25
o-Xylene	3.6	25
Styrene	4.2	25
Bromoform	9.4	25
Isopropylbenzene	2.2	25
1,1,2,2-Tetrachloroethane	11	25
Bromobenzene	4.4	25
1,2,3-Trichloropropane	16	50
n-Propylbenzene	1.9	25
2-Chlorotoluene	2.8	25
1,3,5-Trimethylbenzene	3.3	25
4-Chlorotoluene	3.4	25
t-Butylbenzene	3.0	25
1,2,4-Trimethylbenzene	3.1	25
s-Butylbenzene	1.9	25
1,3-Dichlorobenzene	3.2	25
p-Isopropyltoluene	3.7	25
1,4-Dichlorobenzene	2.0	25
n-Butylbenzene	4.1	25
1,2-Dichlorobenzene	2.9	25
1,2-Dibromo-3-chloropropane	22	25
1,2,4-Trichlorobenzene	3.5	50
Hexachlorobutadiene	6.2	50
Naphthalene	13	250
1,2,3-Trichlorobenzene	5.0	50

Soils analyzed in GC-1637 and GC-1639, March 2007

TABLE 2
DEMONSTRATION OF CAPABILITY
WATER

Compound	Spike Amount (µg/L)	Mean Recovery	% RSD
Dichlorodifluromethane	5	102	3.74
Chloromethane	5	99	2.44
Vinyl Chloride	5	102	2.30
Bromomethane	5	165	13.8
Chloroethane	5	139	2.54
Trichlorofluoromethane	5	108	4.12
1,1,2-Trichlorotrifluoroethane	5	102	4.26
1,1-Dichloroethene	5	101	4.29
Acetone	50	92	8.46
Carbon Disulfide	5	99	2.17
Methylene Chloride	5	107	3.60
Methyl-t-butyl Ether	5	91	4.47
trans-1,2-Dichloroethene	5	101	2.52
n-Hexane	5	100	1.40
1,1-Dichloroethane	5	104	2.01
Diisopropyl Ether	5	96	4.72
2,2-Dichloropropane	5	90	3.19
cis-1,2-Dichloroethene	5	99	2.66
2-Butanone (MEK)	50	89	9.27
Tetrahydrofuran	25	84	7.25
Bromochloromethane	5	102	2.30
Chloroform	5	103	2.46
1,1,1-Trichloroethane	5	102	1.85
Carbon Tetrachloride	5	104	3.78
1,1-Dichloropropene	5	101	2.14
Benzene	5	101	3.27
1,2-Dichloroethane	5	104	3.00
Trichloroethene	5	100	0.18
1,2-Dichloropropane	5	98	1.55
Dibromomethane	5	96	1.74
Bromodichloromethane	5	99	1.78
c-1,3-Dichloropropene	5	92	2.77
Methyl Isobutyl Ketone (MIBK)	50	85	1.89
Toluene	5	97	1.15
t-1,3-Dichloropropene	5	93	1.56

TABLE 2
DEMONSTRATION OF CAPABILITY
WATER

Compound	Spike Amount (µg/L)	Mean Recovery	% RSD
1,1,2-Trichloroethane	5	97	4.04
Tetrachloroethene	5	103	2.08
1,3-Dichloropropane	5	97	3.45
2-Hexanone	50	84	5.02
Dibromochloromethane	5	97	2.91
1,2-Dibromoethane	5	94	3.16
Chlorobenzene	5	101	0.90
1,1,1,2-Tetrachloroethane	5	100	0.81
Ethyl Benzene	5	102	1.12
m-+ p-Xylenes	10	101	1.16
o-Xylene	5	98	0.37
Styrene	5	97	2.07
Bromoform	5	92	2.43
Isopropylbenzene	5	99	1.23
1,1,2,2-Tetrachloroethane	5	87	1.00
Bromobenzene	5	98	3.02
1,2,3-Trichloropropane	5	90	2.15
n-Propylbenzene	5	101	2.57
2-Chlorotoluene	5	101	2.00
1,3,5-Trimethylbenzene	5	99	1.81
4-Chlorotoluene	5	100	1.51
t-Butylbenzene	5	98	3.61
1,2,4-Trimethylbenzene	5	100	2.13
s-Butylbenzene	5	101	1.69
1,3-Dichlorobenzene	5	100	1.28
p-Isopropyltoluene	5	100	1.11
1,4-Dichlorobenzene	5	99	1.49
n-Butylbenzene	5	102	2.16
1,2-Dichlorobenzene	5	98	1.97
1,2-Dibromo-3-chloropropane	5	84	1.43
1,2,4-Trichlorobenzene	5	95	1.04
Hexachlorobutadiene	5	107	2.34
Naphthalene	5	78	3.96
1,2,3-Trichlorobenzene	5	94	2.21

Waters analyzed in GC-1626, March 2007

TABLE 2
DEMONSTRATION OF CAPABILITY
SOIL

Compound	Spike Amount (µg/kg)	Mean Recovery	% RSD
Dichlorodifluromethane	250	81	2.39
Chloromethane	250	95	2.73
Vinyl Chloride	250	93	3.11
Bromomethane	250	99	8.16
Chloroethane	250	90	10.1
Trichlorofluoromethane	250	102	4.35
1,1,2-Trichlorotrifluoroethane	250	100	1.62
1,1-Dichloroethene	250	98	2.43
Acetone	2500	104	8.17
Carbon Disulfide	250	96	2.32
Methylene Chloride	250	104	2.35
Methyl-t-butyl Ether	250	94	3.56
trans-1,2-Dichloroethene	250	99	2.79
n-Hexane	250	97	1.28
1,1-Dichloroethane	250	102	2.21
Diisopropyl Ether	250	95	1.02
2,2-Dichloropropane	250	97	2.96
cis-1,2-Dichloroethene	250	99	2.31
2-Butanone (MEK)	2500	99	7.45
Tetrahydrofuran	250	97	7.43
Bromochloromethane	250	101	1.67
Chloroform	250	101	3.12
1,1,1-Trichloroethane	250	101	1.41
Carbon Tetrachloride	250	105	2.93
1,1-Dichloropropene	250	102	2.06
Benzene	250	103	1.55
1,2-Dichloroethane	250	102	1.90
Trichloroethene	250	100	1.77
1,2-Dichloropropane	250	98	1.58
Dibromomethane	250	98	1.61
Bromodichloromethane	250	98	1.99
c-1,3-Dichloropropene	250	97	2.62
Methyl Isobutyl Ketone	2500	98	4.91
Toluene	250	95	1.90
t-1,3-Dichloropropene	250	97	2.41
1,1,2-Trichloroethane	250	98	2.96
Tetrachloroethene	250	101	1.41

TABLE 2
DEMONSTRATION OF CAPABILITY
SOIL

Compound	Spike Amount (µg/kg)	Mean Recovery	% RSD
1,3-Dichloropropane	250	101	1.38
2-Hexanone	2500	96	6.67
Dibromochloromethane	250	98	3.63
1,2-Dibromoethane	250	96	2.74
Chlorobenzene	250	99	0.41
1,1,1,2-Tetrachloroethane	250	98	1.77
Ethyl Benzene	250	100	0.79
m-+ p-Xylenes	500	100	1.90
o-Xylene	250	97	1.25
Styrene	250	98	0.76
Bromoform	250	98	4.53
Isopropylbenzene	250	99	1.19
1,1,2,2-Tetrachloroethane	250	91	2.66
Bromobenzene	250	95	1.16
1,2,3-Trichloropropane	250	94	1.65
n-Propylbenzene	250	97	1.36
2-Chlorotoluene	250	97	2.02
1,3,5-Trimethylbenzene	250	96	2.55
4-Chlorotoluene	250	99	0.66
t-Butylbenzene	250	95	1.91
1,2,4-Trimethylbenzene	250	96	1.87
s-Butylbenzene	250	98	1.61
1,3-Dichlorobenzene	250	97	1.79
p-Isopropyltoluene	250	96	1.00
1,4-Dichlorobenzene	250	97	0.97
n-Butylbenzene	500	97	1.46
1,2-Dichlorobenzene	250	97	1.51
1,2-Dibromo-3-chloropropane	250	93	7.69
1,2,4-Trichlorobenzene	250	94	2.17
Hexachlorobutadiene	250	99	1.41
Naphthalene	250	86	2.96
1,2,3-Trichlorobenzene	250	92	2.30

Soils analyzed in GC-1639, March 2007

TABLE 3
ABSOLUTE RETENTION TIME ORDER
RTX-624, 30 m x 0.32 mm, 1.8 µm

Compound	**Absolute RT (min)**
Dichlorodifluromethane	1.33
Chloromethane	1.47
Vinyl Chloride	1.55
Bromomethane	1.80
Chloroethane	1.90
Trichlorofluoromethane	2.11
1,1,2-Trichlorotrifluoroethane	2.55
1,1-Dichloroethene	2.59
Acetone	2.68
Carbon Disulfide	2.77
Methylene Chloride	3.03
Metyl-t-butyl Ether	3.19
trans-1,2-Dichloroethene	3.21
n-Hexane	3.36
1,1-Dichloroethane	3.58
Diisopropyl Ether	3.55
2,2-Dichloropropane	4.01
cis-1,2-Dichloroethene	4.03
2-Butanone (MEK)	4.04
Tetrahydrofuran	4.22
Bromochloromethane	4.22
Chloroform	4.25
Dibromofluoromethane (Surrogate)	4.38
1,1,1-Trichloroethane	4.38
Pentafluorobenzene (Internal Standard)	4.40
Carbon Tetrachloride	4.48
1,1-Dichloropropene	4.49
Benzene	4.65
1,2-Dichloroethane	4.72
1,4-Difluorobenzene (Internal Standard)	4.94
Trichloroethene	5.12
1,2-Dichloropropane	5.33
Dibromomethane	5.42
Bromodichloromethane	5.50
c-1,3-Dichloropropene	5.83
Methyl Isobutyl Ketone (MIBK)	5.92
Toluene-d8 (Surrogate)	6.00

TABLE 3
ABSOLUTE RETENTION TIME ORDER
RTX-624, 30 m x 0.32 mm, 1.8 µm

Compound	**Absolute RT (min)**
Toluene	6.05
t-1,3-Dichloropropene	6.25
1,1,2-Trichloroethane	6.39
Tetrachloroethene	6.43
1,3-Dichloropropane	6.52
2-Hexanone	6.53
Dibromochloromethane	6.67
1,2-Dibromoethane	6.78
Chlorobenzene-d5 (Internal Standard)	7.09
Chlorobenzene	7.11
1,1,1,2-Tetrachloroethane	7.16
Ethyl Benzene	7.15
m-+ p-Xylenes	7.24
o-Xylene	7.55
Styrene	7.56
Bromoform	7.74
Isopropylbenzene	7.80
4-Bromofluorobenzene (Surrogate)	7.96
1,1,2,2-Tetrachloroethane	8.07
Bromobenzene	8.09
1,2,3-Trichloropropane	8.12
n-Propylbenzene	8.11
2-Chlorotoluene	8.22
1,3,5-Trimethylbenzene	8.24
4-Chlorotoluene	8.30
t-Butylbenzene	8.48
1,2,4-Trimethylbenzene	8.53
s-Butylbenzene	8.65
1,3-Dichlorobenzene	8.79
p-Isopropyltoluene	8.75
1,4-Dichlorobenzene-d4 (Internal Std)	8.85
1,4-Dichlorobenzene	8.79
n-Butylbenzene	9.07
1,2-Dichlorobenzene	9.16
1,2-Dibromo-3-chloropropane	9.77
1,2,4-Trichlorobenzene	10.38
Hexachlorobutadiene	10.44
Naphthalene	10.58
1,2,3-Trichlorobenzene	10.77

TABLE 3
CHARACTERISTIC MASSES (m/z)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	--
Benzene	78	--
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromdichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone (MEK)	72	57
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Dibromochloromethane	129	127
1,2-Dibromo-3-chloro-propane	75	155, 157
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
Dichlorodifluromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	12
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77,

TABLE 3
CHARACTERISTIC MASSES (m/z)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
trans-1,3-Dichloropropene	75	77
Di-isopropylether	87	59,69,102
Ethylbenzene	91	106
Hexachlorobutadiene	225	223, 227
Hexachlorobutadiene	225	223, 227
Hexane	57	55, 86
2-Hexanone	58	100, 85
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Methylene Chloride	84	86, 49
Methyl-t-butylether (MTBE)	73	57
2-Pentanone, 4-methyl (MIBK)	58	100, 85
Naphthalene	128	-
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	72	71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,1,2-Trichlorotrifluoroethane	101	103, 151, 153
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl chloride	62	64
Xylenes	106	91

TABLE 3
CHARACTERISTIC MASSES (m/z)

Compound	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Internal Standards		
Chlorobenzene-d5	117	
1,4-Dichlorobenzene-d4	152	115, 150
1,4-Difluorobenzene	114	
Pentafluorobenzene	168	
Surrogate Standards		
Dibromofluoromethane	113	
Toluene-d8	98	
4-Bromofluorobenzene	95	174, 176

TABLE 4
INITIAL CALIBRATION STOCK STANDARD SOLUTIONS

8260B Liquids, Absolute Part #32001, 2000 µg/mL		
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1-Dichloroethane	1,1-Dichloroethene	1,1-Dichloropropene
1,2,3-Trichlorobenzen	1,2,3-Trichloropropane	1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane	1,2-Dibromoethane
1,2-Dichlorobenzene	1,2-Dichloroethane	1,2-Dichloropropane
1,3,5-Trimethylbenzene	1,3-Dichlorobenzene	1,3-Dichloropropane
1,4-Dichlorobenzene	2,2-Dichloropropane	2-Chlorotoluene
4-Chlorotoluene	Benzene	Bromobenzene
Bromochloromethane	Bromodichloromethane	Bromoform
c-1,3-Dichloropropene	Carbon Tetrachloride	Chloroform
cis-1,2-Dichloroethene	Dibromochloromethane	Dibromomethane
Ethyl Benzene	Hexachlorobutadiene	Isopropylbenzene
m-+ p-Xylenes	Methylene Chloride	Methyl-t-butyl Ether
Naphthalene	n-Butylbenzene	n-Propylbenzene
o-Xylene	p-Isopropyltoluene	s-Butylbenzene
Styrene	t-1,3-Dichloropropene	t-Butylbenzene
Tetrachloroethene	Toluene	trans-1,2-Dichloroethene
Trichloroethene		

8260B Gases, Absolute Part # 30058, 2000 µg/mL		
Bromomethane	Chloroethane	Chloromethane
Dichlorodifluoromethane	Trichlorofluoromethane	Vinyl Chloride

8260B Ketones, Absolute Part # 82402, 2000 µg/mL		
Acetone	2-Butanone	2-Hexanone
4-Methyl-2-Pentanone		

Single Component Solutions
Methyl-t-butyl Ether, Absolute Part # 70209, 1000 µg/mL
Carbon Disulfide, Absolute Part # 70060, 1000 µg/mL
Diisopropyl Ether, Absolute Part # 70987, 1000 µg/mL
n-Hexane, Absolute Part # 70962, 1000 µg/mL
Tetrahydrofuran, Absolute Part # 70380, 1000 µg/mL
1,1,2-Trichlorotrifluoroethane, Absolute Part # 70474, 1000 µg/mL

TABLE 4
SECOND SOURCE STOCK STANDARD SOLUTIONS

8260B Mega Mix Calibration Mix, Restek Part #30475, 2000 µg/mL		
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1,2-Trichlorotrifluoroethane	1,1-Dichloroethane	1,1-Dichloroethene
1,1-Dichloropropene	1,2,3-Trichlorobenzene	1,2,3-Trichloropropane
1,2,4-Trichlorobenzene	1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane
1,2-Dibromoethane	1,2-Dichlorobenzene	1,2-Dichloroethane
1,2-Dichloropropane	1,3,5-Trimethylbenzene	1,3-Dichlorobenzene
1,3-Dichloropropane	1,4-Dichlorobenzene	2,2-Dichloropropane
2-Chlorotoluene	4-Chlorotoluene	Benzene
Bromobenzene	Bromochloromethane	Bromodichloromethane
Bromoform	c-1,3-Dichloropropene	Carbon Disulfide
Carbon Tetrachloride	Chloroform	cis-1,2-Dichloroethene
Dibromochloromethane	Dibromomethane	Ethyl Benzene
Hexachlorobutadiene	Isopropylbenzene	m-+ p-Xylenes
Methylene Chloride	Methyl-t-butyl Ether	Naphthalene
n-Butylbenzene	n-Propylbenzene	o-Xylene
p-Isopropyltoluene	s-Butylbenzene	Styrene
t-1,3-Dichloropropene	t-Butylbenzene	Tetrachloroethene
Tetrahydrofuran	Toluene	trans-1,2-Dichloroethene
Trichloroethene		

502.2 Calibration Mix #1 (gases), Restek Part # 30042, 2000 µg/mL		
Bromomethane	Chloroethane	Chloromethane
Dichlorodifluoromethane	Trichlorofluoromethane	Vinyl Chloride

VOA Calibration Mix #1 (ketones), Restek Part # 30006, 5000 µg/mL		
Acetone	2-Butanone	2-Hexanone
4-Methyl-2-Pentanone		

California Oxygenates Mix, Restek Part # 30465, 2000 µg/mL	
Methyl tert-butyl ether	Diisopropyl Ether

TABLE 4
INTERNAL AND SURROGATE STOCK STANDARD SOLUTIONS

8260 Internal Standard Mix, Restek Part # 30074, 2500 µg/mL	
Chlorobenzene-d5	1,4-Dichlorobenzene-d4
1,4-Difluorobenzene	Pentafluorobenzene

8260 Surrogate Mix, Restek Part # 30073, 2500 µg/mL		
4-Bromofluorobenzene	Dibromofluoromethane	Toluene-d8

TABLE 5
INTERMEDIATE INITIAL CALIBRATION STANDARD SOLUTION PREPARATION
(5/25/50 µg/mL)

Description	Absolute Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
54 Liquids	32001	2000	125	50	5.0
6 Gases	30058	2000	125	50	5.0
4 Ketones	82402	2000	1250	50	50
MTBE	70209	1000	250	50	5.0
Carbon Disulfide	70060	1000	250	50	5.0
Diisopropyl ether	70987	1000	250	50	5.0
Hexane	70962	1000	250	50	5.0
Tetrahydrofuran	70380	1000	1250	50	25
1,1,2-Trichlorotrifluoroethane	70474	1000	250	50	5.0

NOTES: (1) Solvent Purge and trap methanol.
(2) Compounds may be added or deleted for project/site specific requirements.

TABLE 5
INTERMEDIATE INITIAL CALIBRATION STANDARD SOLUTION PREPARATION
(50/250/500 µg/mL)

Description	Absolute Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
54 Liquids	32001	2000	250	10	50
6 Gases	30058	2000	250	10	50
4 Ketones	82402	2000	2500	10	500
MTBE	70209	1000	500	10	50
Carbon Disulfide	70060	1000	500	10	50
Diisopropyl ether	70987	1000	500	10	50
Hexane	70962	1000	500	10	50
Tetrahydrofuran	70380	1000	2500	10	250
1,1,2-Trichlorotrifluoroethane	70474	1000	50500	10	50

NOTES: (1) Solvent Purge and trap methanol.
(2) Compounds may be added or deleted for project/site specific requirements.

TABLE 5
INTERMEDIATE SECOND SOURCE STANDARD SOLUTION PREPARATION

Description	Restek Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
Mega Mix	30633	2000	125	50	5.0
6 Gases	30042	2000	125	50	5.0
4 Ketones	30006	5000	500	50	50
Oxygenates	30465	2000	125	50	5.0

TABLE 5
INTERMEDIATE INTERNAL STANDARD/SURROGATE SOLUTION PREPARATION
(25 µg/mL)

Description	Restek Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
8260 Internal Standards	30074	2500	500	50	25
8260 Surrogate Standards	30073	2500	500	50	25

TABLE 6
INITIAL CALIBRATION STANDARDS
PREPARATION IN 10 mL OF REAGENT WATER (METHOD A)

Calibration Level	**L-1**	**L-2**	**L-3**	**L-4**	**L-5**	**L-6**	**L-7**	**L-8**
µg/L Final in 10 mL	0.5/2.5/5.0	1.0/5.0/10.0	2.0/10.0/20	3/15/30	5/25/50	10/50/100	25/125/2500	50/250/5000
Volume of Makeup Methanol (µL)	199	198	196	194	190	180	150	100
Volume of IS/Surr. Solution (µL)(5.11.2)	10	10	10	10	10	10	10	10
Volume of Standard Solution (µL) (5.5.1)	1.0	2.0	4.0	6.0	10	20	50	100

NOTE: Calculation Formula: µg/L

$$\frac{A \times B}{V} \times 1000\ ng/\mu g$$

Where:
A = Volume in µL added to 10 mL
B = Concentration of analyte in µg/mL
V = Volume in syringe (usually 10 mL) in L (0.01 L)

TABLE 6
INITIAL CALIBRATION STANDARDS
PREPARATION IN 10 mL OF REAGENT WATER (METHOD B)

Calibration Level	**L-1**	**L-2**	**L-3**	**L-4**	**L-5**	**L-6**	**L-7**	**L-8**
µg/L Final in 10 mL	0.5/2.5/5.0	1.0/5.0/10.0	2.0/10.0/20	3/15/30	5/25/50	10/50/100	25/125/2500	50/250/5000
Volume of Makeup Methanol (µL)	99	98	96	94	90	80	50	0
Volume of IS/Surr. Solution (µL)(5.11.2)	10	10	10	10	10	10	10	10
Volume of Standard Solution (µL) (5.5.1)	1.0	2.0	4.0	6.0	10	20	50	100

NOTE: Calculation Formula: µg/L

$$\frac{A \times B}{V} \times 1000\ ng/\mu g$$

Where:
A = Volume in µL added to 10 mL
B = Concentration of analyte in µg/mL
V = Volume in syringe (usually 10 mL) in L (0.01 L)

TABLE 6
INITIAL CALIBRATION CALIBRATION STANDARDS
PREPARATION IN 10 mL OF REAGENT WATER (METHOD C)

Calibration Level	**L-1**	**L-2**	**L-3**	**L-4**	**L-5**	**L-6**	**L-7**	**L-8**
µg/L Final in 10 mL	0.5/2.5/5.0	1.0/5.0/10.0	2.0/10.0/20	3/15/30	5/25/50	10/50/100	25/125/2500	50/250/5000
Volume of IS/Surr. Solution (µL)(5.11.2)	10	10	10	10	10	10	10	10
Volume of Standard Solution (µL) (5.5.1)	1.0	2.0	4.0	6.0	10	-	-	-
Volume of Standard Solution (µL) (5.5.2)	-	-	-	-	-	2	5	10

NOTE: Calculation Formula: µg/L

$$\frac{A \times B}{V} \times 1000\ ng/\mu g$$

Where:
A = Volume in µL added to 10 mL
B = Concentration of analyte in µg/mL
V = Volume in syringe (usually 10 mL) in L (0.01 L)

TABLE 7
INTERNAL STANDARDS AND CORRESPONDING TARGET COMPOUNDS

Pentafluorobenzene		
Dichlorodifluromethane	Chloromethane	Vinyl Chloride
Bromomethane	Chloroethane	Trichlorofluoromethane
1,1,2-Trichlorotrifluoroethane	1,1-Dichloroethene	Acetone
Carbon Disulfide	Methylene Chloride	Methyl-t-butyl Ether
trans-1,2-Dichloroethene	n-Hexane	1,1-Dichloroethane
Diisopropyl Ether	2,2-Dichloropropane	cis-1,2-Dichloroethene
2-Butanone (MEK)	Tetrahydrofuran	Bromochloromethane
Chloroform	Dibromofluoromethane (Surrogate)	1,1,1-Trichloroethane
Carbon Tetrachloride	1,1-Dichloropropene	Benzene
1,2-Dichloroethane		

1,4-Difluorobenzene		
Trichloroethene	1,2-Dichloropropane	Dibromomethane
Bromodichloromethane	c-1,3-Dichloropropene	Methyl Isobutyl Ketone (MIBK)
Toluene-d8 (Surrogate)	Toluene	t-1,3-Dichloropropene
1,1,2-Trichloroethane		

Chlorobenzene-d5		
Tetrachloroethene	1,3-Dichloropropane	2-Hexanone
Dibromochloromethane	1,2-Dibromoethane	Ethyl Benzene
m-+ p-Xylenes	o-Xylene	Styrene
Bromoform	Isopropylbenzene	Bromofluorobenzene (Surrogate)

1,4-Dichlorobenzene-d4		
1,1,2,2-Tetrachloroethane	Bromobenzene	1,2,3-Trichloropropane
n-Propylbenzene	2-Chlorotoluene	1,3,5-Trimethylbenzene
4-Chlorotoluene	t-Butylbenzene	1,2,4-Trimethylbenzene
s-Butylbenzene	1,3-Dichlorobenzene	p-Isopropyltoluene
1,4-Dichlorobenzene	n-Butylbenzene	1,2-Dichlorobenzene
1,2-Dibromo-3-chloropropane	1,2,4-Trichlorobenzene	Hexachlorobutadiene
Naphthalene	1,2,3-Trichlorobenzene	

TABLE 8
CONDITIONS FOR THE PURGE AND TRAP "CLEAN CYCLE"

Step 1, No Purge Tube Attached	
Purge and Trap:	Method "2"
Purge Flow:	Helium, 35 mL/min.
Purge Time:	1.5 minutes
Dry Purge:	0 minutes
Desorb:	0.2 minutes at 0 °C
Bake Time:	0 minutes at 0 °C

GC/MS Conditions: Method and Sequence "Purge"	
Oven Temperature Program	
Initial Temperature:	45 °C
Initial Hold:	0.5 minutes
EM Voltage:	1800

Step 2, Purge Tube Attached	
Purge and Trap:	Method "3"
Purge Flow:	Helium, 35 mL/min.
Purge Time:	5.0 minutes
Dry Purge:	0 minutes
Desorb:	0.2 minutes at 0 °C
Bake Time:	0 minutes at 0 °C

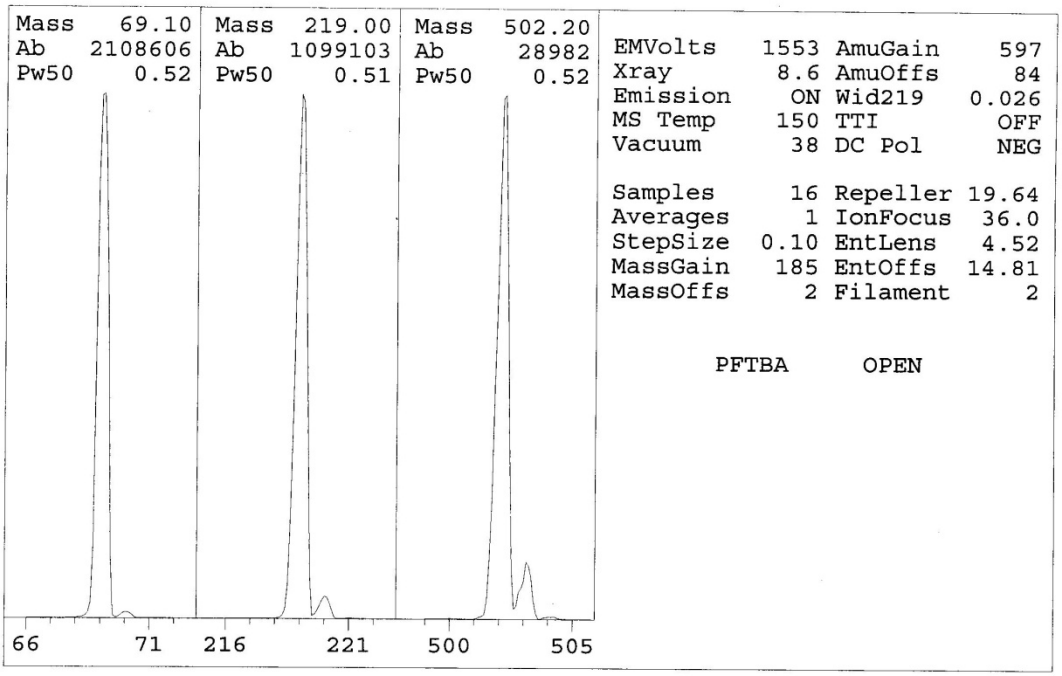
GC/MS Conditions: Method and Sequence "Purge"	
Oven Temperature Program	
Initial Temperature:	45 °C
Initial Hold:	0.5 minutes
EM Voltage:	1800

TABLE 9
BFB MASS INTENSITY CRITERIA

m/z	Required Intensity (Relative Abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base Peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Greater than 50% of m/z 95
174	5 to 9% of m/z 174
175	Greater than 95% but less than 101% of
176	m/z 174
177	5 to 9% of m/z 176

FIGURE 1
MAXIMUM SENSITIVITY AUTOTUNE REPORT

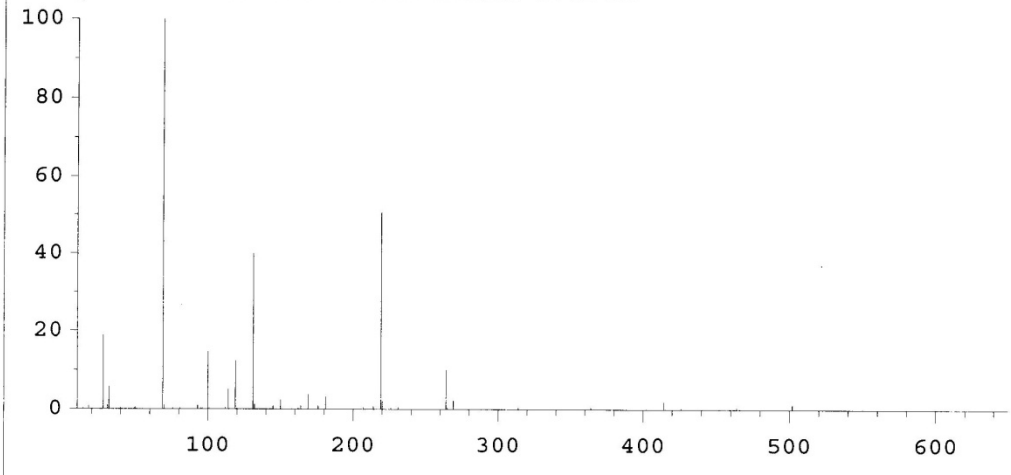
HP5971 Maximum Sensitivity Autotune
Instrument: GC/MS Instrument #1
Tue Feb 12 12:26:47 2008 D:\HPCHEM\1\5971\ATUNE.U



EMVolts	1553	AmuGain	597
Xray	8.6	AmuOffs	84
Emission	ON	Wid219	0.026
MS Temp	150	TTI	OFF
Vacuum	38	DC Pol	NEG
Samples	16	Repeller	19.64
Averages	1	IonFocus	36.0
StepSize	0.10	EntLens	4.52
MassGain	185	EntOffs	14.81
MassOffs	2	Filament	2

PFTBA OPEN

Scan: 10.00 - 650.00 Samples: 16 Thresh: 150
227 peaks Base: 69.00 Abundance: 1781248



Mass	Abund	Rel Abund	Iso Mass	Iso Abund	Iso Ratio
69.00	1781248	100.00	70.00	19560	1.10
218.95	904128	50.76	219.95	39224	4.34
502.05	23968	1.35	503.05	2416	10.08

FIGURE 2
TOTAL ION CHROMATOGRAM OF A 5 µg/L CCV AND SUMMARY REPORT

Quantitation Report (Not Reviewed)

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Feb 13 13:29 2008 Quant Results File: VOCA0212.RES

Quant Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration
DataAcq Meth : 8260

CPS 2/13/08

Internal Standards	R.T.	QIon	Response	Conc	Units		Dev(Min)
1) Pentafluorobenzene	4.53	168	1646282	25.00	ug/l		0.00
30) 1,4-Difluorobenzene	5.08	114	2428392	25.00	ug/l		0.00
40) Chlorobenzene-D5	7.24	117	2190468	25.00	ug/l		0.00
56) 1,4-Dichlorobenzene-D4	8.99	152	1125532	25.00	ug/l		0.00
System Monitoring Compounds							
24) Dibromofluoromethane	4.52	113	738831	22.01	ug/l		0.00
Spiked Amount 25.000			Recovery = 88.04%				
37) Toluene-D8	6.14	98	2578616	25.03	ug/l		0.00
Spiked Amount 25.000			Recovery = 100.12%				
55) Bromofluorobenzene	8.11	95	881882	24.04	ug/l		0.00
Spiked Amount 25.000			Recovery = 96.16%				
Target Compounds							Qvalue
2) Dichlorodifluoromethane	1.42	85	133423	4.94	ug/l		99
3) Chloromethane	1.58	50	90602	4.93	ug/l		89
4) Vinyl Chloride	1.66	62	77674	4.60	ug/l		97
5) Bromomethane	1.91	96	9209	3.82	ug/l	#	13
6) Chloroethane	1.99	64	55564	6.36	ug/l		95
7) Trichlorofluoromethane	2.21	101	117100	3.32	ug/l	#	93
8) 1,1,2-Trichlorotrifluoroet	2.66	101	143241	4.79	ug/L		96
9) 1,1-Dichloroethene	2.70	96	135926	4.99	ug/l		93
10) Acetone	2.80	58	44047	46.14	ug/L		100
11) Carbon Disulfide	2.89	76	461673	4.85	ug/L		99
12) Methylene Chloride	3.16	84	129724	4.90	ug/l		97
13) Methyl-t-butyl Ether	3.31	73	151105	4.10	ug/L		97
14) t-1,2-Dichloroethene	3.34	96	137303	4.49	ug/l		92
15) n-Hexane	3.48	57	169714	5.19	ug/L		100
16) 1,1-Dichloroethane	3.72	63	197595	4.66	ug/l		99
17) Diisopropyl Ether	3.67	87	88123	4.97	ug/L		96
18) 2.2- Dichloropropane	4.14	77	88433	3.14	ug/l		96
19) c-1,2-Dichloroethene	4.16	96	155263	4.58	ug/l		97
20) 2-Butanone(MEK)	4.18	72	64360	45.48	ug/l		99
21) Tetrahydrofuran	4.35	72	36739	23.98	ug/L		99
22) Bromochloromethane	4.36	128	73342	4.21	ug/l		97
23) Chloroform	4.39	83	207405	4.63	ug/l		97
25) 1,1,1-Trichloroethane	4.52	97	165855	4.43	ug/l		99
26) Carbon tetrachloride	4.60	117	150110	4.49	ug/l		95
27) 1.1-Dichloropropene	4.63	75	175512	4.87	ug/l		99
28) Benzene	4.79	78	522438	4.83	ug/l		99
29) 1,2-Dichloroethane	4.86	62	89431	4.54	ug/l		95
31) Trichloroethene	5.25	95	149765	4.76	ug/l		99

(#) = qualifier out of range (m) = manual integration
004A.D VOCA0212.M Wed Feb 13 13:29:44 2008 Page 1

FIGURE 2
TOTAL ION CHROMATOGRAM OF A 5 µg/L CCV AND SUMMARY REPORT

Quantitation Report (Not Reviewed)

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Feb 13 13:29 2008 Quant Results File: VOCA0212.RES

Quant Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration
DataAcq Meth : 8260

	Compound	R.T.	QIon	Response	Conc	Unit		Qvalue
32)	1,2-Dichloropropane	5.47	63	122527	4.95	ug/l		96
33)	Dibromomethane	5.56	93	76899	4.57	ug/l		96
34)	Bromodichloromethane	5.65	83	152464	4.72	ug/l		100
35)	c-1,3-Dichloropropene	5.97	75	181341	4.86	ug/l		99
36)	Methyl Isobutyl Ketone (MI	6.07	58	235719	51.79	ug/l		98
38)	Toluene	6.19	92	387541	5.13	ug/l		96
39)	1,1,2-Trichloroethane	6.53	83	83438	4.72	ug/l		98
41)	t-1,3-Dichloropropene	6.39	75	127746	4.69	ug/l	#	89
42)	Tetrachloroethene	6.57	164	155189	5.04	ug/l		96
43)	1,3-Dichloropropane	6.67	76	160881	4.92	ug/l		96
44)	2-Hexanone	6.67	58	222153	53.74	ug/l	#	95
45)	Dibromochloromethane	6.82	129	127664	4.65	ug/l		96
46)	1,2-Dibromoethane	6.93	107	110160	4.78	ug/l	#	98
47)	Chlorobenzene	7.26	112	439888	5.17	ug/l		98
48)	1,1,1,2-Tetrachloroethane	7.32	131	141899	4.92	ug/l		97
49)	Ethylbenzene	7.30	91	682203	5.14	ug/l		98
50)	m+p-Xylene	7.38	106	571758	10.35	ug/l		96
51)	o-Xylene	7.70	106	275747	5.18	ug/l		99
52)	Styrene	7.71	104	449245	5.19	ug/l		99
53)	Bromoform	7.90	173	83417	4.66	ug/l	#	98
54)	Isopropylbenzene	7.94	105	611509	5.28	ug/l		100
57)	1,1,2,2-Tetrachloroethane	8.22	83	105229	4.98	ug/l		94
58)	Bromobenzene	8.25	156	189176	5.01	ug/l		98
59)	1,2,3-Trichloropropane	8.28	75	82167	4.94	ug/l		86
60)	n-Propyl benzene	8.26	91	811071	5.32	ug/l		99
61)	2-Chlorotoluene	8.37	91	501878	5.13	ug/l		97
62)	1,3,5-Trimethylbenzene	8.39	105	524641	5.18	ug/l		97
63)	4-Chlorotoluene	8.45	91	450035	5.19	ug/l		100
64)	t-Butyl benzene	8.63	119	516364	5.46	ug/l		95
65)	1,2,4-Trimethylbenzene	8.68	105	535928	5.19	ug/l		99
66)	sec-Butyl benzene	8.80	105	792268	5.33	ug/l		98
67)	1,3-Dichlorobenzene	8.95	146	359797	5.11	ug/l		99
68)	p-Isopropyl toluene	8.90	119	643468	5.31	ug/l		99
69)	1,4-Dichlorobenzene	9.02	146	347429	5.05	ug/l		96
70)	n-Butyl benzene	9.21	91	603802	5.33	ug/l		98
71)	1,2-Dichlorobenzene	9.31	146	307081	5.14	ug/l		99
72)	1,2-Dibromo-3-chloropropan	9.92	157	20317	4.76	ug/l		98
73)	1,2,4-Trichlorobenzene	10.52	180	231459	5.23	ug/l		96
74)	Hexachlorobutadiene	10.59	225	137900	5.45	ug/l		95
75)	Naphthalene	10.74	128	313529	4.72	ug/l	#	97
76)	1,2,3-Trichlorobenzene	10.93	180	180977	4.83	ug/l		98

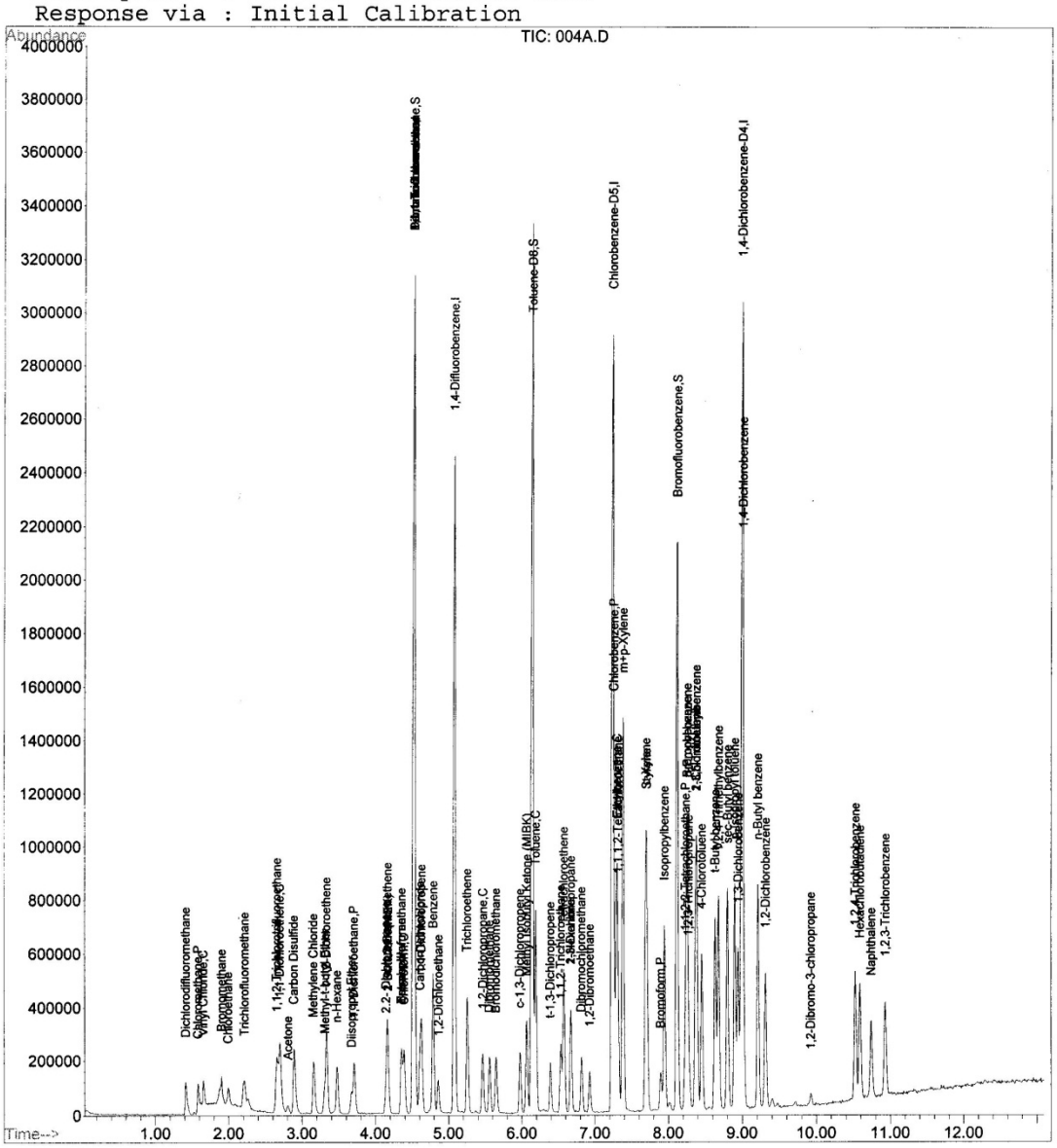
(#) = qualifier out of range (m) = manual integration
004A.D VOCA0212.M Wed Feb 13 13:29:44 2008 Page 2

FIGURE 2
TOTAL ION CHROMATOGRAM OF A 5 µg/L CCV AND SUMMARY REPORT

Quantitation Report

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Feb 13 13:29 2008 Quant Results File: VOCA0212.RES

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration



004A.D VOCA0212.M Wed Feb 13 13:29:45 2008 Page 3

FIGURE 3
DATA SYSTEM SUMMARY OF % DIFFERENCE AND RESPONSE FACTOR FOR CONTINUING CALIBRATION VERIFICATION

Evaluate Continuing Calibration Report

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Multiple Level Calibration

pass CPS 2/13/08

Min. RRF : 0.000 Min. Rel. Area : 50% Max. R.T. Dev 0.06min
Max. RRF Dev : 20% Max. Rel. Area : 150%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
1	I	Pentafluorobenzene	1.000	1.000	0.0	113	0.00
2		Dichlorodifluoromethane	0.410	0.405	1.2	112	0.00
3	P	Chloromethane	0.279	0.275	1.4	111	0.02
4	C	Vinyl Chloride	0.257	0.236	8.2	113	0.00
5		Bromomethane	0.037	0.028	24.3#	102	0.00
6		Chloroethane	0.138	0.169	-22.5#	120	0.01
7		Trichlorofluoromethane	0.536	0.356	33.6#	76	0.00
8		1,1,2-Trichlorotrifluoroeth	0.454	0.435	4.2	111	0.00
9	C	1,1-Dichloroethene	0.413	0.413	0.0	115	0.00
10		Acetone	0.015	0.013	13.3	112	0.00
11		Carbon Disulfide	1.445	1.402	3.0	112	0.00
12		Methylene Chloride	0.406	0.394	3.0	112	0.00
13		Methyl-t-butyl Ether	0.560	0.459	18.0	92	0.00
14		t-1,2-Dichloroethene	0.464	0.417	10.1	101	0.00
15		n-Hexane	0.497	0.515	-3.6	120	0.02
16	P	1,1-Dichloroethane	0.644	0.600	6.8	107	0.02
17		Diisopropyl Ether	0.269	0.268	0.4	114	0.00
18		2.2- Dichloropropane	0.427	0.269	37.0#	71	0.00
19		c-1,2-Dichloroethene	0.514	0.472	8.2	102	0.00
20		2-Butanone(MEK)	0.022	0.020	9.1	103	0.00
21		Tetrahydrofuran	0.023	0.022	4.3	108	0.00
22		Bromochloromethane	0.265	0.223	15.8	98	0.00
23	C	Chloroform	0.681	0.630	7.5	106	0.00
24	S	Dibromofluoromethane	0.510	0.449	12.0	101	0.00
25		1,1,1-Trichloroethane	0.568	0.504	11.3	98	0.02
26		Carbon tetrachloride	0.507	0.456	10.1	102	0.00
27		1.1-Dichloropropene	0.547	0.533	2.6	110	0.00
28		Benzene	1.644	1.587	3.5	108	0.00
29		1,2-Dichloroethane	0.299	0.272	9.0	99	0.02
30	I	1,4-Difluorobenzene	1.000	1.000	0.0	111	0.00
31		Trichloroethene	0.324	0.308	4.9	107	0.00
32	C	1,2-Dichloropropane	0.255	0.252	1.2	113	0.00
33		Dibromomethane	0.173	0.158	8.7	100	0.00
34		Bromodichloromethane	0.333	0.314	5.7	104	0.00
35		c-1,3-Dichloropropene	0.384	0.373	2.9	110	0.00
36		Methyl Isobutyl Ketone (MIB	0.046	0.049	-6.5	119	0.00
37	S	Toluene-D8	1.060	1.062	-0.2	112	0.00
38	C	Toluene	0.805	0.798	0.9	114	0.00
39		1,1,2-Trichloroethane	0.182	0.172	5.5	107	0.00

(#) = Out of Range
004A.D VOCA0212.M Thu Feb 14 13:00:30 2008 Page 1

FIGURE 3
DATA SYSTEM SUMMARY OF % DIFFERENCE AND RESPONSE FACTOR FOR CONTINUING CALIBRATION VERIFICATION

Evaluate Continuing Calibration Report

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Multiple Level Calibration

Min. RRF : 0.000 Min. Rel. Area : 50% Max. R.T. Dev 0.06min
Max. RRF Dev : 20% Max. Rel. Area : 150%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
41		t-1,3-Dichloropropene	0.311	0.292	6.1	99	0.00
42		Tetrachloroethene	0.351	0.354	-0.9	109	0.00
43		1,3-Dichloropropane	0.373	0.367	1.6	107	0.00
44		2-Hexanone	0.046	0.051	-10.9	122	0.00
45		Dibromochloromethane	0.313	0.291	7.0	99	0.00
46		1,2-Dibromoethane	0.263	0.251	4.6	108	0.00
47	P	Chlorobenzene	0.971	1.004	-3.4	114	0.00
48		1,1,1,2-Tetrachloroethane	0.329	0.324	1.5	113	0.00
49	C	Ethylbenzene	1.514	1.557	-2.8	114	0.00
50		m+p-Xylene	0.631	0.653	-3.5	112	0.00
51		o-Xylene	0.607	0.629	-3.6	114	0.00
52		Styrene	0.988	1.025	-3.7	115	0.00
53	P	Bromoform	0.205	0.190	7.3	100	0.00
54		Isopropylbenzene	1.323	1.396	-5.5	117	0.00
55	S	Bromofluorobenzene	0.419	0.403	3.8	108	0.00
56	I	1,4-Dichlorobenzene-D4	1.000	1.000	0.0	110	0.00
57	P	1,1,2,2-Tetrachloroethane	0.469	0.467	0.4	111	0.00
58		Bromobenzene	0.838	0.840	-0.2	114	0.00
59		1,2,3-Trichloropropane	0.370	0.365	1.4	131	0.00
60		n-Propyl benzene	3.387	3.603	-6.4	114	0.00
61		2-Chlorotoluene	2.174	2.230	-2.6	113	0.00
62		1,3,5-Trimethylbenzene	2.248	2.331	-3.7	112	0.00
63		4-Chlorotoluene	1.924	1.999	-3.9	115	0.00
64		t-Butyl benzene	2.102	2.294	-9.1	116	0.00
65		1,2,4-Trimethylbenzene	2.295	2.381	-3.7	115	0.00
66		sec-Butyl benzene	3.301	3.520	-6.6	119	0.00
67		1,3-Dichlorobenzene	1.563	1.598	-2.2	115	0.00
68		p-Isopropyl toluene	2.694	2.859	-6.1	114	0.00
69		1,4-Dichlorobenzene	1.528	1.543	-1.0	109	0.00
70		n-Butyl benzene	2.515	2.682	-6.6	119	0.00
71		1,2-Dichlorobenzene	1.326	1.364	-2.9	113	0.00
72		1,2-Dibromo-3-chloropropane	0.095	0.090	5.3	108	0.00
73		1,2,4-Trichlorobenzene	0.984	1.028	-4.5	117	0.00
74		Hexachlorobutadiene	0.563	0.613	-8.9	122	0.00
75		Naphthalene	1.475	1.393	5.6	109	0.00
76		1,2,3-Trichlorobenzene	0.832	0.804	3.4	109	0.00

(#) = Out of Range SPCC's out = 0 CCC's out = 0
004A.D VOCA0212.M Thu Feb 14 13:00:31 2008 Page 2

FIGURE 4
CALCULATION PAGE FOR AVERAGE RESPONSE FACTORS AND STATISTICS

Response Factor Report GC/MS Ins

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration

Calibration Files
1 =012A.D 2 =013A.D 3 =014A.D
4 =015A.D 5 =016A.D 6 =017A.D

		Compound	1	2	3	4	5	6	Avg	%RSD
1)	I	Pentafluorobenzene	----------ISTD----------							
2)		Dichlorodifluoromet	0.403	0.402	0.407	0.411	0.425	0.413	0.410	1.87
3)	P	Chloromethane	0.277	0.286	0.279	0.282	0.284	0.274	0.279	2.17
4)	C	Vinyl Chloride	0.262	0.268	0.273	0.236	0.263	0.252	0.257	5.25
5)		Bromomethane	0.034	0.037	0.047	0.031	0.040	0.035	0.037	14.37
6)		Chloroethane	0.106	0.167		0.159		0.128	0.138	18.11
7)		Trichlorofluorometh	0.581	0.493	0.572	0.529	0.538	0.534	0.536	5.90
8)		1,1,2-Trichlorotrif	0.486	0.470	0.477	0.443	0.437	0.436	0.454	5.01
9)	C	1,1-Dichloroethene	0.421	0.441	0.423	0.408	0.417	0.385	0.413	4.38
10)		Acetone	0.022	0.014	0.015	0.013	0.014	0.014	0.015	19.65
11)		Carbon Disulfide	1.602	1.491	1.460	1.418	1.427	1.371	1.445	5.94
12)		Methylene Chloride	0.443	0.402	0.389	0.397	0.417	0.399	0.406	4.53
13)		Methyl-t-butyl Ethe	0.555	0.594	0.542	0.568	0.551	0.546	0.560	3.16
14)		t-1,2-Dichloroethen	0.484	0.476	0.460	0.469	0.459	0.454	0.464	2.60
15)		n-Hexane	0.562	0.472	0.482	0.485	0.495	0.482	0.497	6.05
16)	P	1,1-Dichloroethane	0.684	0.640	0.676	0.633	0.641	0.614	0.644	4.13
17)		Diisopropyl Ether	0.277	0.269	0.285	0.266	0.260	0.257	0.269	3.51
18)		2.2- Dichloropropan	0.465	0.450	0.432	0.426	0.407	0.405	0.427	5.53
19)		c-1,2-Dichloroethen	0.525	0.520	0.519	0.522	0.514	0.498	0.514	2.05
20)		2-Butanone(MEK)	0.025	0.025	0.022	0.022	0.021	0.020	0.022	8.39
21)		Tetrahydrofuran	0.021	0.026	0.022	0.023	0.023	0.023	0.023	6.07
22)		Bromochloromethane	0.244	0.287	0.285	0.259	0.260	0.259	0.265	5.91
23)	C	Chloroform	0.686	0.692	0.702	0.677	0.693	0.659	0.681	2.55
24)	S	Dibromofluoromethan	0.508	0.516	0.511	0.506	0.504	0.505	0.510	1.07
25)		1,1,1-Trichloroetha	0.633	0.533	0.569	0.583	0.560	0.548	0.568	5.80
26)		Carbon tetrachlorid	0.521	0.502	0.509	0.509	0.510	0.496	0.507	1.57
27)		1.1-Dichloropropene	0.579	0.536	0.558	0.552	0.539	0.532	0.547	3.09
28)		Benzene	1.726	1.695	1.617	1.671	1.663	1.585	1.644	3.80
29)		1,2-Dichloroethane	0.279	0.298	0.306	0.312	0.307	0.293	0.299	3.68
30)	I	1,4-Difluorobenzene	----------ISTD----------							
31)		Trichloroethene	0.345	0.326	0.338	0.320	0.321	0.305	0.324	4.15
32)	C	1,2-Dichloropropane	0.272	0.255	0.260	0.248	0.248	0.248	0.255	3.43
33)		Dibromomethane	0.187	0.174	0.174	0.176	0.168	0.167	0.173	4.42
34)		Bromodichloromethan	0.338	0.336	0.339	0.336	0.334	0.319	0.333	2.24
35)		c-1,3-Dichloroprope	0.409	0.398	0.367	0.380	0.371	0.386	0.384	3.81
36)		Methyl Isobutyl Ket	0.043	0.045	0.045	0.045	0.047	0.049	0.046	4.09
37)	S	Toluene-D8	1.050	1.060	1.071	1.054	1.062	1.064	1.060	0.64
38)	C	Toluene	0.967	0.813	0.827	0.778	0.773	0.741	0.805	9.85
39)		1,1,2-Trichloroetha	0.176	0.194	0.184	0.180	0.176	0.182	0.182	3.38
40)	I	Chlorobenzene-D5	----------ISTD----------							
41)		t-1,3-Dichloroprope	0.331	0.283	0.301	0.327	0.308	0.312	0.311	5.20

(#) = Out of Range ### Number of calibration levels exceeded format ###
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FIGURE 4
CALCULATION PAGE FOR AVERAGE RESPONSE FACTORS AND STATISTICS

Response Factor Report GC/MS Ins

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration

Calibration Files
1 =012A.D 2 =013A.D 3 =014A.D
4 =015A.D 5 =016A.D 6 =017A.D

		Compound	1	2	3	4	5	6	Avg	%RSD
42)		Tetrachloroethene	0.374	0.369	0.336	0.361	0.343	0.341	0.351	4.68
43)		1,3-Dichloropropane	0.362	0.401	0.368	0.383	0.372	0.368	0.373	3.91
44)		2-Hexanone	0.045	0.043	0.045	0.046	0.047	0.050	0.046	4.84
45)		Dibromochloromethan	0.306	0.321	0.307	0.326	0.307	0.311	0.313	2.46
46)		1,2-Dibromoethane	0.272	0.261	0.261	0.259	0.268	0.258	0.263	1.95
47)	P	Chlorobenzene	0.999	1.017	0.965	0.983	0.980	0.943	0.971	3.74
48)		1,1,1,2-Tetrachloro	0.367	0.335	0.323	0.319	0.333	0.314	0.329	5.85
49)	C	Ethylbenzene	1.643	1.528	1.535	1.523	1.528	1.469	1.514	5.32
50)		m+p-Xylene	0.707	0.633	0.642	0.646	0.614	0.605	0.631	6.78
51)		o-Xylene	0.634	0.612	0.608	0.613	0.604	0.602	0.607	2.71
52)		Styrene	1.006	0.974	0.972	0.993	1.022	0.992	0.988	2.28
53)	P	Bromoform	0.221	0.190	0.203	0.211	0.203	0.200	0.205	4.72
54)		Isopropylbenzene	1.394	1.338	1.310	1.327	1.329	1.313	1.323	3.29
55)	S	Bromofluorobenzene	0.406	0.420	0.413	0.416	0.437	0.426	0.419	2.41
56)	I	1,4-Dichlorobenzene-D	----------	ISTD	----------					
57)	P	1,1,2,2-Tetrachloro	0.504	0.487	0.481	0.465	0.424	0.466	0.469	5.40
58)		Bromobenzene	0.988	0.820	0.826	0.814	0.820	0.796	0.838	8.03
59)		1,2,3-Trichloroprop	0.423	0.393	0.384	0.308	0.359	0.361	0.370	9.72
60)		n-Propyl benzene	3.616	3.439	3.531	3.480	3.311	3.248	3.387	5.42
61)		2-Chlorotoluene	2.402	2.231	2.192	2.167	2.105	2.086	2.174	5.55
62)		1,3,5-Trimethylbenz	2.375	2.284	2.345	2.292	2.183	2.143	2.248	4.50
63)		4-Chlorotoluene	1.995	2.014	1.929	1.918	1.887	1.863	1.924	3.15
64)		t-Butyl benzene	2.162	2.091	2.092	2.184	2.042	2.068	2.102	2.45
65)		1,2,4-Trimethylbenz	2.521	2.289	2.397	2.279	2.197	2.209	2.295	5.47
66)		sec-Butyl benzene	3.563	3.347	3.407	3.254	3.219	3.192	3.301	4.51
67)		1,3-Dichlorobenzene	1.756	1.639	1.591	1.527	1.490	1.481	1.563	6.85
68)		p-Isopropyl toluene	2.729	2.656	2.756	2.776	2.679	2.668	2.694	2.38
69)		1,4-Dichlorobenzene	1.674	1.567	1.555	1.564	1.464	1.445	1.528	5.71
70)		n-Butyl benzene	2.564	2.451	2.722	2.485	2.460	2.480	2.515	3.97
71)		1,2-Dichlorobenzene	1.380	1.380	1.363	1.333	1.296	1.258	1.326	3.88
72)		1,2-Dibromo-3-chlor	0.077	0.107	0.095	0.092	0.098	0.097	0.095	9.71
73)		1,2,4-Trichlorobenz	1.069	1.019	1.035	0.966	0.937	0.921	0.984	5.84
74)		Hexachlorobutadiene	0.580	0.597	0.549	0.555	0.560	0.547	0.563	3.32
75)		Naphthalene	1.630	1.562	1.443	1.412	1.357	1.441	1.475	6.29
76)		1,2,3-Trichlorobenz	0.898	0.833	0.881	0.814	0.785	0.799	0.832	5.07

(#) = Out of Range ### Number of calibration levels exceeded format ###
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FIGURE 5
ELECTRON CAPTURE CHROMATOGRAM FOR SCREENING DATA

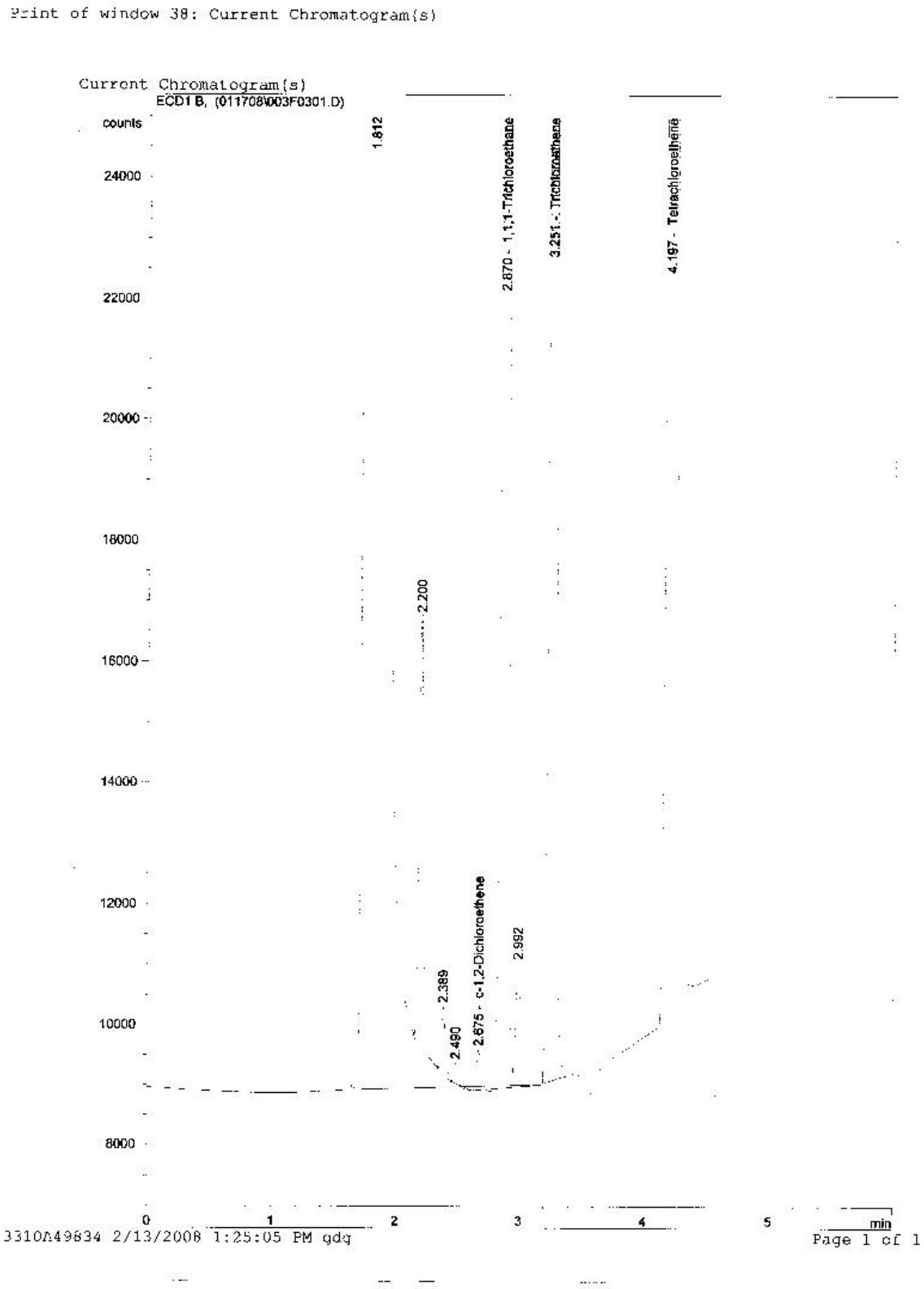
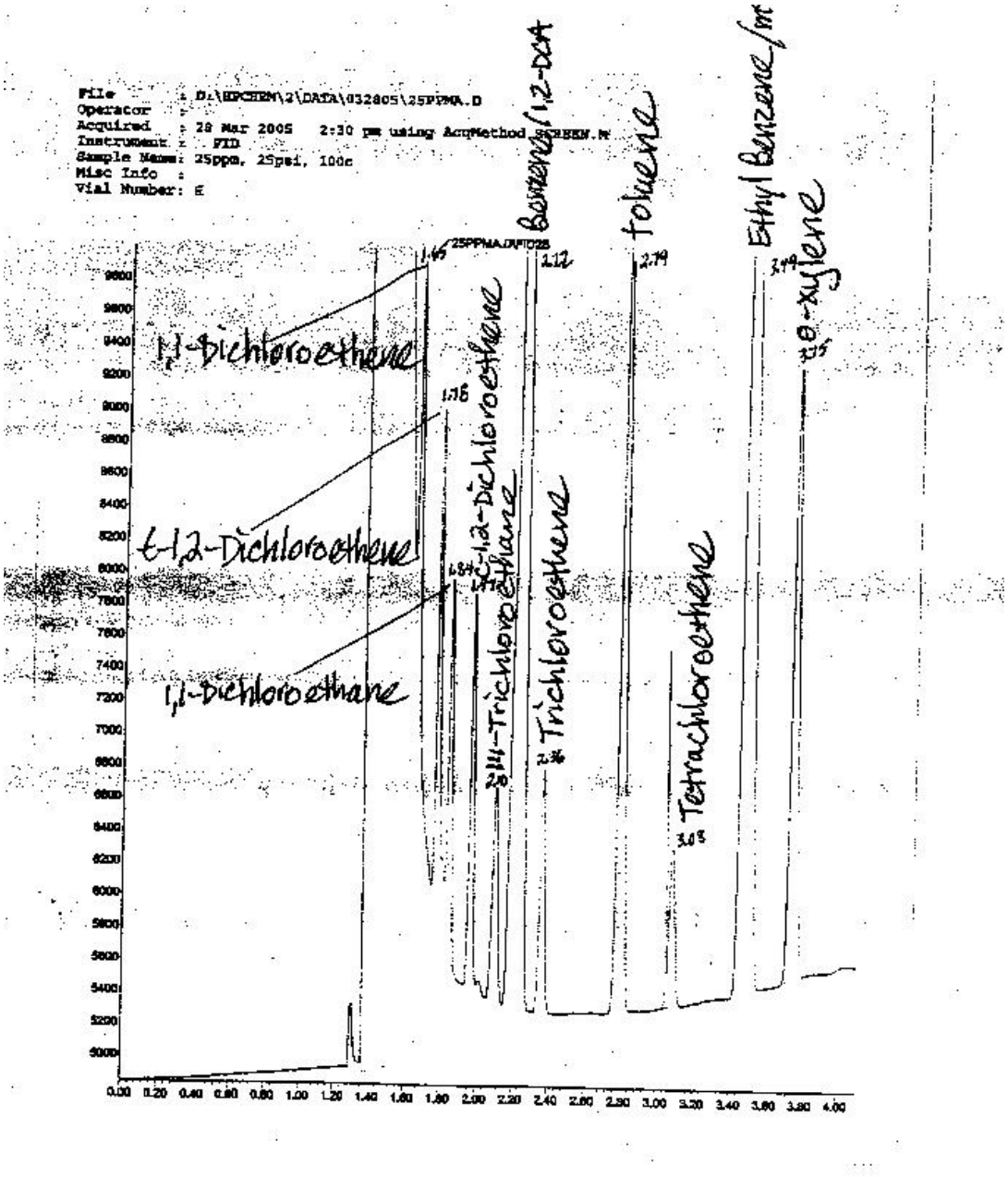


FIGURE 6
FID CHROMATOGRAM FOR SCREENING DATA



The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

Reviewed By:

02/25/10

Karl Olm, Operations Manager — Date

02/25/10

Michael Linskens, Quality Manager — Date

Approved By:

02/25/10

Nick Nigro, President — Date

Appendix D
Michigan Part 201 Criteria Tables

TABLE 1. GROUNDWATER: RESIDENTIAL AND INDUSTRIAL-COMMERCIAL PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

All criteria, unless otherwise noted, are expressed in units of parts per billion (ppb). One ppb is equivalent to one microgram per liter (ug/L). Criteria with six or more digits are expressed in scientific notation. For example, 200,000 ppb is presented as 2.0E+5. The lowest generic groundwater criterion for a given hazardous substance is presented in a bold box. A footnote is designated by a letter in parentheses and is explained in the footnote pages that follow the criteria tables. When the risk-based criterion is less than the target detection limit (TDL), the TDL is listed as the criterion (R 299.5707). In these cases, two numbers are presented in the cell. The first number is the criterion (i.e., TDL), and the second number is the risk-based or solubility value, whichever is lower (R 299.5708). Criteria were promulgated December 21, 2002 within the Administrative Rules for Part 201, Environmental Remediation, of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended. These tables reflect modifications to the TDLs and new criteria consistent with the provisions of R299.5103(l) and R299.5706a, respectively.

Guidesheet Number →		#1	#2	#3	#4	#5	#6	#7	#8	#9
	Chemical Abstract Service Number	**Residential & Commercial I Drinking Water Criteria & RBSLs**	**Industrial & Commercial II, III & IV Drinking Water Criteria & RBSLs**	**Groundwater Surface Water Interface Criteria & RBSLs**	**Residential & Commercial I Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Industrial & Commercial II, III & IV Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Groundwater Contact Criteria & RBSLs**	**Water Solubility**	**Flammability and Explosivity Screening Level**	**Acute Inhalation Screening Level**
Acenaphthene	83329	1,300	3,800	19	4,200 (S)	4,200 (S)	4,200 (S)	4,240	ID	ID
Acenaphthylene	208968	52	150	ID	3,900 (S)	3,900 (S)	3,900 (S)	3,930	ID	ID
Acetaldehyde (I)	75070	950	2,700	130	1.1E+6	2.3E+6	4.2E+7	1.0E+9	8.9E+6	2.6E+7
Acetate	71501	4,200	12,000	(G)	ID	ID	ID	ID	ID	ID
Acetic acid	64197	4,200	12,000	1,000 (M); 360	NLV	NLV	1.8E+8	6.0E+9	1.0E+9 (D)	1.0E+9 (D)
Acetone (I)	67641	730	2,100	1,700	1.0E+9 (D,S)	1.0E+9 (D,S)	3.1E+7	1.0E+9	1.5E+7	1.0E+9 (D)
Acetonitrile	75058	140	400	NA	2.4E+7	4.5E+7	5.6E+6	2.00E+8	2.1E+7	2.0E+8
Acetophenone	98862	1,500	4,400	ID	6.1E+6 (S)	6.1E+6 (S)	6.1E+6 (S)	6.1E+6	ID	ID
Acrolein (I)	107028	120	330	NA	2,100	4,200	3.4E+6	2.10E+8	6.7E+6	3.4E+5
Acrylamide	79061	0.5 (A)	0.5 (A)	NA	NLV	NLV	13,000	2.20E+9	NA	ID
Acrylic acid	79107	3,900	11,000	NA	1.2E+7	2.8E+7	7.6E+7	1.0E+9	1.0E+9 (D)	ID
Acrylonitrile (I)	107131	2.6	11	4.9 (X)	34,000	1.9E+5	14,000	7.50E+7	6.4E+6	ID
Alachlor	15972608	2.0 (A)	2.0 (A)	11 (X)	NLV	NLV	1,700	1.83E+5	ID	ID
Aldicarb	116063	3.0 (A)	3.0 (A)	NA	NLV	NLV	1.2E+5	6.00E+6	ID	ID
Aldicarb sulfoxide	1646873	4.0 (A)	4.0 (A)	NA	NLV	NLV	2.7E+6	2.80E+7	ID	ID
Aldicarb sulfone	1646884	2.0 (A)	2.0 (A)	NA	NLV	NLV	2.1E+6	7.80E+6	ID	ID
Aldrin	309002	0.098	0.4	0.01 (M,X); 8.7E-6	180 (S)	180 (S)	0.34 (AA)	180	ID	ID
Aluminum (B)	7429905	50 (V)	50 (V)	NA	NLV	NLV	6.4E+7	NA	ID	ID
Ammonia	7664417	10,000 (N)	10,000 (N)	(CC)	3.2E+6	7.1E+6	ID	5.30E+8	ID	3.5E+6

TABLE 1. GROUNDWATER: RESIDENTIAL AND INDUSTRIAL-COMMERCIAL PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

Guidesheet Number →		#1	#2	#3	#4	#5	#6	#7	#8	#9
	Chemical Abstract Service Number	**Residential & Commercial I Drinking Water Criteria & RBSLs**	**Industrial & Commercial II, III & IV Drinking Water Criteria & RBSLs**	**Groundwater Surface Water Interface Criteria & RBSLs**	**Residential & Commercial I Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Industrial & Commercial II, III & IV Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Groundwater Contact Criteria & RBSLs**	**Water Solubility**	**Flammability and Explosivity Screening Level**	**Acute Inhalation Screening Level**
t-Amyl methyl ether (TAME)	994058	190 (E)	190 (E)	NA	2.6E+5	5.7E+5	2.6E+6 (S)	2.64E+6	NA	NA
Aniline	62533	53	220	4	NLV	NLV	1.4E+5	3.60E+7	NA	ID
Anthracene	120127	43 (S)	43 (S)	ID	43 (S)	43 (S)	43 (S)	43.4	ID	ID
Antimony	7440360	6.0 (A)	6.0 (A)	130 (X)	NLV	NLV	68,000	NA	ID	ID
Arsenic	7440382	10 (A)	10 (A)	150 (X)	NLV	NLV	4,300	NA	ID	ID
Asbestos (BB)	1332214	7.0E+6 f/mL (A)	7.0E+6 f/mL (A)	NA	NLV	NLV	ID	NA	NA	ID
Atrazine	1912249	3.0 (A)	3.0 (A)	7.3 (X)	NLV	NLV	5,400	70,000	ID	ID
Azobenzene	103333	23	94	NA	6,400 (S)	6,400 (S)	1,600	6,400	ID	ID
Barium (B)	7440393	2,000 (A)	2,000 (A)	(G,X)	NLV	NLV	1.4E+7	NA	ID	ID
Benzene (I)	71432	5.0 (A)	5.0 (A)	200 (X)	5,600	35,000	11,000	1.75E+6	68,000	67,000
Benzidine	92875	0.3 (M); 0.0037	0.3 (M); 0.015	ID	NLV	NLV	7.1	5.20E+5	ID	ID
Benzo(a)anthracene (Q)	56553	2.1	8.5	ID	NLV	NLV	9.4 (S,AA)	9.4	ID	ID
Benzo(b)fluoranthene (Q)	205992	1.5 (S, AA)	1.5 (S, AA)	ID	ID	ID	1.5 (S,AA)	1.5	ID	ID
Benzo(k)fluoranthene (Q)	207089	1.0 (M); 0.8 (S)	1.0 (M); 0.8 (S)	NA	NLV	NLV	1.0 (M,AA); 0.8 (S)	0.8	ID	ID
Benzo(g,h,i)perylene	191242	1.0 (M); 0.26 (S)	1.0 (M); 0.26 (S)	NA	NLV	NLV	1.0 (M,AA); 0.26 (S)	0.26	ID	ID
Benzo(a)pyrene (Q)	50328	5.0 (A)	5.0 (A)	ID	NLV	NLV	1.0 (M,AA); 0.64	1.62	ID	ID
Benzoic acid	65850	32,000	92,000	NA	NLV	NLV	3.5E+6 (S)	3.50E+6	ID	ID
Benzyl alcohol	100516	10,000	29,000	NA	NLV	NLV	4.4E+7 (S)	4.40E+7	ID	ID
Benzyl chloride	100447	7.7	32	NA	12,000	77,000	3,600	4.90E+5	NA	ID
Beryllium	7440417	4.0 (A)	4.0 (A)	(G)	NLV	NLV	2.9E+5	NA	ID	ID
bis(2-Chloroethoxy)ethane	112265	ID	ID	ID	NLV	NLV	ID	1.89E+7	ID	ID
bis(2-Chloroethyl)ether (I)	111444	2.0	8.3	15 (X)	38,000	2.1E+5	5,700	1.72E+7	1.7E+7 (S)	1.7E+7 (S)
bis(2-Ethylhexyl)phthalate	117817	6.0 (A)	6.0 (A)	32	NLV	NLV	320 (AA)	340	NA	340 (S)
Boron (B)	7440428	500 (F)	500 (F)	1,900	NLV	NLV	6.2E+7	NA	ID	ID
Bromate	15541454	10 (A)	10 (A)	40 (X)	NLV	NLV	4,800	38,000	ID	ID
Bromobenzene (I)	108861	18	50	NA	1.8E+5	3.9E+5	12,000	4.13E+5	ID	ID

TABLE 1. GROUNDWATER: RESIDENTIAL AND INDUSTRIAL-COMMERCIAL PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

Guidesheet Number →		#1	#2	#3	#4	#5	#6	#7	#8	#9
	Chemical Abstract Service Number	**Residential & Commercial I Drinking Water Criteria & RBSLs**	**Industrial & Commercial II, III & IV Drinking Water Criteria & RBSLs**	**Groundwater Surface Water Interface Criteria & RBSLs**	**Residential & Commercial I Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Industrial & Commercial II, III & IV Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Groundwater Contact Criteria & RBSLs**	**Water Solubility**	**Flammability and Explosivity Screening Level**	**Acute Inhalation Screening Level**
Bromodichloromethane	75274	80 (A,W)	80 (A,W)	ID	4,800	37,000	14,000	6.74E+6	ID	ID
Bromoform	75252	80 (A,W)	80 (A,W)	ID	4.7E+5	3.1E+6 (S)	1.4E+5	3.10E+6	ID	ID
Bromomethane	74839	10	29	35	4,000	9,000	70,000	1.45E+7	ID	ID
n-Butanol (I)	71363	950	2,700	NA	NLV	NLV	8.8E+6	7.40E+7	4.7E+7	7.4E+7 (S)
2-Butanone (MEK) (I)	78933	13,000	38,000	2,200	2.4E+8 (S)	2.4E+8 (S)	2.4E+8 (S)	2.40E+8	ID	2.4E+8 (S)
n-Butyl acetate	123864	550	1,600	NA	6.7E+6 (S)	6.7E+6 (S)	1.8E+6	6.70E+6	2.5E+6	6.7E+6 (S)
t-Butyl alcohol	75650	3,900	11,000	NA	1.0E+9 (D,S)	1.0E+9 (D,S)	7.9E+7	1.0E+9	6.1E+7	ID
Butyl benzyl phthalate	85687	1,200	2,700 (S)	14 (X)	NLV	NLV	2,700 (S)	2,690	ID	ID
n-Butylbenzene	104518	80	230	ID	ID	ID	5,900	NA	ID	ID
sec-Butylbenzene	135988	80	230	ID	ID	ID	4,400	NA	ID	ID
tert-Butylbenzene (I)	98066	80	230	ID	ID	ID	8,900	NA	ID	ID
Cadmium (B)	7440439	5.0 (A)	5.0 (A)	(G,X)	NLV	NLV	1.9E+5	NA	ID	ID
Camphene (I)	79925	ID	ID	NA	ID	ID	ID	33,400	ID	ID
Caprolactam	105602	5,800	17,000	NA	NLV	NLV	3.9E+8	5.25E+9	NA	1.0E+9 (D)
Carbaryl	63252	700	2,000	NA	ID	ID	1.3E+5 (S)	1.26E+5	ID	ID
Carbazole	86748	85	350	10 (M); 3.9	NLV	NLV	7,400	7,480	ID	ID
Carbofuran	1563662	40 (A)	40 (A)	NA	NLV	NLV	3.4E+5	7.00E+5	ID	ID
Carbon disulfide (I,R)	75150	800	2,300	ID	2.5E+5	5.5E+5	1.2E+6 (S)	1.19E+6	13,000	ID
Carbon tetrachloride	56235	5.0 (A)	5.0 (A)	45 (X)	370	2,400	4,600	7.93E+5	ID	96,000
Chlordane (J)	57749	2.0 (A)	2.0 (A)	NA	56 (S)	56 (S)	15 (AA)	56	ID	ID
Chloride	16887006	2.5E+5 (E)	2.5E+5 (E)	(FF)	NLV	NLV	ID	NA	ID	ID
Chlorobenzene (I)	108907	100 (A)	100 (A)	47	2.1E+5	4.7E+5 (S)	86,000	4.72E+5	1.6E+5	ID
para-Chlorobenzenesulfonic acid	98668	7,300	21,000	NA	ID	ID	ID	NA	ID	ID
1-Chloro-1,1-difluoroethane	75683	15,000	44,000	NA	3.9E+6 (S)	3.9E+6 (S)	3.9E+6 (S)	3.9E+06	NA	ID
Chloroethane	75003	430	1,700	ID	5.7E+6 (S)	5.7E+6 (S)	4.4E+5	5.74E+6	1.1E+5	ID
2-Chloroethyl vinyl ether	110758	ID	ID	NA	ID	ID	ID	1.50E+7	ID	ID

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Chloroform	67663	80 (A,W)	80 (A,W)	170 (X)	28,000	1.8E+5	1.5E+5	7.92E+6	ID	ID
Chloromethane (I)	74873	260	1,100	ID	8,600	45,000	4.9E+5	6.34E+6	36,000	2.1E+5
4-Chloro-3-methylphenol	59507	150	420	7.4	NLV	NLV	79,000	3.90E+6	ID	ID
beta-Chloronaphthalene	91587	1,800	5,200	NA	ID	ID	6,700 (S)	6,740	ID	ID
2-Chlorophenol	95578	45	130	22	ID	ID	94,000	2.20E+7	ID	ID
o-Chlorotoluene (I)	95498	150	420	ID	2.2E+5	3.7E+5 (S)	44,000	3.73E+5	ID	ID
Chlorpyrifos	2921882	22	63	2.0 (M); 0.002	2.9	6.6	1,100 (S)	1,120	ID	ID
Chromium (III) (B,H)	16065831	100 (A)	100 (A)	(G,X)	NLV	NLV	2.9E+8	NA	ID	ID
Chromium (VI)	18540299	100 (A)	100 (A)	11	NLV	NLV	4.6E+5	NA	ID	ID
Chrysene (Q)	218019	1.6 (S)	1.6 (S)	ID	ID	ID	1.6 (S,AA)	1.6	ID	ID
Cobalt	7440484	40	100	100	NLV	NLV	2.4E+6	NA	ID	ID
Copper (B)	7440508	1,000 (E)	1,000 (E)	(G)	NLV	NLV	7.4E+6	NA	ID	ID
Cyanazine	21725462	2.3	9.4	56 (X)	NLV	NLV	2,800	1.70E+5	ID	ID
Cyanide (P,R)	57125	200 (A)	200 (A)	5.2	NLV	NLV	57,000	NA	ID	ID
Cyclohexanone	108941	33,000	94,000	NA	1,500	3,300	2.3E+7 (S)	2.30E+7	NA	ID
Dacthal	1861321	73	210	NA	NLV	NLV	500 (S)	500	ID	ID
Dalapon	75990	200 (A)	200 (A)	NA	NLV	NLV	1.2E+7	5.02E+8	ID	ID
4-4'-DDD	72548	9.1	37	NA	NLV	NLV	44 (AA)	90	ID	ID
4-4'-DDE	72559	4.3	15	NA	NLV	NLV	27 (AA)	120	ID	ID
4-4'-DDT	50293	3.6	10	0.02 (M); 1.1E-5	NLV	NLV	13 (AA)	25	NA	ID
Decabromodiphenyl ether	1163195	30 (S)	30 (S)	NA	30 (S)	30 (S)	30 (S)	30	ID	ID
Di-n-butyl phthalate	84742	880	2,500	9.7	NLV	NLV	11,000 (S)	11,200	NA	ID
Di(2-ethylhexyl) adipate	103231	400 (A)	400 (A)	ID	NLV	NLV	470 (S)	471	ID	ID
Di-n-octyl phthalate	117840	130	380	ID	NLV	NLV	400	3,000	ID	ID
Diacetone alcohol (I)	123422	ID	ID	NA	NLV	NLV	ID	1.0E+9	1.0E+9 (S)	ID
Diazinon	333415	1.3	3.8	NA	NLV	NLV	1,300	68,800	NA	ID

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	Chemical Abstract Service Number	**Residential & Commercial I Drinking Water Criteria & RBSLs**	**Industrial & Commercial II, III & IV Drinking Water Criteria & RBSLs**	**Groundwater Surface Water Interface Criteria & RBSLs**	**Residential & Commercial I Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Industrial & Commercial II, III & IV Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Groundwater Contact Criteria & RBSLs**	**Water Solubility**	**Flammability and Explosivity Screening Level**	**Acute Inhalation Screening Level**
Dibenzo(a,h)anthracene (Q)	53703	2.0 (M); 0.21	2.0 (M); 0.85	ID	NLV	NLV	2.0 (M,AA); 0.31	2.49	ID	ID
Dibenzofuran	132649	ID	ID	4	ID	ID	ID	10,000	ID	ID
Dibromochloromethane	124481	80 (A,W)	80 (A,W)	ID	14,000	1.1E+5	18,000	2.60E+6	ID	ID
Dibromochloropropane	96128	0.2 (A)	0.2 (A)	NA	1,200 (S)	1,200 (S)	390	1,230	NA	ID
Dibromomethane	74953	80	230	NA	ID	ID	5.3E+5	1.10E+7	ID	ID
Dicamba	1918009	220	630	NA	NLV	NLV	5.9E+5	4.5E+6	ID	ID
1,2-Dichlorobenzene	95501	600 (A)	600 (A)	16	1.6E+5 (S)	1.6E+5 (S)	1.6E+5 (S)	1.56E+5	NA	1.6E+5 (S)
1,3-Dichlorobenzene	541731	6.6	19	38	ID	ID	2,000	1.11E+5	ID	ID
1,4-Dichlorobenzene	106467	75 (A)	75 (A)	13	16,000	74,000 (S)	6,400	73,800	NA	ID
3,3'-Dichlorobenzidine	91941	1.1	4.3	0.3 (X)	NLV	NLV	180	3,110	ID	ID
Dichlorodifluoromethane	75718	1,700	4,800	ID	2.2E+5	3.0E+5 (S)	3.0E+5 (S)	3.00E+5	ID	ID
1,1-Dichloroethane	75343	880	2,500	740	1.0E+6	2.3E+6	2.4E+6	5.06E+6	3.8E+5	ID
1,2-Dichloroethane (I)	107062	5.0 (A)	5.0 (A)	360 (X)	9,600	59,000	19,000	8.52E+6	2.5E+6	ID
1,1-Dichloroethylene (I)	75354	7.0 (A)	7.0 (A)	65 (X)	200	1,300	11,000	2.25E+6	97,000	1.4E+5
cis-1,2-Dichloroethylene	156592	70 (A)	70 (A)	620	93,000	2.1E+5	2.0E+5	3.50E+6	5.3E+5	ID
trans-1,2-Dichloroethylene	156605	100 (A)	100 (A)	1,500	85,000	2.0E+5	2.2E+5	6.30E+6	2.3E+5	ID
2,6-Dichloro-4-nitroaniline	99309	2,200	6,300	NA	NLV	NLV	7,000 (S)	7,000	ID	ID
2,4-Dichlorophenol	120832	73	210	19	NLV	NLV	48,000	4.50E+6	ID	ID
2,4-Dichlorophenoxyacetic acid	94757	70 (A)	70 (A)	220	NLV	NLV	1.2E+5	6.80E+5	ID	ID
1,2-Dichloropropane (I)	78875	5.0 (A)	5.0 (A)	290 (X)	16,000	36,000	16,000	2.80E+6	5.5E+5	2.8E+6 (S)
1,3-Dichloropropene	542756	8.5	35	NA	3,900	26,000	5,500	2.80E+6	1.3E+5	ID
Dichlorovos	62737	1.6	6.7	NA	NLV	NLV	5,900	1.60E+7	NA	ID
Dicyclohexyl phthalate	84617	ID	ID	NA	ID	ID	ID	4,000	ID	ID
Dieldrin	60571	0.11	0.43	0.02 (M); 6.5E-6	200 (S)	200 (S)	2.4 (AA)	195	ID	ID
Diethyl ether	60297	10 (E)	10 (E)	ID	6.1E+7 (S)	6.1E+7 (S)	3.5E+7	6.10E+7	6.5E+5	6.1E+7 (S)
Diethyl phthalate	84662	5,500	16,000	110	NLV	NLV	1.1E+6 (S)	1.08E+6	NA	ID

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Diethylene glycol monobutyl ether	112345	88	250	NA	NLV	NLV	4.0E+6	1.0E+9	ID	ID
Diisopropyl ether	108203	30	86	ID	8,000 (S)	8,000 (S)	8,000 (S)	8,041	8,000 (S)	ID
Diisopropylamine (I)	108189	5.6	16	NA	ID	ID	21,000	3.69E+7	4.6E+6	ID
Dimethyl phthalate	131113	73,000	2.1E+5	NA	NLV	NLV	4.2E+6 (S)	4.19E+6	NA	ID
N,N-Dimethylacetamide	127195	180	520	4,100 (X)	NLV	NLV	2.3E+7	1.0E+9	NA	ID
N,N-Dimethylaniline	121697	16	46	NA	2.4E+5	1.3E+6 (S)	20,000	1.27E+6	NA	1.3E+6 (S)
Dimethylformamide (I)	68122	700	2,000	NA	NLV	NLV	1.1E+8	1.0E+9	ID	ID
2,4-Dimethylphenol	105679	370	1,000	380	NLV	NLV	5.2E+5	7.87E+6	ID	ID
2,6-Dimethylphenol	576261	4.4	13	NA	NLV	NLV	6,300	6.14E+6	ID	ID
3,4-Dimethylphenol	95658	10	29	NA	NLV	NLV	18,000	4.93E+6	ID	ID
Dimethylsulfoxide	67685	2.2E+5	6.3E+5	1.9E+5	NLV	NLV	1.7E+8 (S)	1.66E+8	ID	ID
2,4-Dinitrotoluene	121142	7.7	32	NA	NLV	NLV	8,600	2.70E+5	ID	ID
Dinoseb	88857	7.0 (A)	7.0 (A)	1.0 (M); 0.48	NLV	NLV	7,000	52,000	ID	ID
1,4-Dioxane (I)	123911	85	350	2,800 (X)	NLV	NLV	1.7E+6	9.00E+8	1.4E+8	ID
Diquat	85007	20 (A)	20 (A)	NA	NLV	NLV	7.0E+5 (S)	7.00E+5	ID	ID
Diuron	330541	31	90	NA	NLV	NLV	37,000 (S)	37,300	ID	ID
Endosulfan (J)	115297	44	130	0.03	ID	ID	510 (S)	510	ID	ID
Endothall	145733	100 (A)	100 (A)	NA	NLV	NLV	2.5E+7 (AA)	1.00E+8	ID	ID
Endrin	72208	2.0 (A)	2.0 (A)	NA	NLV	NLV	160 (AA)	250	ID	ID
Epichlorohydrin (I)	106898	5.0 (M); 2.0 (A)	5.0 (M); 2.0 (A)	NA	3.2E+5	6.3E+5	11,000	6.60E+7	4.7E+7	ID
Ethanol (I)	64175	1.9E+6	3.8E+6	NA	NLV	NLV	1.0E+9 (S)	1.0E+9	9.7E+7	ID
Ethyl acetate (I)	141786	6,600	19,000	NA	6.4E+7 (S)	6.4E+7 (S)	6.4E+7 (S)	6.40E+7	4.2E+6	ID
Ethyl-tert-butyl ether (ETBE)	637923	49 (E)	49 (E)	NA	2.9E+6	5.6E+6 (S)	ID	5.63E+6	ID	ID
Ethylbenzene (I)	100414	74 (E)	74 (E)	18	1.1E+5	1.7E+5 (S)	1.7E+5 (S)	1.69E+5	43,000	1.7E+5 (S)
Ethylene dibromide	106934	0.05 (A)	0.05 (A)	0.2 (X)	2,400	15,000	25	4.20E+6	ID	ID
Ethylene glycol	107211	15,000	42,000	1.9E+5 (X)	NLV	NLV	1.0E+9 (S)	1.0E+9	NA	1.0E+9 (S)

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Ethylene glycol monobutyl ether	111762	3,700	10,000	NA	2.9E+6	6.5E+6	5.3E+7	2.24E+8	NA	ID
Fluoranthene	206440	210 (S)	210 (S)	1.6	210 (S)	210 (S)	210 (S)	206	ID	ID
Fluorene	86737	880	2,000 (S)	12	2,000 (S)	2,000 (S)	2,000 (S)	1,980	ID	ID
Fluorine (soluble fluoride) (B)	7782414	2,000 (E)	2,000 (E)	NA	NLV	NLV	1.2E+7	NA	ID	ID
Formaldehyde	50000	1,300	3,800	120	63,000	3.6E+5	3.0E+7	5.50E+8	ID	61,000
Formic acid (I,U)	64186	10,000	29,000	ID	7.7E+6	1.5E+7	6.0E+8	1.0E+9	1.0E+9 (D)	3.5E+8
1-Formylpiperidine	2591868	80	230	NA	ID	ID	ID	NA	ID	ID
Gentian violet	548629	15	63	NA	NLV	NLV	1.0E+6 (S)	1.00E+6	ID	ID
Glyphosate	1071836	700 (A)	700 (A)	NA	NLV	NLV	1.2E+7 (S,AA)	1.16E+7	ID	ID
Heptachlor	76448	0.4 (A)	0.4 (A)	0.01 (M,X); 0.0018	180 (S)	180 (S)	2.9 (AA)	180	ID	ID
Heptachlor epoxide	1024573	0.2 (A)	0.2 (A)	ID	NLV	NLV	9.0 (AA)	200	ID	ID
n-Heptane	142825	2,700 (S)	2,700 (S)	NA	2,700 (S)	2,700 (S)	2,700 (S)	2,690	200	2,700 (S)
Hexabromobenzene	87821	0.17 (S); 20	0.17 (S); 58	ID	ID	ID	0.17 (S); 1,500	0.17	ID	ID
Hexachlorobenzene (C-66)	118741	1.0 (A)	1.0 (A)	0.2 (M); 0.0003	440	3,000	4.6	6,200	ID	ID
Hexachlorobutadiene (C-46)	87683	15	42	0.05	1,600	3,200 (S)	400	3,230	ID	ID
alpha-Hexachlorocyclohexane	319846	0.43	1.7	NA	2,000 (S)	2,000 (S)	60	2,000	ID	ID
beta-Hexachlorocyclohexane	319857	0.88	3.6	ID	NLV	NLV	120	240	ID	ID
Hexachlorocyclopentadiene (C-56)	77474	50 (A)	50 (A)	ID	130	420	1,600	1,800	ID	ID
Hexachloroethane	67721	7.3	21	6.7 (X)	27,000	50,000 (S)	1,900	50,000	ID	ID
n-Hexane	110543	3,000	8,600	NA	12,000 (S)	12,000 (S)	12,000 (S)	12,000	12,000 (S)	ID
2-Hexanone	591786	1,000	2,900	NA	4.2E+6	8.7E+6	5.2E+6	1.60E+7	NA	ID
Indeno(1,2,3-cd)pyrene (Q)	193395	2.0 (M); 0.022 (S)	2.0 (M); 0.022 (S)	ID	NLV	NLV	2.0 (M, AA); 0.022 (S)	0.022	ID	ID
Iron (B)	7439896	300 (E)	300 (E)	NA	NLV	NLV	5.8E+7	NA	ID	ID
Isobutyl alcohol (I)	78831	2,300	6,700	NA	7.6E+7 (S)	7.6E+7 (S)	2.5E+7	7.60E+7	ID	ID
Isophorone	78591	770	3,100	570 (X)	NLV	NLV	9.9E+5	1.20E+7	ID	1.2E+7 (S)
Isopropyl alcohol (I)	67630	470	1,300	57,000 (X)	NLV	NLV	1.3E+7	1.0E+9	6.0E+7	1.0E+9 (S)

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Isopropyl benzene	98828	800	2,300	ID	56,000 (S)	56,000 (S)	56,000 (S)	56,000	29,000	ID
Lead (B)	7439921	4.0 (L)	4.0 (L)	(G,X)	NLV	NLV	ID	NA	ID	ID
Lindane	58899	0.2 (A)	0.2 (A)	0.03	ID	ID	190	6,800	ID	ID
Lithium (B)	7439932	170	350	96	NLV	NLV	5.4E+6	NA	ID	ID
Magnesium (B)	7439954	4.0E+5	1.1E+6	NA	NLV	NLV	1.0E+9 (D)	NA	ID	ID
Manganese (B)	7439965	50 (E)	50 (E)	(G,X)	NLV	NLV	9.1E+6	NA	ID	ID
Mercury (Total) (B,Z)	Varies	2.0 (A)	2.0 (A)	0.0013	56 (S)	56 (S)	56 (S)	56	ID	ID
Methane (K)	74828	ID	ID	NA	(K)	(K)	ID	NA	520	ID
Methanol	67561	3,700	10,000	480	2.9E+7 (S)	2.9E+7 (S)	2.9E+7 (S)	2.90E+7	4.5E+6	2.9E+7 (S)
Methoxychlor	72435	40 (A)	40 (A)	NA	ID	ID	45 (S)	45	ID	ID
2-Methoxyethanol (I)	109864	7.3	21	NA	NLV	NLV	8.3E+5	1.0E+9	ID	ID
2-Methyl-4-chlorophenoxyacetic acid	94746	7.3	21	NA	NLV	NLV	9,200	9.24E+5	ID	ID
2-Methyl-4,6-dinitrophenol	534521	20 (M); 2.6	20 (M); 7.3	NA	NLV	NLV	9,500	2.00E+5	ID	ID
N-Methyl-morpholine (I)	109024	20	56	NA	NLV	NLV	1.5E+6	1.0E+9	ID	ID
Methyl parathion	298000	1.8	5.2	NA	NLV	NLV	3,000	50,000	ID	ID
4-Methyl-2-pentanone (MIBK) (I)	108101	1,800	5,200	ID	2.0E+7 (S)	2.0E+7 (S)	1.3E+7	2.00E+7	ID	2.0E+7 (S)
Methyl-tert-butyl ether (MTBE)	1634044	40 (E)	40 (E)	730 (X)	4.7E+7 (S)	4.7E+7 (S)	6.1E+5	4.68E+7	ID	ID
Methylcyclopentane (I)	96377	ID	ID	NA	ID	ID	ID	73,890	ID	ID
4,4'-Methylene-bis-2-chloroaniline (MBOCA)	101144	1.1	4.5	NA	NLV	NLV	110 (AA)	14,000	ID	ID
Methylene chloride	75092	5.0 (A)	5.0 (A)	940 (X)	2.2E+5	1.4E+6	2.2E+5	1.70E+7	ID	ID
2-Methylnaphthalene	91576	260	750	ID	ID	ID	25,000 (S)	24,600	ID	ID
Methylphenols (J)	1319773	370	1,000	71	NLV	NLV	8.1E+5	2.80E+7	NA	ID
Metolachlor	51218452	240	990	NA	NLV	NLV	91,000	5.30E+5	ID	ID
Metribuzin	21087649	180	520	NA	ID	ID	1.2E+6 (S)	1.2E+6	ID	ID
Mirex	2385855	0.02 (M); 6.8E-6 (S)	0.02 (M); 6.8E-6 (S)	0.02 (M); 6.8E-6 (S)	ID	ID	0.02 (M); 6.8E-6 (S)	6.8E-6	NA	ID
Molybdenum (B)	7439987	73	210	800 (X)	NLV	NLV	9.7E+5	NA	ID	ID

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Naphthalene	91203	520	1,500	13	31,000 (S)	31,000 (S)	31,000 (S)	31,000	NA	31,000 (S)
Nickel (B)	7440020	100 (A)	100 (A)	(G)	NLV	NLV	7.4E+7	NA	ID	ID
Nitrate (B,N)	14797558	10,000 (A,N)	10,000 (A,N)	NA	NLV	NLV	3.1E+8	NA	ID	ID
Nitrite (B,N)	14797650	1,000 (A,N)	1,000 (A,N)	NA	NLV	NLV	ID	NA	ID	ID
Nitrobenzene (I)	98953	3.4	9.6	180 (X)	2.8E+5	5.5E+5	11,000	2.09E+6	NA	ID
2-Nitrophenol	88755	20	58	ID	NLV	NLV	79,000	2.50E+6	ID	ID
n-Nitroso-di-n-propylamine	621647	5.0 (M); 0.19	5.0 (M); 0.77	NA	NLV	NLV	360	9.89E+6	ID	ID
N-Nitrosodiphenylamine	86306	270	1,100	NA	NLV	NLV	35,000 (S)	35,100	ID	ID
Oxamyl	23135220	200 (A)	200 (A)	NA	NLV	NLV	6.2E+7	2.80E+8	ID	ID
Oxo-hexyl acetate	88230357	73	210	NA	ID	ID	ID	NA	ID	ID
Pendimethalin	40487421	280 (S)	280 (S)	NA	NLV	NLV	280 (S)	275	ID	ID
Pentachlorobenzene	608935	6.1	17	5.0 (M); 0.019	ID	ID	240	650	ID	ID
Pentachloronitrobenzene	82688	32 (S)	32 (S)	NA	32 (S)	32 (S)	32 (S)	32	ID	ID
Pentachlorophenol	87865	1.0 (A)	1.0 (A)	(G,X)	NLV	NLV	200	1.85E+6	ID	ID
Pentane	109660	ID	ID	NA	38,000 (S)	38,000 (S)	ID	38,200	340	38,000 (S)
2-Pentene (I)	109682	ID	ID	NA	ID	ID	ID	2.03E+5	ID	ID
pH	NA	6.5 to 8.5 (E)	6.5 to 8.5 (E)	6.5 to 9.0	NA	NA	NA	NA	NA	NA
Phenanthrene	85018	52	150	2.4	1,000 (S)	1,000 (S)	1,000 (S)	1,000	ID	ID
Phenol	108952	4,400	13,000	210	NLV	NLV	2.9E+7	8.28E+7	NA	ID
Phosphorus (Total)	7723140	63,000	2.4E+5	(EE)	NLV	NLV	ID	NA	ID	ID
Phthalic acid	88993	14,000	40,000	NA	NLV	NLV	1.4E+7 (S)	1.42E+7	ID	ID
Phthalic anhydride	85449	15,000	44,000	NA	NLV	NLV	6.2E+6 (S)	6.2E+6	NA	ID
Picloram	1918021	500 (A)	500 (A)	46	NLV	NLV	4.3E+5 (S)	4.30E+5	ID	ID
Piperidine	110894	3.2	9.2	NA	NLV	NLV	34,000	1.0E+9	ID	ID
Polybrominated biphenyls (J)	67774327	0.03	0.09	NA	NLV	NLV	ID	1.66E+7	ID	ID
Polychlorinated biphenyls (PCBs) (J,T)	1336363	0.5 (A)	0.5 (A)	0.2 (M); 2.6E-5	45 (S)	45 (S)	3.3 (AA)	44.7	ID	ID

TABLE 1. GROUNDWATER: RESIDENTIAL AND INDUSTRIAL-COMMERCIAL PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

Guidesheet Number →		#1	#2	#3	#4	#5	#6	#7	#8	#9
	Chemical Abstract Service Number	**Residential & Commercial I Drinking Water Criteria & RBSLs**	**Industrial & Commercial II, III & IV Drinking Water Criteria & RBSLs**	**Groundwater Surface Water Interface Criteria & RBSLs**	**Residential & Commercial I Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Industrial & Commercial II, III & IV Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Groundwater Contact Criteria & RBSLs**	**Water Solubility**	**Flammability and Explosivity Screening Level**	**Acute Inhalation Screening Level**
Prometon	1610180	160	460	NA	NLV	NLV	1.8E+5	7.50E+5	ID	ID
Propachlor	1918167	95	270	NA	NLV	NLV	4.4E+5	6.55E+5	ID	ID
Propazine	139402	200	560	NA	NLV	NLV	8,600 (S)	8,600	ID	ID
Propionic acid	79094	12,000	35,000	ID	NLV	NLV	2.8E+8	1.0E+9	1.0E+9 (D)	ID
Propyl alcohol (I)	71238	1,400	4,000	NA	NLV	NLV	2.8E+7	1.0E+9	7.1E+7	1.0E+9 (S)
n-Propylbenzene (I)	103651	80	230	ID	ID	ID	15,000	NA	ID	ID
Propylene glycol	57556	1.5E+5	4.2E+5	2.9E+5	NLV	NLV	1.0E+9 (D,S)	1.0E+9	ID	ID
Pyrene	129000	140 (S)	140 (S)	ID	140 (S)	140 (S)	140 (S)	135	ID	ID
Pyridine (I)	110861	20 (M); 7.3	21	NA	5,500	12,000	94,000	3.00E+5	81,000	ID
Selenium (B)	7782492	50 (A)	50 (A)	5.0	NLV	NLV	9.7E+5	NA	ID	ID
Silver (B)	7440224	34	98	0.2 (M); 0.06	NLV	NLV	1.5E+6	NA	ID	ID
Silvex (2,4,5-TP)	93721	50 (A)	50 (A)	30	NLV	NLV	43,000	1.40E+5	ID	ID
Simazine	122349	4.0 (A)	4.0 (A)	NA	NLV	NLV	4,500 (S)	4,470	ID	ID
Sodium	17341252	1.2E+5	3.5E+5	NA	NLV	NLV	1.0E+9 (D)	NA	ID	ID
Sodium azide	26628228	88	250	NA	ID	ID	ID	NA	ID	ID
Strontium (B)	7440246	4,600	13,000	2,300 (X)	NLV	NLV	1.2E+8	NA	ID	ID
Styrene	100425	100 (A)	100 (A)	80	1.7E+5	3.1E+5 (S)	9,700	3.10E+5	1.4E+5	3.1E+5 (S)
Sulfate	14808798	2.5E+5 (E)	2.5E+5 (E)	NA	NLV	NLV	ID	NA	ID	ID
Tebuthiuron	34014181	510	1,500	NA	NLV	NLV	2.5E+6 (S)	2.50E+6	ID	ID
2,3,7,8-Tetrabromodibenzo-p-dioxin (O)	50585416	(O)	(O)	(O)	NLV	NLV	(O)	0.00996	ID	ID
1,2,4,5-Tetrachlorobenzene	95943	1,300 (S)	1,300 (S)	2.9 (X)	ID	ID	1,300 (S)	1,300	ID	ID
2,3,7,8-Tetrachlorodibenzo-p-dioxin (O)	1746016	3.0E-5 (A)	3.0E-5 (A)	1.0E-5 (M); 3E-9	NLV	NLV	1.0E-5 (M,O,AA)	0.019	ID	ID
1,1,1,2-Tetrachloroethane	630206	77	320	ID (X)	15,000	96,000	30,000	1.10E+6	ID	ID
1,1,2,2-Tetrachloroethane	79345	8.5	35	78 (X)	12,000	77,000	4,700	2.97E+6	ID	ID
Tetrachloroethylene	127184	5.0 (A)	5.0 (A)	45 (X)	25,000	1.7E+5	12,000	2.0E+5	ID	2.0E+5 (S)
Tetrahydrofuran	109999	95	270	11,000 (X)	6.9E+6	1.6E+7	1.6E+6	1.0E+9	60,000	3.6E+6

TABLE 1. GROUNDWATER: RESIDENTIAL AND INDUSTRIAL-COMMERCIAL PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

Guidesheet Number →		#1	#2	#3	#4	#5	#6	#7	#8	#9
	Chemical Abstract Service Number	**Residential & Commercial I Drinking Water Criteria & RBSLs**	**Industrial & Commercial II, III & IV Drinking Water Criteria & RBSLs**	**Groundwater Surface Water Interface Criteria & RBSLs**	**Residential & Commercial I Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Industrial & Commercial II, III & IV Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Groundwater Contact Criteria & RBSLs**	**Water Solubility**	**Flammability and Explosivity Screening Level**	**Acute Inhalation Screening Level**
Tetranitromethane	509148	ID	ID	NA	580	3,200	ID	85,000	ID	ID
Thallium (B)	7440280	2.0 (A)	2.0 (A)	3.7 (X)	NLV	NLV	13,000	NA	ID	ID
Toluene (I)	108883	790 (E)	790 (E)	140	5.3E+5 (S)	5.3E+5 (S)	5.3E+5 (S)	5.26E+5	61,000	ID
p-Toluidine	106490	15	62	NA	NLV	NLV	24,000	7.60E+6	NA	ID
Total dissolved solids (TDS)	NA	5.0E+5 (E)	5.0E+5 (E)	(EE)	NA	NA	NA	NA	NA	NA
Toxaphene	8001352	3.0 (A)	3.0 (A)	1.0 (M); 6.8E-5	NLV	NLV	44	740	ID	740 (S)
Triallate	2303175	95	270	NA	ID	ID	4,000 (S)	4,000	ID	ID
Tributylamine	102829	10	29	ID	14,000	32,000	2,300	75,400	ID	ID
1,2,4-Trichlorobenzene	120821	70 (A)	70 (A)	30	3.0E+5 (S)	3.0E+5 (S)	19,000	3.00E+5	NA	3.0E+5 (S)
1,1,1-Trichloroethane	71556	200 (A)	200 (A)	200	6.6E+5	1.3E+6 (S)	1.3E+6 (S)	1.33E+6	ID	1.3E+6 (S)
1,1,2-Trichloroethane	79005	5.0 (A)	5.0 (A)	330 (X)	17,000	1.1E+5	21,000	4.42E+6	NA	ID
Trichloroethylene	79016	5.0 (A)	5.0 (A)	200 (X)	15,000	97,000	22,000	1.10E+6	ID	1.1E+6 (S)
Trichlorofluoromethane	75694	2,600	7,300	NA	1.1E+6 (S)	1.1E+6 (S)	1.1E+6 (S)	1.10E+6	ID	1.1E+6 (S)
2,4,5-Trichlorophenol	95954	730	2,100	NA	NLV	NLV	1.7E+5	1.20E+6	ID	ID
2,4,6-Trichlorophenol	88062	120	470	4.4	NLV	NLV	10,000	8.00E+5	ID	ID
1,2,3-Trichloropropane	96184	42	120	NA	ID	ID	84,000	1.90E+6	NA	ID
1,1,2-Trichloro-1,2,2-trifluoroethane	76131	1.7E+5 (S)	1.7E+5 (S)	32	1.7E+5 (S)	1.7E+5 (S)	1.7E+5 (S)	1.70E+5	ID	1.7E+5 (S)
Triethanolamine	102716	3,700	10,000	NA	NLV	NLV	1.0E+9 (D,S)	1.0E+9	ID	ID
Triethylene glycol	112276	4,300	12,000	NA	NLV	NLV	1.0E+6 (S)	1.00E+6	ID	ID
3-Trifluoromethyl-4-nitrophenol	88302	4,500	13,000	NA	NLV	NLV	5.0E+6 (S)	5.00E+6	ID	ID
Trifluralin	1582098	37	110	NA	ID	ID	2,400	8,100	ID	ID
2,2,4-Trimethyl pentane	540841	ID	ID	NA	ID	ID	ID	2,330	160	ID
2,4,4-Trimethyl-2-pentene (I)	107404	ID	ID	NA	ID	ID	ID	11,900	ID	ID
1,2,4-Trimethylbenzene (I)	95636	63 (E)	63 (E)	17	56,000 (S)	56,000 (S)	56,000 (S)	55,890	56,000 (S)	ID
1,3,5-Trimethylbenzene (I)	108678	72 (E)	72 (E)	45	61,000 (S)	61,000 (S)	61,000 (S)	61,150	ID	ID
Triphenyl phosphate	115866	1,200	1,400 (S)	NA	NLV	NLV	1,400 (S)	1,430	ID	ID

TABLE 1. GROUNDWATER: RESIDENTIAL AND INDUSTRIAL-COMMERCIAL PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

Guidesheet Number →		#1	#2	#3	#4	#5	#6	#7	#8	#9
	Chemical Abstract Service Number	**Residential & Commercial I Drinking Water Criteria & RBSLs**	**Industrial & Commercial II, III & IV Drinking Water Criteria & RBSLs**	**Groundwater Surface Water Interface Criteria & RBSLs**	**Residential & Commercial I Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Industrial & Commercial II, III & IV Groundwater Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Groundwater Contact Criteria & RBSLs**	**Water Solubility**	**Flammability and Explosivity Screening Level**	**Acute Inhalation Screening Level**
tris(2,3-Dibromopropyl)phosphate	126727	10 (M); 0.71	10 (M); 2.9	NA	4,700 (S)	4,700 (S)	2,100	4,700	ID	ID
Urea	57136	ID (N)	ID (N)	NA	NLV	NLV	ID	NA	ID	ID
Vanadium	7440622	4.5	62	12	NLV	NLV	9.7E+5	NA	ID	ID
Vinyl acetate (I)	108054	640	1,800	NA	4.1E+6	8.9E+6	8.0E+6	2.00E+7	1.8E+6	4.8E+6
Vinyl chloride	75014	2.0 (A)	2.0 (A)	15	1,100	13,000	1,000	2.76E+6	33,000	ID
White phosphorus (R)	12185103	0.11	0.31	NA	NLV	NLV	2,900	NA	ID	ID
Xylenes (I)	1330207	280 (E)	280 (E)	35	1.9E+5 (S)	1.9E+5 (S)	1.9E+5 (S)	1.86E+5	70,000	1.9E+5 (S)
Zinc (B)	7440666	2,400	5,000 (E)	(G)	NLV	NLV	1.1E+8	NA	ID	ID
Water Quality Characteristic										
Dissolved oxygen (DO)	NA	NA	NA	(EE)	NA	NA	NA	NA	NA	NA

TABLE 2. SOIL: RESIDENTIAL AND COMMERCIAL I
PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS;
PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

All criteria, unless otherwise noted, are expressed in units of parts per billion (ppb). One ppb is equivalent to one microgram per kilogram (ug/kg). Criteria with six or more digits are expressed in scientific notation. For example, 200,000 ppb is presented as 2.0E+5. The lowest generic soil criterion for a given hazardous substance is presented in a bold box. A footnote is designated by a letter in parentheses and is explained in the footnote pages that follow the criteria tables. When the risk-based criterion is less than the target detection limit (TDL), the TDL is listed as the criterion (R 299.5707). In these cases, two numbers are presented in the cell. The first number is the criterion (i.e., TDL), and the second number is the risk-based value. Criteria were promulgated December 21, 2002 within the Administrative Rules for Part 201, Environmental Remediation, of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended. These tables reflect modifications to the TDLs and new criteria consistent with the provisions of R299.5103(l) and R299.5706a, respectively.

			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
Hazardous Substance	**Chemical Abstract Service Number**	**Statewide Default Background Levels**	**Drinking Water Protection Criteria & RBSLs**	**Groundwater Surface Water Interface Protection Criteria & RBSLs**	**Groundwater Contact Protection Criteria & RBSLs**	**Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs**	**Finite VSIC for 5 Meter Source Thickness**	**Finite VSIC for 2 Meter Source Thickness**	**Particulate Soil Inhalation Criteria & RBSLs**	**Direct Contact Criteria & RBSLs**	**Soil Saturation Concentration Screening Levels**
Acenaphthene	83329	NA	3.0E+5	4,400	9.7E+5	1.9E+8	8.1E+7	8.1E+7	8.1E+7	1.4E+10	4.1E+7	NA
Acenaphthylene	208968	NA	5,900	ID	4.4E+5	1.6E+6	2.2E+6	2.2E+6	2.2E+6	2.3E+9	1.6E+6	NA
Acetaldehyde (I)	75070	NA	19,000	2,600	1.1E+8 (C)	2.2E+5	1.7E+5	1.7E+5	2.8E+5	6.0E+8	2.9E+7	1.1E+8
Acetate	71501	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	ID
Acetic acid	64197	NA	84,000	3.6E+5	6.5E+8 (C)	NLV	NLV	NLV	NLV	1.7E+10	1.3E+8	6.5E+8
Acetone (I)	67641	NA	15,000	34,000	1.1E+8 (C)	1.1E+8 (C)	1.3E+8	1.3E+8	1.9E+8	3.9E+11	2.3E+7	1.1E+8
Acetonitrile	75058	NA	2,800	NA	2.2E+7 (C)	4.8E+6	1.6E+6	1.6E+6	2.1E+6	4.0E+9	4.3E+6	2.2E+7
Acetophenone	98862	NA	30,000	NA	1.1E+6 (C)	1.1E+6 (C)	4.4E+7	4.4E+7	4.4E+7	3.3E+10	1.1E+6 (C)	1.1E+6
Acrolein (I)	107028	NA	2,400	NA	2.3E+7 (C)	410	310	310	610	1.3E+6	3.6E+6	2.3E+7
Acrylamide	79061	NA	10	NA	2.6E+5	NLV	NLV	NLV	NLV	2.4E+6	1,900	NA
Acrylic acid	79107	NA	78,000	NA	1.1E+8 (C)	2.4E+6	1.9E+5	2.3E+5	2.3E+5	6.7E+7	3.5E+7 (DD)	1.1E+8
Acrylonitrile (I)	107131	NA	100 (M); 52	100 (M,X); 98	2.8E+5	6,600	5,000	5,100	10,000	4.6E+7	16,000	8.3E+6
Alachlor	15972608	NA	52	290 (X)	44,000	NLV	NLV	NLV	NLV	ID	93,000	NA
Aldicarb	116063	NA	60	NA	2.4E+6	NLV	NLV	NLV	NLV	ID	2.3E+5	NA
Aldicarb sulfoxide	1646873	NA	200 (M)	NA	5.4E+7	NLV	NLV	NLV	NLV	ID	2.9E+5	NA
Aldicarb sulfone	1646884	NA	200 (M); 40	NA	4.2E+7	NLV	NLV	NLV	NLV	ID	2.5E+5	NA
Aldrin	309002	NA	NLL	NLL	NLL	1.3E+6	58,000	58,000	58,000	6.4E+5	1,000	NA
Aluminum (B)	7429905	6.9E+6	1,000	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	5.0E+7 (DD)	NA
Ammonia	7664417	NA	ID	(CC)	ID	ID	ID	ID	ID	6.7E+9	ID	1.0E+7

TABLE 2. SOIL: RESIDENTIAL AND COMMERCIAL I
PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS;
PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Direct Contact Criteria & RBSLs	Soil Saturation Concentration Screening Levels
t-Amyl methyl ether (TAME)	994058	NA	3,900	NA	4.4E+5 (C)	58,000	3.4E+5	7.6E+5	1.8E+6	4.1E+9	4.4E+5 (C)	4.4E+5
Aniline	62533	NA	1,100	330 (M); 80	2.8E+6	NLV	NLV	NLV	NLV	6.7E+7	3.3E+5	4.5E+6
Anthracene	120127	NA	41,000	ID	41,000	1.0E+9 (D)	1.4E+9	1.4E+9	1.4E+9	6.7E+10	2.3E+8	NA
Antimony	7440360	NA	4,300	94,000	4.9E+7	NLV	NLV	NLV	NLV	1.3E+7	1.8E+5	NA
Arsenic	7440382	5,800	4,600	70,000 (X)	2.0E+6	NLV	NLV	NLV	NLV	7.2E+5	7,600	NA
Asbestos (BB)	1332214	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	1.0E+7 (M); 68,000	ID	NA
Atrazine	1912249	NA	60	150 (X)	1.1E+5	NLV	NLV	NLV	NLV	ID	71,000 (DD)	NA
Azobenzene	103333	NA	4,200	NA	3.0E+5	6.1E+6	6.3E+5	ID	ID	1.0E+8	1.4E+5	NA
Barium (B)	7440393	75,000	1.3E+6	(G,X)	1.0E+9 (D)	NLV	NLV	NLV	NLV	3.3E+8	3.7E+7	NA
Benzene (I)	71432	NA	100	4,000 (X)	2.2E+5	1,600	13,000	34,000	79,000	3.8E+8	1.8E+5	4.0E+5
Benzidine	92875	NA	1,000 (M); 6.0	ID	1,000 (M); 140	NLV	NLV	NLV	NLV	46,000	1,000 (M); 23	NA
Benzo(a)anthracene (Q)	56553	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	20,000	NA
Benzo(b)fluoranthene (Q)	205992	NA	NLL	NLL	NLL	ID	ID	ID	ID	ID	20,000	NA
Benzo(k)fluoranthene (Q)	207089	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	2.0E+5	NA
Benzo(g,h,i)perylene	191242	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	8.0E+8	2.5E+6	NA
Benzo(a)pyrene (Q)	50328	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	1.5E+6	2,000	NA
Benzoic acid	65850	NA	6.4E+5	NA	7.0E+7	NLV	NLV	NLV	NLV	ID	9.9E+8	NA
Benzyl alcohol	100516	NA	2.0E+5	NA	5.8E+6 (C)	NLV	NLV	NLV	NLV	3.3E+11	5.8E+6 (C)	5.8E+6
Benzyl chloride	100447	NA	150	NA	72,000	6,300	14,000	14,000	17,000	6.2E+7	48,000	2.3E+5
Beryllium	7440417	NA	51,000	(G)	1.0E+9 (D)	NLV	NLV	NLV	NLV	1.3E+6	4.1E+5	NA
bis(2-Chloroethoxy)ethane	112265	NA	ID	ID	ID	NLV	NLV	NLV	NLV	ID	ID	2.7E+6
bis(2-Chloroethyl)ether (I)	111444	NA	100	300	1.1E+5	8,300	3,800	3,800	3,800	9.4E+6	13,000	2.2E+6
bis(2-Ethylhexyl)phthalate	117817	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	7.0E+8	2.8E+6	1.0E+7
Boron (B)	7440428	NA	10,000	38,000	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	4.8E+7 (DD)	NA

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PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS;
PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Direct Contact Criteria & RBSLs	Soil Saturation Concentration Screening Levels
Bromate	15541454	NA	200	800	96,000	NLV	NLV	NLV	NLV	ID	17,000	NA
Bromobenzene (I)	108861	NA	550	NA	3.6E+5	3.1E+5	4.5E+5	4.5E+5	4.5E+5	5.3E+8	5.4E+5	7.6E+5
Bromodichloromethane	75274	NA	1,600 (W)	ID	2.8E+5	1,200	9,100	9,700	19,000	8.4E+7	1.1E+5	1.5E+6
Bromoform	75252	NA	1,600 (W)	ID	8.7E+5 (C)	1.5E+5	9.0E+5	9.0E+5	9.0E+5	2.8E+9	8.2E+5	8.7E+5
Bromomethane	74839	NA	200	700	1.4E+6	860	11,000	57,000	1.4E+5	3.3E+8	3.2E+5	2.2E+6
n-Butanol (I)	71363	NA	19,000	NA	8.7E+6 (C)	NLV	NLV	NLV	NLV	2.3E+10	8.7E+6 (C)	8.7E+6
2-Butanone (MEK) (I)	78933	NA	2.6E+5	44,000	2.7E+7 (C)	2.7E+7 (C)	2.9E+7	2.9E+7	3.5E+7	6.7E+10	2.7E+7 (C,DD)	2.7E+7
n-Butyl acetate	123864	NA	11,000	NA	1.1E+6 (C)	1.1E+6 (C)	1.1E+8	2.6E+8	3.2E+8	4.7E+11	1.1E+6 (C)	1.1E+6
t-Butyl alcohol	75650	NA	78,000	NA	1.1E+8 (C)	1.1E+8 (C)	9.7E+7	2.0E+8	2.0E+8	1.3E+11	1.1E+8 (C)	1.1E+8
Butyl benzyl phthalate	85687	NA	3.1E+5 (C)	26,000 (X)	3.1E+5 (C)	NLV	NLV	NLV	NLV	4.7E+10	3.1E+5 (C)	3.1E+5
n-Butylbenzene	104518	NA	1,600	ID	1.2E+5	ID	ID	ID	ID	ID	2.5E+6	1.0E+7
sec-Butylbenzene	135988	NA	1,600	ID	88,000	ID	ID	ID	ID	ID	2.5E+6	1.0E+7
t-Butylbenzene (I)	98066	NA	1,600	NA	1.8E+5	ID	ID	ID	ID	ID	2.5E+6	1.0E+7
Cadmium (B)	7440439	1,200	6,000	(G,X)	2.3E+8	NLV	NLV	NLV	NLV	1.7E+6	5.5E+5	NA
Camphene (I)	79925	NA	ID	NA	ID	ID	ID	ID	ID	ID	ID	NA
Caprolactam	105602	NA	1.2E+5	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	6.7E+8	5.3E+7 (DD)	NA
Carbaryl	63252	NA	14,000	NA	2.6E+6	ID	ID	ID	ID	ID	2.2E+7	NA
Carbazole	86748	NA	9,400	1,100	8.2E+5	NLV	NLV	NLV	NLV	ID	5.3E+5	NA
Carbofuran	1563662	NA	800	NA	6.8E+6	NLV	NLV	NLV	NLV	ID	1.1E+6	NA
Carbon disulfide (I,R)	75150	NA	16,000	ID	2.8E+5 (C)	76,000	1.3E+6	7.9E+6	1.9E+7	4.7E+10	2.8E+5 (C,DD)	2.8E+5
Carbon tetrachloride	56235	NA	100	900 (X)	92,000	190	3,500	12,000	28,000	1.3E+8	96,000	3.9E+5
Chlordane (J)	57749	NA	NLL	NLL	NLL	1.1E+7	1.2E+6	1.2E+6	1.2E+6	3.1E+7	31,000	NA
Chloride	16887006	NA	5.0E+6	2.5E+6 (X)	ID	NLV	NLV	NLV	NLV	ID	5.0E+5 (F)	NA
Chlorobenzene (I)	108907	NA	2,000	940	2.6E+5 (C)	1.2E+5	7.7E+5	9.9E+5	2.1E+6	4.7E+9	2.6E+5 (C)	2.6E+5

TABLE 2. SOIL: RESIDENTIAL AND COMMERCIAL I
PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS;
PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
Hazardous Substance	**Chemical Abstract Service Number**	**Statewide Default Background Levels**	**Drinking Water Protection Criteria & RBSLs**	**Groundwater Surface Water Interface Protection Criteria & RBSLs**	**Groundwater Contact Protection Criteria & RBSLs**	**Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs**	**Finite VSIC for 5 Meter Source Thickness**	**Finite VSIC for 2 Meter Source Thickness**	**Particulate Soil Inhalation Criteria & RBSLs**	**Direct Contact Criteria & RBSLs**	**Soil Saturation Concentration Screening Levels**
para-Chlorobenzenesulfonic acid	98668	NA	1.5E+05	NA	NA	ID	ID	ID	ID	ID	2.3E+8	ID
1-Chloro-1,1-difluoroethane	75683	NA	3.0E+5	NA	9.6E+5 (C)	9.6E+5 (C)	7.9E+7	5.6E+8	1.4E+9	3.3E+12	9.6E+5 (C)	9.6E+5
Chloroethane	75003	NA	8,600	ID	9.5E+5 (C)	9.5E+5 (C)	3.0E+7	1.2E+8	2.8E+8	6.7E+11	9.5E+5 (C)	9.5E+5
2-Chloroethyl vinyl ether	110758	NA	ID	NA	ID	ID	ID	ID	ID	ID	ID	1.9E+6
Chloroform	67663	NA	1,600 (W)	3,400 (X)	1.5E+6 (C)	7,200	45,000	1.2E+5	2.7E+5	1.3E+9	1.2E+6	1.5E+6
Chloromethane (I)	74873	NA	5,200	ID	1.1E+6 (C)	2,300	40,000	4.1E+5	1.0E+6	4.9E+9	1.1E+6 (C)	1.1E+6
4-Chloro-3-methylphenol	59507	NA	5,800	280	3.0E+6	NLV	NLV	NLV	NLV	ID	4.5E+6	NA
beta-Chloronaphthalene	91587	NA	6.2E+5	NA	2.3E+6	ID	ID	ID	ID	ID	5.6E+7	NA
2-Chlorophenol	95578	NA	900	440	1.9E+6	ID	ID	ID	ID	ID	1.4E+6	1.9E+7
o-Chlorotoluene (I)	95498	NA	3,300	NA	5.0E+5 (C)	2.7E+5	1.2E+6	2.9E+6	6.3E+6	4.7E+9	5.0E+5 (C)	5.0E+5
Chlorpyrifos	2921882	NA	17,000	1,500	8.4E+5	130	4,600	23,000	55,000	1.3E+8	1.1E+7	NA
Chromium (III) (B,H)	16065831	18,000 (total)	1.0E+9 (D)	(G,X)	1.0E+9 (D)	NLV	NLV	NLV	NLV	3.3E+8	7.9E+8	NA
Chromium (VI)	18540299	NA	30,000	3,300	1.4E+8	NLV	NLV	NLV	NLV	2.6E+5	2.5E+6	NA
Chrysene (Q)	218019	NA	NLL	NLL	NLL	ID	ID	ID	ID	ID	2.0E+6	NA
Cobalt	7440484	6,800	800	2,000	4.8E+7	NLV	NLV	NLV	NLV	1.3E+7	2.6E+6	NA
Copper (B)	7440508	32,000	5.8E+6	(G)	1.0E+9 (D)	NLV	NLV	NLV	NLV	1.3E+8	2.0E+7	NA
Cyanazine	21725462	NA	200	1,100 (X)	56,000	NLV	NLV	NLV	NLV	ID	14,000	NA
Cyanide (P,R)	57125	390 (total)	4,000	100	2.5E+5	NLV	NLV	NLV	NLV	2.5E+5	12,000	NA
Cyclohexanone	108941	NA	5.2E+6	NA	2.2E+8 (C)	17,000	1.0E+6	1.1E+7	2.7E+7	6.7E+10	2.2E+8 (C)	2.2E+8
Dacthal	1861321	NA	50,000	NA	3.4E+5	NLV	NLV	NLV	NLV	ID	2.3E+6	NA
Dalapon	75990	NA	4,000	NA	5.9E+7 (C)	NLV	NLV	NLV	NLV	ID	1.9E+7	5.9E+7
4-4'-DDD	72548	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	4.4E+7	95,000	NA
4-4'-DDE	72559	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	3.2E+7	45,000	NA
4-4'-DDT	50293	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	3.2E+7	57,000	NA

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			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Direct Contact Criteria & RBSLs	Soil Saturation Concentration Screening Levels
Decabromodiphenyl ether	1163195	NA	1.4E+5	NA	1.4E+5	1.0E+9 (D)	8.6E+7	8.6E+7	8.6E+7	2.3E+9	3.8E+6	NA
Di-n-butyl phthalate	84742	NA	7.6E+5 (C)	11,000	7.6E+5 (C)	NLV	NLV	NLV	NLV	3.3E+9	7.6E+5 (C)	7.6E+5
Di(2-ethylhexyl) adipate	103231	NA	9.6E+5 (C)	NA	9.6E+5 (C)	NLV	NLV	NLV	NLV	9.2E+9	9.6E+5 (C,DD)	9.6E+5
Di-n-octyl phthalate	117840	NA	1.0E+8	ID	1.4E+8 (C)	NLV	NLV	NLV	NLV	ID	6.9E+6	1.4E+8
Diacetone alcohol (I)	123422	NA	ID	NA	ID	NLV	NLV	NLV	NLV	1.6E+11	ID	1.1E+8
Diazinon	333415	NA	95	NA	95,000	NLV	NLV	NLV	NLV	ID	12,000 (DD)	3.1E+5
Dibenzo(a,h)anthracene (Q)	53703	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	2,000	NA
Dibenzofuran	132649	NA	ID	1,700	ID	ID	ID	ID	ID	ID	ID	NA
Dibromochloromethane	124481	NA	1,600 (W)	ID	3.6E+5	3,900	24,000	24,000	33,000	1.3E+8	1.1E+5	6.1E+5
Dibromochloropropane	96128	NA	10 (M); 4.0	NA	1,200 (C)	1,200 (C)	13,000	13,000	13,000	1.3E+7	1,200 (C)	1,200
Dibromomethane	74953	NA	1,600	NA	2.0E+6 (C)	ID	ID	ID	ID	ID	2.0E+6 (C)	2.0E+6
Dicamba	1918009	NA	4,400	NA	1.2E+7	NA	NLV	NLV	NLV	ID	3.4E+6	NA
1,2-Dichlorobenzene	95501	NA	14,000	360	2.1E+5 (C)	2.1E+5 (C)	3.9E+7	3.9E+7	5.2E+7	1.0E+11	2.1E+5 (C)	2.1E+5
1,3-Dichlorobenzene	541731	NA	170	1,100	51,000	ID	ID	ID	ID	ID	1.7E+5 (C)	1.7E+5
1,4-Dichlorobenzene	106467	NA	1,700	290	1.4E+5	19,000	77,000	77,000	1.1E+5	4.5E+8	4.0E+5	NA
3,3'-Dichlorobenzidine	91941	NA	2,000 (M); 28	2,000 (M,X); 510	4,600	NLV	NLV	NLV	NLV	6.5E+6	6,600	NA
Dichlorodifluoromethane	75718	NA	95,000	ID	1.0E+6 (C)	9.0E+5	5.3E+7	5.5E+8	1.4E+9	3.3E+12	1.0E+6 (C)	1.0E+6
1,1-Dichloroethane	75343	NA	18,000	15,000	8.9E+5 (C)	2.3E+5	2.1E+6	5.9E+6	1.4E+7	3.3E+10	8.9E+5 (C)	8.9E+5
1,2-Dichloroethane (I)	107062	NA	100	7,200 (X)	3.8E+5	2,100	6,200	11,000	26,000	1.2E+8	91,000	1.2E+6
1,1-Dichloroethylene (I)	75354	NA	140	1,300 (X)	2.2E+5	62	1,100	5,300	13,000	6.2E+7	2.0E+5	5.7E+5
cis-1,2-Dichloroethylene	156592	NA	1,400	12,000	6.4E+5 (C)	22,000	1.8E+5	4.2E+5	9.9E+5	2.3E+9	6.4E+5 (C)	6.4E+5
trans-1,2-Dichloroethylene	156605	NA	2,000	30,000	1.4E+6 (C)	23,000	2.8E+5	8.3E+5	2.0E+6	4.7E+9	1.4E+6 (C)	1.4E+6
2,6-Dichloro-4-nitroaniline	99309	NA	44,000	NA	1.4E+5	NLV	NLV	NLV	NLV	ID	6.8E+7	NA
2,4-Dichlorophenol	120832	NA	1,500	380	9.6E+5	NLV	NLV	NLV	NLV	5.1E+9	6.6E+5 (DD)	1.8E+6

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			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
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2,4-Dichlorophenoxyacetic acid	94757	NA	1,400	4,400	2.4E+6	NLV	NLV	NLV	NLV	6.7E+9	2.5E+6	NA
1,2-Dichloropropane (I)	78875	NA	100	5,800 (X)	3.2E+5	4,000	25,000	50,000	1.1E+5	2.7E+8	1.4E+5	5.5E+5
1,3-Dichloropropene	542756	NA	170	NA	1.1E+5	1,000	18,000	68,000	1.6E+5	7.8E+8	10,000	6.2E+5
Dichlorovos	62737	NA	50 (M); 32	NA	1.2E+5	NLV	NLV	NLV	NLV	3.3E+7	10,000	2.2E+6
Dicyclohexyl phthalate	84617	NA	ID	NA	ID	ID	ID	ID	ID	ID	ID	NA
Dieldrin	60571	NA	NLL	NLL	NLL	1.4E+5	19,000	19,000	19,000	6.8E+5	1,100	NA
Diethyl ether	60297	NA	200	ID	7.4E+6 (C)	7.4E+6 (C)	8.5E+7	1.5E+8	3.4E+8	8.0E+11	7.4E+6 (C)	7.4E+6
Diethyl phthalate	84662	NA	1.1E+5	2,200	7.4E+5 (C)	NLV	NLV	NLV	NLV	3.3E+9	7.4E+5 (C)	7.4E+5
Diethylene glycol monobutyl ether	112345	NA	1,800	NA	8.0E+7	NLV	NLV	NLV	NLV	1.3E+9	2.7E+6	1.1E+8
Diisopropyl ether	108203	NA	600	ID	1,300 (C)	1,300 (C)	3.4E+5	7.6E+5	1.8E+6	4.1E+9	1,300 (C)	1,300
Diisopropylamine (I)	108189	NA	110	NA	4.2E+5	ID	ID	ID	ID	ID	1.7E+5	6.7E+6
Dimethyl phthalate	131113	NA	7.9E+5 (C)	NA	7.9E+5 (C)	NLV	NLV	NLV	NLV	3.3E+9	7.9E+5 (C)	7.9E+5
N,N-Dimethylacetamide	127195	NA	3,600	82,000 (X)	1.1E+8 (C)	NLV	NLV	NLV	NLV	ID	5.6E+6	1.1E+8
N,N-Dimethylaniline	121697	NA	320	NA	4.0E+5	1.7E+5	1.5E+5	1.5E+5	1.5E+5	2.6E+8	5.0E+5	8.0E+5
Dimethylformamide (I)	68122	NA	14,000	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	2.0E+9	2.2E+7	1.1E+8
2,4-Dimethylphenol	105679	NA	7,400	7,600	1.0E+7	NLV	NLV	NLV	NLV	4.7E+9	1.1E+7	NA
2,6-Dimethylphenol	576261	NA	330 (M); 88	NA	1.3E+5	NLV	NLV	NLV	NLV	ID	1.4E+5	NA
3,4-Dimethylphenol	95658	NA	330 (M); 200	NA	3.6E+5	NLV	NLV	NLV	NLV	ID	3.2E+5	NA
Dimethylsulfoxide	67685	NA	4.4E+6	3.8E+6	1.8E+7 (C)	NLV	NLV	NLV	NLV	ID	1.8E+7 (C)	1.8E+7
2,4-Dinitrotoluene	121142	NA	430	NA	1.7E+5	NLV	NLV	NLV	NLV	1.6E+7	48,000	NA
Dinoseb	88857	NA	300	200 (M); 43	1.4E+5 (C)	NLV	NLV	NLV	NLV	ID	66,000 (DD)	1.4E+5
1,4-Dioxane (I)	123911	NA	1,700	56,000	3.4E+7	NLV	NLV	NLV	NLV	5.7E+8	5.3E+5	9.7E+7
Diquat	85007	NA	400	NA	1.4E+7	NLV	NLV	NLV	NLV	ID	5.0E+5	NA
Diuron	330541	NA	620	NA	7.4E+5	NLV	NLV	NLV	NLV	4.7E+8	9.7E+5	NA

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Endosulfan (J)	115297	NA	NLL	NLL	NLL	ID	ID	ID	ID	ID	1.4E+6	NA
Endothall	145733	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	2.3E+9	3.8E+6	NA
Endrin	72208	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	65,000	NA
Epichlorohydrin (I)	106898	NA	100	NA	2.2E+5	64,000	31,000	31,000	35,000	6.7E+7	8,900	7.3E+6
Ethanol (I)	64175	NA	3.8E+7	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	1.3E+12	1.1E+8 (C,DD)	1.1E+8
Ethyl acetate (I)	141786	NA	1.3E+5	NA	7.5E+6 (C)	7.5E+6 (C)	4.9E+7	4.9E+7	9.8E+7	2.1E+11	7.5E+6 (C)	7.5E+6
Ethyl-tert-butyl ether (ETBE)	637923	NA	980	ID	ID	5.4E+5	1.9E+6	4.5E+6	1.1E+7	2.5E+10	ID	6.5E+5
Ethylbenzene (I)	100414	NA	1,500	360	1.4E+5 (C)	87,000	7.2E+5	1.0E+6	2.2E+6	1.0E+10	1.4E+5 (C)	1.4E+5
Ethylene dibromide	106934	NA	20 (M); 1.0	20 (M); 4.0	500	670	1,700	1,700	3,300	1.4E+7	92	8.9E+5
Ethylene glycol	107211	NA	3.0E+5	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	6.7E+10	1.1E+8 (C)	1.1E+8
Ethylene glycol monobutyl ether	111762	NA	74,000	NA	4.1E+7 (C)	7.4E+5	1.8E+7	1.5E+8	3.6E+8	8.7E+11	4.1E+7 (C)	4.1E+7
Fluoranthene	206440	NA	7.3E+5	5,500	7.3E+5	1.0E+9 (D)	7.4E+8	7.4E+8	7.4E+8	9.3E+9	4.6E+7	NA
Fluorene	86737	NA	3.9E+5	5,300	8.9E+5	5.8E+8	1.3E+8	1.3E+8	1.3E+8	9.3E+9	2.7E+7	NA
Fluorine (soluble fluoride) (B)	7782414	NA	40,000	NA	2.4E+8	NLV	NLV	NLV	NLV	ID	9.0E+6 (DD)	NA
Formaldehyde	50000	NA	26,000	2,400	6.0E+7 (C)	12,000	13,000	23,000	52,000	2.4E+8	4.1E+7	6.0E+7
Formic acid (I,U)	64186	NA	2.0E+5	ID	1.1E+8 (C)	1.5E+6	2.1E+5	1.4E+5	1.4E+5	1.3E+8	1.1E+8 (C)	1.1E+8
1-Formylpiperidine	2591868	NA	1,600	NA	ID	ID	ID	ID	ID	ID	2.5E+6	1.0E+7
Gentian violet	548629	NA	300	NA	2.0E+7	NLV	NLV	NLV	NLV	ID	96,000	NA
Glyphosate	1071836	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	1.1E+7 (DD)	NA
Heptachlor	76448	NA	NLL	NLL	NLL	3.5E+5	62,000	62,000	62,000	2.4E+6	5,600	NA
Heptachlor epoxide	1024573	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	1.2E+6	3,100	NA
n-Heptane	142825	NA	2.4E+5 (C)	NA	2.4E+5 (C)	2.4E+5 (C)	2.1E+7	4.4E+7	1.0E+8	2.3E+11	2.4E+5 (C)	2.4E+5
Hexabromobenzene	87821	NA	5,400	ID	5,400	ID	ID	ID	ID	ID	1.1E+6	NA
Hexachlorobenzene (C-66)	118741	NA	1,800	350	8,200	41,000	17,000	17,000	17,000	6.8E+6	8,900	NA

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PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
Hazardous Substance	**Chemical Abstract Service Number**	**Statewide Default Background Levels**	**Drinking Water Protection Criteria & RBSLs**	**Groundwater Surface Water Interface Protection Criteria & RBSLs**	**Groundwater Contact Protection Criteria & RBSLs**	**Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs**	**Finite VSIC for 5 Meter Source Thickness**	**Finite VSIC for 2 Meter Source Thickness**	**Particulate Soil Inhalation Criteria & RBSLs**	**Direct Contact Criteria & RBSLs**	**Soil Saturation Concentration Screening Levels**
Hexachlorobutadiene (C-46)	87683	NA	26,000	91	3.5E+5 (C)	1.3E+5	1.3E+5	1.3E+5	1.3E+5	1.4E+8	1.0E+5	3.5E+5
alpha-Hexachlorocyclohexane	319846	NA	18	NA	2,500	30,000	12,000	22,000	25,000	1.7E+6	2,600	NA
beta-Hexachlorocyclohexane	319857	NA	37	ID	5,100	NLV	NLV	NLV	NLV	5.9E+6	5,400	NA
Hexachlorocyclopentadiene (C-56)	77474	NA	3.2E+5	ID	7.2E+5 (C)	30,000	50,000	50,000	50,000	1.3E+7	7.2E+5 (C)	7.2E+5
Hexachloroethane	67721	NA	430	1,800 (X)	1.1E+5	40,000	5.5E+5	9.3E+5	9.3E+5	2.3E+8	2.3E+5	NA
n-Hexane	110543	NA	44,000 (C)	NA	44,000 (C)	44,000 (C)	3.0E+6	3.2E+6	6.2E+6	1.3E+10	44,000 (C)	44,000
2-Hexanone	591786	NA	20,000	NA	2.5E+6 (C)	9.9E+5	1.1E+6	1.1E+6	1.4E+6	2.7E+9	2.5E+6 (C)	2.5E+6
Indeno(1,2,3-cd)pyrene (Q)	193395	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	20,000	NA
Iron (B)	7439896	1.2E+7	6,000	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	1.6E+8	NA
Isobutyl alcohol (I)	78831	NA	46,000	NA	8.9E+6 (C)	8.9E+6 (C)	7.9E+7	7.9E+7	7.9E+7	1.0E+11	8.9E+6 (C)	8.9E+6
Isophorone	78591	NA	15,000	11,000 (X)	2.4E+6 (C)	NLV	NLV	NLV	NLV	1.2E+10	2.4E+6 (C)	2.4E+6
Isopropyl alcohol (I)	67630	NA	9,400	1.1E+6 (X)	1.1E+8 (C)	NLV	NLV	NLV	NLV	1.5E+10	1.4E+7	1.1E+8
Isopropyl benzene	98828	NA	91,000	ID	3.9E+5 (C)	3.9E+5 (C)	1.7E+6	1.7E+6	2.8E+6	5.8E+9	3.9E+5 (C)	3.9E+5
Lead (B)	7439921	21,000	7.0E+5	(G,X)	ID	NLV	NLV	NLV	NLV	1.0E+8	4.0E+5	NA
Lindane	58899	NA	20 (M); 7.0	20 (M); 0.99	7,100	ID	ID	ID	ID	ID	8,300	NA
Lithium (B)	7439932	9,800	3,400	1,900	1.1E+8	NLV	NLV	NLV	NLV	ID	4.2E+6 (DD)	NA
Magnesium (B)	7439954	NA	8.0E+6	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	6.7E+9	1.0E+9 (D)	NA
Manganese (B)	7439965	4.4E+5	1,000	(G,X)	1.8E+8	NLV	NLV	NLV	NLV	3.3E+6	2.5E+7	NA
Mercury (Total) (B,Z)	Varies	130	1,700	50 (M); 1.2	47,000	48,000	52,000	52,000	52,000	2.0E+7	1.6E+5	NA
Methane	74828	NA	ID	NA	ID	8.4E+6 ug/m3 (GG)	ID	ID	ID	ID	ID	ID
Methanol	67561	NA	74,000	9,600	3.1E+6 (C)	3.1E+6 (C)	3.1E+7	4.4E+7	9.6E+7	2.2E+11	3.1E+6 (C)	3.1E+6
Methoxychlor	72435	NA	16,000	NA	18,000	ID	ID	ID	ID	ID	1.9E+6	NA
2-Methoxyethanol (I)	109864	NA	150	NA	1.7E+7	NLV	NLV	NLV	NLV	1.3E+9	2.3E+5	1.1E+8
2-Methyl-4-chlorophenoxyacetic acid	94746	NA	390	NA	4.9E+5	NLV	NLV	NLV	NLV	ID	2.3E+5	NA

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			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
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2-Methyl-4,6-dinitrophenol	534521	NA	830 (M); 400	NA	1.9E+5	NLV	NLV	NLV	NLV	ID	79,000	NA
N-Methyl-morpholine (I)	109024	NA	400	NA	3.0E+7	NLV	NLV	NLV	NLV	ID	6.1E+5	1.1E+8
Methyl parathion	298000	NA	46	NA	76,000	NLV	NLV	NLV	NLV	ID	56,000	NA
4-Methyl-2-pentanone (MIBK) (I)	108101	NA	36,000	ID	2.7E+6 (C)	2.7E+6 (C)	4.5E+7	4.5E+7	6.7E+7	1.4E+11	2.7E+6 (C)	2.7E+6
Methyl-tert-butyl ether (MTBE)	1634044	NA	800	15,000 (X)	5.9E+6 (C)	5.9E+6 (C)	2.5E+7	3.9E+7	8.7E+7	2.0E+11	1.5E+6	5.9E+6
Methylcyclopentane (I)	96377	NA	ID	NA	ID	ID	ID	ID	ID	ID	ID	3.5E+5
4,4'-Methylene-bis-2-chloroaniline (MBOCA)	101144	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	8.4E+7	6,800	NA
Methylene chloride	75092	NA	100	19,000 (X)	2.3E+6 (C)	45,000	2.1E+5	5.9E+5	1.4E+6	6.6E+9	1.3E+6	2.3E+6
2-Methylnaphthalene	91576	NA	57,000	ID	5.5E+6	ID	ID	ID	ID	ID	8.1E+6	NA
Methylphenols (J)	1319773	NA	7,400	1,400	1.6E+7	NLV	NLV	NLV	NLV	6.7E+9	1.1E+7	NA
Metolachlor	51218452	NA	4,800	NA	4.4E+5 (C)	NLV	NLV	NLV	NLV	ID	4.4E+5 (C,DD)	4.4E+5
Metribuzin	21087649	NA	3,600	NA	2.4E+7	ID	ID	ID	ID	ID	9.6E+6	NA
Mirex	2385855	NA	NLL	NLL	NLL	ID	ID	ID	ID	ID	9,600	NA
Molybdenum (B)	7439987	NA	1,500	16,000 (X)	1.9E+7	NLV	NLV	NLV	NLV	ID	2.6E+6	NA
Naphthalene	91203	NA	35,000	870	2.1E+6	2.5E+5	3.0E+5	3.0E+5	3.0E+5	2.0E+8	1.6E+7	NA
Nickel (B)	7440020	20,000	1.0E+5	(G)	1.0E+9 (D)	NLV	NLV	NLV	NLV	1.3E+7	4.0E+7	NA
Nitrate (B,N)	14797558	NA	2.0E+5 (N)	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	ID	NA
Nitrite (B,N)	14797650	NA	20,000 (N)	NA	3.8E+8	NLV	NLV	NLV	NLV	ID	ID	NA
Nitrobenzene (I)	98953	NA	330 (M); 68	3,600 (X)	2.2E+5	91,000	54,000	54,000	54,000	4.7E+7	1.0E+5	4.9E+5
2-Nitrophenol	88755	NA	400	ID	1.6E+6	NLV	NLV	NLV	NLV	ID	6.3E+5	NA
n-Nitroso-di-n-propylamine	621647	NA	330 (M); 100	NA	7,200	NLV	NLV	NLV	NLV	1.6E+6	1,200	1.5E+6
N-Nitrosodiphenylamine	86306	NA	5,400	NA	7.0E+5	NLV	NLV	NLV	NLV	ID	1.7E+6	NA
Oxamyl	23135220	NA	4,000	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	8.6E+6	NA
Oxo-hexyl acetate	88230357	NA	1,500	NA	ID	ID	ID	ID	ID	5.4E+9	2.3E+6	1.0E+7

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			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
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Pendimethalin	40487421	NA	1.1E+6	NA	1.1E+6	NLV	NLV	NLV	NLV	ID	4.6E+7	NA
Pentachlorobenzene	608935	NA	29,000	9,500	1.9E+5 (C)	ID	ID	ID	ID	ID	1.9E+5 (C)	1.9E+5
Pentachloronitrobenzene	82688	NA	37,000	NA	37,000	1.2E+5	2.3E+5	2.3E+5	2.3E+5	3.3E+8	1.7E+6	NA
Pentachlorophenol	87865	NA	22	(G,X)	4,300	NLV	NLV	NLV	NLV	1.0E+8	90,000	NA
Pentane	109660	NA	ID	NA	ID	2.4E+5 (C)	3.7E+7	3.1E+8	5.8E+8	1.2E+12	ID	2.4E+5
2-Pentene (I)	109682	NA	ID	NA	ID	ID	ID	ID	ID	ID	ID	2.2E+5
Phenanthrene	85018	NA	56,000	5,300	1.1E+6	2.8E+6	1.6E+5	1.6E+5	1.6E+5	6.7E+6	1.6E+6	NA
Phenol	108952	NA	88,000	4,200	1.2E+7 (C)	NLV	NLV	NLV	NLV	4.0E+10	1.2E+7 (C,DD)	1.2E+7
Phosphorus (Total)	7723140	NA	1.3E+6	(EE)	ID	NLV	NLV	NLV	NLV	ID	1.0E+9 (D)	NA
Phthalic acid	88993	NA	2.8E+5	NA	1.7E+6 (C)	NLV	NLV	NLV	NLV	ID	1.7E+6 (C)	1.7E+6
Phthalic anhydride	85449	NA	3.0E+5	NA	1.1E+6 (C)	NLV	NLV	NLV	NLV	ID	1.1E+6 (C)	1.1E+6
Picloram	1918021	NA	10,000	920	8.6E+6	NLV	NLV	NLV	NLV	ID	1.6E+7	NA
Piperidine	110894	NA	64	NA	6.8E+5	NLV	NLV	NLV	NLV	9.3E+9	99,000	1.2E+8
Polybrominated biphenyls (J)	67774327	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	1,200	NA
Polychlorinated biphenyls (PCBs) (J,T)	1336363	NA	NLL	NLL	NLL	3.0E+6	2.4E+5	7.9E+6	7.9E+6	5.2E+6	(T)	NA
Prometon	1610180	NA	4,900	NA	5.5E+6	NLV	NLV	NLV	NLV	ID	5.0E+6	NA
Propachlor	1918167	NA	1,900	NA	8.8E+6	NLV	NLV	NLV	NLV	ID	2.9E+6	NA
Propazine	139402	NA	4,000	NA	1.7E+5	NLV	NLV	NLV	NLV	ID	6.1E+6	NA
Propionic acid	79094	NA	2.4E+5	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	2.0E+10	1.1E+8 (C)	1.1E+8
Propyl alcohol (I)	71238	NA	28,000	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	4.9E+10	1.3E+7 (DD)	1.1E+8
n-Propylbenzene (I)	103651	NA	1,600	NA	3.0E+5	ID	ID	ID	ID	1.3E+9	2.5E+6	1.0E+7
Propylene glycol	57556	NA	3.0E+6	5.8E+6	1.1E+8 (C)	NLV	NLV	NLV	NLV	4.0E+11	1.1E+8 (C)	1.1E+8
Pyrene	129000	NA	4.8E+5	ID	4.8E+5	1.0E+9 (D)	6.5E+8	6.5E+8	6.5E+8	6.7E+9	2.9E+7	NA
Pyridine (I)	110861	NA	400	NA	37,000 (C)	1,100	8,200	40,000	97,000	2.3E+8	37,000 (C)	37,000

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Selenium (B)	7782492	410	4,000	400	7.8E+7	NLV	NLV	NLV	NLV	1.3E+8	2.6E+6	NA
Silver (B)	7440224	1,000	4,500	100 (M); 27	2.0E+8	NLV	NLV	NLV	NLV	6.7E+6	2.5E+6	NA
Silvex (2,4,5-TP)	93721	NA	3,600	2,200	3.1E+6	NLV	NLV	NLV	NLV	ID	1.7E+6	NA
Simazine	122349	NA	80	NA	90,000	NLV	NLV	NLV	NLV	ID	1.2E+6	NA
Sodium	17341252	NA	2.5E+6	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	1.0E+9 (D)	NA
Sodium azide	26628228	NA	1,800	NA	ID	ID	ID	ID	ID	ID	2.7E+6	NA
Strontium (B)	7440246	NA	92,000	46,000 (X)	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	3.3E+8	NA
Styrene	100425	NA	2,700	2,200	2.7E+5	2.5E+5	9.7E+5	9.7E+5	1.4E+6	5.5E+9	4.0E+5	5.2E+5
Sulfate	14808798	NA	5.0E+6	NA	ID	NLV	NLV	NLV	NLV	ID	ID	NA
Tebuthiuron	34014181	NA	10,000	NA	5.0E+7	NLV	NLV	NLV	NLV	ID	4.6E+6 (DD)	NA
2,3,7,8-Tetrabromodibenzo-p-dioxin (O)	50585416	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	(O)	(O)	NA
1,2,4,5-Tetrachlorobenzene	95943	NA	1.5E+6	3,400 (X)	1.5E+6	ID	ID	ID	ID	ID	7.7E+7	NA
2,3,7,8-Tetrachlorodibenzo-p-dioxin (O)	1746016	NA	NLL	NLL	NLL	NLV	NLV	NLV	NLV	71 (O)	0.09 (O)	NA
1,1,1,2-Tetrachloroethane	630206	NA	1,500	ID (X)	4.4E+5 (C)	6,200	36,000	54,000	1.0E+5	4.2E+8	4.4E+5 (C)	4.4E+5
1,1,2,2-Tetrachloroethane	79345	NA	170	1,600 (X)	94,000	4,300	10,000	10,000	14,000	5.4E+7	53,000	8.7E+5
Tetrachloroethylene	127184	NA	100	900 (X)	88,000 (C)	11,000	1.8E+5	4.8E+5	1.1E+6	5.4E+9	88,000 (C)	88,000
Tetrahydrofuran	109999	NA	1,900	2.2E+5 (X)	3.2E+7	1.3E+6	1.3E+7	6.7E+7	1.6E+8	3.9E+11	2.9E+6	1.2E+8
Tetranitromethane	509148	NA	ID	ID	ID	500 (M); 110	500 (M); 51	ID	ID	2.1E+5	ID	ID
Thallium (B)	7440280	NA	2,300	4,200 (X)	1.5E+7	NLV	NLV	NLV	NLV	ID	35,000	NA
Toluene (I)	108883	NA	16,000	2,800	2.5E+5 (C)	2.5E+5 (C)	2.8E+6	5.1E+6	1.2E+7	2.7E+10	2.5E+5 (C)	2.5E+5
p-Toluidine	106490	NA	660 (M); 300	NA	4.8E+5	NLV	NLV	NLV	NLV	1.0E+8	94,000	1.2E+6
Toxaphene	8001352	NA	24,000	860	3.6E+5	NLV	NLV	NLV	NLV	9.7E+6	20,000	NA
Triallate	2303175	NA	95,000	NA	2.5E+5 (C)	ID	ID	ID	ID	ID	2.5E+5 (C)	2.5E+5
Tributylamine	102829	NA	7,800	ID	1.8E+6	5.8E+5	6.0E+5	6.0E+5	6.0E+5	4.7E+8	7.9E+5	3.7E+6

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Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
Hazardous Substance	**Chemical Abstract Service Number**	**Statewide Default Background Levels**	**Drinking Water Protection Criteria & RBSLs**	**Groundwater Surface Water Interface Protection Criteria & RBSLs**	**Groundwater Contact Protection Criteria & RBSLs**	**Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs**	**Finite VSIC for 5 Meter Source Thickness**	**Finite VSIC for 2 Meter Source Thickness**	**Particulate Soil Inhalation Criteria & RBSLs**	**Direct Contact Criteria & RBSLs**	**Soil Saturation Concentration Screening Levels**
1,2,4-Trichlorobenzene	120821	NA	4,200	1,800	1.1E+6	1.1E+6 (C)	2.8E+7	2.8E+7	2.8E+7	2.5E+10	9.9E+5 (DD)	1.1E+6
1,1,1-Trichloroethane	71556	NA	4,000	4,000	4.6E+5 (C)	2.5E+5	3.8E+6	1.2E+7	2.8E+7	6.7E+10	4.6E+5 (C)	4.6E+5
1,1,2-Trichloroethane	79005	NA	100	6,600 (X)	4.2E+5	4,600	17,000	21,000	44,000	1.9E+8	1.8E+5	9.2E+5
Trichloroethylene	79016	NA	100	4,000 (X)	4.4E+5	7,100	78,000	1.7E+5	3.9E+5	1.8E+9	5.0E+5 (C,DD)	5.0E+5
Trichlorofluoromethane	75694	NA	52,000	NA	5.6E+5 (C)	5.6E+5 (C)	9.2E+7	6.3E+8	1.5E+9	3.8E+12	5.6E+5 (C)	5.6E+5
2,4,5-Trichlorophenol	95954	NA	39,000	NA	9.1E+6	NLV	NLV	NLV	NLV	2.3E+10	2.3E+7	NA
2,4,6-Trichlorophenol	88062	NA	2,400	NA	2.0E+5	NLV	NLV	NLV	NLV	1.0E+9	7.1E+5	NA
1,2,3-Trichloropropane	96184	NA	840	NA	8.3E+5 (C)	ID	ID	ID	ID	ID	8.3E+5 (C)	8.3E+5
1,1,2-Trichloro-1,2,2-trifluoroethane	76131	NA	5.5E+5 (C)	1,700	5.5E+5 (C)	5.5E+5 (C)	1.8E+8	8.8E+8	2.1E+9	5.1E+12	5.5E+5 (C)	5.5E+5
Triethanolamine	102716	NA	74,000	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	3.3E+9	1.1E+8	1.1E+8
Triethylene glycol	112276	NA	1.1E+5 (C)	NA	1.1E+5 (C)	NLV	NLV	NLV	NLV	ID	1.1E+5 (C,DD)	1.1E+5
3-Trifluoromethyl-4-nitrophenol	88302	NA	1.1E+5	NA	1.2E+8	NLV	NLV	NLV	NLV	ID	4.1E+7 (DD)	NA
Trifluralin	1582098	NA	1.9E+5	NA	1.2E+7	ID	ID	ID	ID	ID	2.0E+6	NA
2,2,4-Trimethyl pentane	540841	NA	ID	NA	ID	ID	ID	ID	ID	ID	ID	19,000
2,4,4-Trimethyl-2-pentene (I)	107404	NA	ID	NA	ID	ID	ID	ID	ID	ID	ID	56,000
1,2,4-Trimethylbenzene (I)	95636	NA	2,100	570	1.1E+5 (C)	1.1E+5 (C)	2.1E+7	5.0E+8	5.0E+8	8.2E+10	1.1E+5 (C)	1.1E+5
1,3,5-Trimethylbenzene (I)	108678	NA	1,800	1,100	94,000 (C)	94,000 (C)	1.6E+7	3.8E+8	3.8E+8	8.2E+10	94,000 (C)	94,000
Triphenyl phosphate	115866	NA	1.1E+5 (C)	NA	1.1E+5 (C)	NLV	NLV	NLV	NLV	ID	1.1E+5 (C)	1.1E+5
tris(2,3-Dibromopropyl)phosphate	126727	NA	930	NA	27,000 (C)	27,000 (C)	18,000	18,000	18,000	5.9E+6	4,400	27,000
Urea	57136	NA	ID (N)	NA	ID	NLV	NLV	NLV	NLV	ID	ID	NA
Vanadium	7440622	NA	72,000	1.9E+5	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	7.5E+5 (DD)	NA
Vinyl acetate (I)	108054	NA	13,000	NA	2.4E+6 (C)	7.9E+5	1.7E+6	2.6E+6	5.8E+6	1.3E+10	2.4E+6 (C,DD)	2.4E+6
Vinyl chloride	75014	NA	40	300	20,000	270	4,200	30,000	73,000	3.5E+8	3,800	4.9E+5
White phosphorus (R)	12185103	NA	2.2	NA	58,000	NLV	NLV	NLV	NLV	ID	2,300 (DD)	NA

TABLE 2. SOIL: RESIDENTIAL AND COMMERCIAL I
PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS;
PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection			Indoor Air	Ambient Air (Y)				Direct Contact	
Guidesheet Number →		#10	#11	#12	#13	#14	#15	#16	#17	#18	#19	#20
Hazardous Substance	**Chemical Abstract Service Number**	**Statewide Default Background Levels**	**Drinking Water Protection Criteria & RBSLs**	**Groundwater Surface Water Interface Protection Criteria & RBSLs**	**Groundwater Contact Protection Criteria & RBSLs**	**Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs**	**Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs**	**Finite VSIC for 5 Meter Source Thickness**	**Finite VSIC for 2 Meter Source Thickness**	**Particulate Soil Inhalation Criteria & RBSLs**	**Direct Contact Criteria & RBSLs**	**Soil Saturation Concentration Screening Levels**
Xylenes (I)	1330207	NA	5,600	700	1.5E+5 (C)	1.5E+5 (C)	4.6E+7	6.1E+7	1.3E+8	2.9E+11	1.5E+5 (C)	1.5E+5
Zinc (B)	7440666	47,000	2.4E+6	(G)	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	1.7E+8	NA

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

All criteria, unless otherwise noted, are expressed in units of parts per billion (ppb). One ppb is equivalent to one microgram per kilogram (ug/kg). Criteria with six or more digits are expressed in scientific notation. For example, 200,000 ppb is presented as 2.0E+5. A footnote is designated by a letter in parentheses and is explained in the footnote pages that follow the criteria tables. When the risk-based criterion is less than the target detection limit (TDL), the TDL is listed as the criterion (R 299.5707). In these cases, two numbers are presented in the cell. The first number is the criterion (i.e., TDL), and the second number is the risk-based value. Criteria were promulgated December 21, 2002 within the Administrative Rules for Part 201, Environmental Remediation, of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended. These tables reflect modifications to the TDLs and new criteria consistent with the provisions of R299.5103(l) and R299.5706a, respectively.

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
Acenaphthene	83329	NA	3.0E+5	8.8E+5	4,400	9.7E+5	3.5E+8	9.7E+7	9.7E+7	9.7E+7	6.2E+9	1.3E+8	1.8E+8	1.5E+8	NA
Acenaphthylene	208968	NA	5,900	17,000	ID	4.4E+5	3.0E+6	2.7E+6	2.7E+6	2.7E+6	1.0E+9	5.2E+6	7.2E+6	6.1E+6	NA
Acetaldehyde (l)	75070	NA	19,000	54,000	2,600	1.1E+8 (C)	4.0E+5	2.1E+5	2.1E+5	2.9E+5	2.6E+8	9.5E+7	1.1E+8 (C)	1.1E+8	1.1E+8
Acetate	71501	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	ID	ID	ID	ID
Acetic acid	64197	NA	84,000	2.4E+5	3.6E+5	6.5E+8 (C)	NLV	NLV	NLV	NLV	7.4E+9	4.2E+8	5.8E+8	4.9E+8	6.5E+8
Acetone (l)	67641	NA	15,000	42,000	34,000	1.1E+8 (C)	1.1E+8 (C)	1.6E+8	1.6E+8	2.0E+8	1.7E+11	7.3E+7	1.0E+8	8.6E+7	1.1E+8
Acetonitrile	75058	NA	2,800	8,000	NA	2.2E+7 (C)	8.8E+6	1.9E+6	1.9E+6	2.2E+6	1.8E+9	1.4E+7	1.9E+7	1.6E+7	2.2E+7
Acetophenone	98862	NA	30,000	88,000	NA	1.1E+6 (C)	1.1E+6 (C)	5.2E+7	5.2E+7	5.2E+7	1.4E+10	1.1E+6 (C)	1.1E+6 (C)	1.1E+6 (C)	1.1E+6
Acrolein (l)	107028	NA	2,400	6,600	NA	2.3E+7 (C)	760	370	370	630	5.9E+5	1.2E+7	1.6E+7	1.4E+7	2.3E+7
Acrylamide	79061	NA	10	10	NA	2.6E+5	NLV	NLV	NLV	NLV	3.0E+6	8,700	12,000	10,000	NA
Acrylic acid	79107	NA	78,000	2.2E+5	NA	1.1E+8 (C)	5.5E+6	2.2E+5	2.7E+5	2.7E+5	2.9E+7	1.1E+8 (C,DD)	1.1E+8 (C,DD)	1.1E+8 (C,DD)	1.1E+8
Acrylonitrile (l)	107131	NA	100 (M); 52	220	100 (M,X); 98	2.8E+5	35,000	17,000	17,000	31,000	5.8E+7	74,000	1.0E+5	87,000	8.3E+6
Alachlor	15972608	NA	52	52	290 (X)	44,000	NLV	NLV	NLV	NLV	ID	3.9E+5	6.9E+5	5.1E+5	NA
Aldicarb	116063	NA	60	60	NA	2.4E+6	NLV	NLV	NLV	NLV	ID	7.3E+5	1.0E+6	8.6E+5	NA
Aldicarb sulfoxide	1646873	NA	200 (M)	200 (M)	NA	5.4E+7	NLV	NLV	NLV	NLV	ID	9.5E+5	1.3E+6	1.1E+6	NA
Aldicarb sulfone	1646884	NA	200 (M); 40	200 (M); 40	NA	4.2E+7	NLV	NLV	NLV	NLV	ID	8.0E+5	1.1E+6	9.4E+5	NA
Aldrin	309002	NA	NLL	NLL	NLL	NLL	7.1E+6	2.0E+5	2.0E+5	2.0E+5	8.0E+5	4,300	7,700	5,600	NA
Aluminum (B)	7429905	6.9E+6	1,000	1,000	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	3.7E+8 (DD)	4.1E+8 (DD)	3.9E+8 (DD)	NA
Ammonia	7664417	NA	ID	ID	(CC)	ID	ID	ID	ID	ID	2.9E+9	ID	ID	ID	1.0E+7

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
t-Amyl methyl ether (TAME)	994058	NA	3,900	3,900	NA	4.4E+5 (C)	1.1E+5	4.0E+5	7.8E+5	1.8E+6	1.8E+9	4.4E+5 (C)	4.4E+5 (C)	4.4E+5 (C)	4.4E+5
Aniline	62533	NA	1,100	4,400	330 (M); 80	2.8E+6	NLV	NLV	NLV	NLV	2.9E+7	1.5E+6	2.1E+6	1.8E+6	4.5E+6
Anthracene	120127	NA	41,000	41,000	ID	41,000	1.0E+9 (D)	1.6E+9	1.6E+9	1.6E+9	2.9E+10	7.3E+8	1.0E+9	8.6E+8	NA
Antimony	7440360	NA	4,300	4,300	94,000	4.9E+7	NLV	NLV	NLV	NLV	5.9E+6	6.7E+5	7.3E+5	7.0E+5	NA
Arsenic	7440382	5,800	4,600	4,600	70,000 (X)	2.0E+6	NLV	NLV	NLV	NLV	9.1E+5	37,000	46,000	41,000	NA
Asbestos (BB)	1332214	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	1.0E+7 (M); 85,000	ID	ID	ID	NA
Atrazine	1912249	NA	60	60	150 (X)	1.1E+5	NLV	NLV	NLV	NLV	ID	3.3E+5 (DD)	4.6E+5 (DD)	3.9E+5 (DD)	NA
Azobenzene	103333	NA	4,200	17,000	NA	3.0E+5	3.2E+7	2.1E+6	ID	ID	1.3E+8	6.6E+5	9.2E+5	7.7E+5	NA
Barium (B)	7440393	75,000	1.3E+6	1.3E+6	(G,X)	1.0E+9 (D)	NLV	NLV	NLV	NLV	1.5E+8	1.3E+8	1.5E+8	1.4E+8	NA
Benzene (I)	71432	NA	100	100	4,000 (X)	2.2E+5	8,400	45,000	99,000	2.3E+5	4.7E+8	4.0E+5 (C)	4.0E+5 (C)	4.0E+5 (C)	4.0E+5
Benzidine	92875	NA	1,000 (M); 6.0	1,000 (M); 6.0	ID	1,000 (M); 140	NLV	NLV	NLV	NLV	59,000	1,000 (M); 110	1,000 (M); 150	1,000 (M); 120	NA
Benzo(a)anthracene (Q)	56553	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	80,000	1.6E+5	1.1E+5	NA
Benzo(b)fluoranthene (Q)	205992	NA	NLL	NLL	NLL	NLL	ID	ID	ID	ID	ID	80,000	1.6E+5	1.1E+5	NA
Benzo(k)fluoranthene (Q)	207089	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	8.0E+5	1.6E+6	1.1E+6	NA
Benzo(g,h,i)perylene	191242	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	3.5E+8	7.0E+6	1.4E+7	9.5E+6	NA
Benzo(a)pyrene (Q)	50328	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	1.9E+6	8,000	16,000	11,000	NA
Benzoic acid	65850	NA	6.4E+5	1.8E+6	NA	7.0E+7	NLV	NLV	NLV	NLV	ID	1.0E+9 (D)	1.0E+9 (D)	1.0E+9 (D)	NA
Benzyl alcohol	100516	NA	2.0E+5	5.8E+5	NA	5.8E+6 (C)	NLV	NLV	NLV	NLV	1.5E+11	5.8E+6 (C)	5.8E+6 (C)	5.8E+6 (C)	5.8E+6
Benzyl chloride	100447	NA	150	640	NA	72,000	33,000	48,000	48,000	52,000	7.8E+7	2.2E+5	2.3E+5 (C)	2.3E+5 (C)	2.3E+5
Beryllium	7440417	NA	51,000	51,000	(G)	1.0E+9 (D)	NLV	NLV	NLV	NLV	5.9E+5	1.6E+6	1.6E+6	1.6E+6	NA
bis(2-Chloroethoxy)ethane	112265	NA	ID	ID	ID	ID	NLV	NLV	NLV	NLV	ID	ID	ID	ID	2.7E+6
bis(2-Chloroethyl)ether (I)	111444	NA	100	170	300	1.1E+5	44,000	13,000	13,000	13,000	1.2E+7	58,000	81,000	68,000	2.2E+6
bis(2-Ethylhexyl)phthalate	117817	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	8.9E+8	1.0E+7 (C)	1.0E+7 (C)	1.0E+7 (C)	1.0E+7
Boron (B)	7440428	NA	10,000	10,000	38,000	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	3.5E+8 (DD)	3.9E+8 (DD)	3.7E+8 (DD)	NA

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
Bromate	15541454	NA	200	200	800	96,000	NLV	NLV	NLV	NLV	ID	91,000	99,000	95,000	NA
Bromobenzene (I)	108861	NA	550	1,500	NA	3.6E+5	5.8E+5	5.4E+5	5.4E+5	5.4E+5	2.4E+8	7.6E+5 (C)	7.6E+5 (C)	7.6E+5 (C)	7.6E+5
Bromodichloromethane	75274	NA	1,600 (W)	1,600 (W)	ID	2.8E+5	6,400	31,000	31,000	57,000	1.1E+8	4.9E+5	6.8E+5	5.7E+5	1.5E+6
Bromoform	75252	NA	1,600 (W)	1,600 (W)	ID	8.7E+5 (C)	7.7E+5	3.1E+6	3.1E+6	3.1E+6	3.6E+9	8.7E+5 (C)	8.7E+5 (C)	8.7E+5 (C)	8.7E+5
Bromomethane	74839	NA	200	580	700	1.4E+6	1,600	13,000	57,000	1.4E+5	1.5E+8	1.0E+6	1.4E+6	1.2E+6	2.2E+6
n-Butanol (I)	71363	NA	19,000	54,000	NA	8.7E+6 (C)	NLV	NLV	NLV	NLV	1.0E+10	8.7E+6 (C)	8.7E+6 (C)	8.7E+6 (C)	8.7E+6
2-Butanone (MEK) (I)	78933	NA	2.6E+5	7.6E+5	44,000	2.7E+7 (C)	2.7E+7 (C)	3.5E+7	3.5E+7	3.6E+7	2.9E+10	2.7E+7 (C,DD)	2.7E+7 (C,DD)	2.7E+7 (C,DD)	2.7E+7
n-Butyl acetate	123864	NA	11,000	32,000	NA	1.1E+6 (C)	1.1E+6 (C)	1.4E+8	3.1E+8	3.5E+8	2.1E+11	1.1E+6 (C)	1.1E+6 (C)	1.1E+6 (C)	1.1E+6
t-Butyl alcohol	75650	NA	78,000	2.2E+5	NA	1.1E+8 (C)	1.1E+8 (C)	1.2E+8	2.4E+8	2.4E+8	5.6E+10	1.1E+8 (C)	1.1E+8 (C)	1.1E+8 (C)	1.1E+8
Butyl benzyl phthalate	85687	NA	3.1E+5 (C)	3.1E+5 (C)	26,000 (X)	3.1E+5 (C)	NLV	NLV	NLV	NLV	2.1E+10	3.1E+5 (C)	3.1E+5 (C)	3.1E+5 (C)	3.1E+5
n-Butylbenzene	104518	NA	1,600	4,600	ID	1.2E+5	ID	ID	ID	ID	ID	8.0E+6	1.0E+7 (C)	9.4E+6	1.0E+7
sec-Butylbenzene	135988	NA	1,600	4,600	ID	88,000	ID	ID	ID	ID	ID	8.0E+6	1.0E+7 (C)	9.4E+6	1.0E+7
t-Butylbenzene (I)	98066	NA	1,600	4,600	NA	1.8E+5	ID	ID	ID	ID	ID	8.0E+6	1.0E+7 (C)	9.4E+6	1.0E+7
Cadmium (B)	7440439	1,200	6,000	6,000	(G,X)	2.3E+8	NLV	NLV	NLV	NLV	2.2E+6	2.1E+6	2.1E+6	2.1E+6	NA
Camphene (I)	79925	NA	ID	ID	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	NA
Caprolactam	105602	NA	1.2E+5	3.4E+5	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	2.9E+8	3.1E+8 (DD)	4.8E+8 (DD)	3.8E+8 (DD)	NA
Carbaryl	63252	NA	14,000	40,000	NA	2.6E+6	ID	ID	ID	ID	ID	7.0E+7	9.8E+7	8.2E+7	NA
Carbazole	86748	NA	9,400	39,000	1,100	8.2E+5	NLV	NLV	NLV	NLV	ID	2.4E+6	3.4E+6	2.9E+6	NA
Carbofuran	1563662	NA	800	800	NA	6.8E+6	NLV	NLV	NLV	NLV	ID	3.6E+6	5.1E+6	4.3E+6	NA
Carbon disulfide (I,R)	75150	NA	16,000	46,000	ID	2.8E+5 (C)	1.4E+5	1.6E+6	8.0E+6	1.9E+7	2.1E+10	2.8E+5 (C,DD)	2.8E+5 (C,DD)	2.8E+5 (C,DD)	2.8E+5
Carbon tetrachloride	56235	NA	100	100	900 (X)	92,000	990	12,000	34,000	79,000	1.7E+8	3.9E+5 (C)	3.9E+5 (C)	3.9E+5 (C)	3.9E+5
Chlordane (J)	57749	NA	NLL	NLL	NLL	NLL	5.9E+7	4.2E+6	4.2E+6	4.2E+6	2.1E+7	1.5E+5	2.0E+5	1.7E+5	NA
Chloride	16887006	NA	5.0E+6	5.0E+6	2.5E+6 (X)	ID	NLV	NLV	NLV	NLV	ID	5.0E+5 (F)	5.0E+5 (F)	5.0E+5 (F)	NA
Chlorobenzene (I)	108907	NA	2,000	2,000	940	2.6E+5 (C)	2.2E+5	9.2E+5	1.1E+6	2.1E+6	2.1E+9	2.6E+5 (C)	2.6E+5 (C)	2.6E+5 (C)	2.6E+5

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
para-Chlorobenzenesulfonic acid	98668	NA	1.5E+05	4.2E+05	NA	NA	ID	ID	ID	ID	ID	7.3E+08	1.0E+09	8.6E+08	ID
1-Chloro-1,1-difluoroethane	75683	NA	3.0E+5	8.8E+05	NA	9.6E+5 (C)	9.6E+5 (C)	9.4E+7	5.7E+8	1.4E+9	1.5E+12	9.6E+5 (C)	9.6E+5 (C)	9.6E+5 (C)	9.6E+5
Chloroethane	75003	NA	8,600	34,000	ID	9.5E+5 (C)	9.5E+5 (C)	3.6E+7	1.2E+8	2.8E+8	2.9E+11	9.5E+5 (C)	9.5E+5 (C)	9.5E+5 (C)	9.5E+5
2-Chloroethyl vinyl ether	110758	NA	ID	ID	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	1.9E+6
Chloroform	67663	NA	1,600 (W)	1,600 (W)	3,400 (X)	1.5E+6 (C)	38,000	1.5E+5	3.4E+5	7.9E+5	1.6E+9	1.5E+6 (C)	1.5E+6 (C)	1.5E+6 (C)	1.5E+6
Chloromethane (I)	74873	NA	5,200	22,000	ID	1.1E+6 (C)	10,000	1.2E+5	1.0E+6	2.5E+6	2.6E+9	1.1E+6 (C)	1.1E+6 (C)	1.1E+6 (C)	1.1E+6
4-Chloro-3-methylphenol	59507	NA	5,800	16,000	280	3.0E+6	NLV	NLV	NLV	NLV	ID	1.5E+7	2.0E+7	1.7E+7	NA
beta-Chloronaphthalene	91587	NA	6.2E+5	1.8E+6	NA	2.3E+6	ID	ID	ID	ID	ID	1.8E+8	2.6E+8	2.1E+8	NA
2-Chlorophenol	95578	NA	900	2,600	440	1.9E+6	ID	ID	ID	ID	ID	4.5E+6	6.3E+6	5.3E+6	1.9E+7
o-Chlorotoluene (I)	95498	NA	3,300	9,300	NA	5.0E+5 (C)	5.0E+5 (C)	1.5E+6	3.1E+6	6.4E+6	2.1E+9	5.0E+5 (C)	5.0E+5 (C)	5.0E+5 (C)	5.0E+5
Chlorpyrifos	2921882	NA	17,000	48,000	1,500	8.4E+5	240	5,500	23,000	56,000	5.9E+7	3.4E+7	6.0E+7	4.4E+7	NA
Chromium (III) (B,H)	16065831	18,000 (total)	1.0E+9 (D)	1.0E+9 (D)	(G,X)	1.0E+9 (D)	NLV	NLV	NLV	NLV	1.5E+8	1.0E+9 (D)	1.0E+9 (D)	1.0E+9 (D)	NA
Chromium (VI)	18540299	NA	30,000	30,000	3,300	1.4E+8	NLV	NLV	NLV	NLV	2.4E+5	9.2E+6	1.0E+7	9.6E+6	NA
Chrysene (Q)	218019	NA	NLL	NLL	NLL	NLL	ID	ID	ID	ID	ID	8.0E+6	1.6E+7	1.1E+7	NA
Cobalt	7440484	6,800	800	2,000	2,000	4.8E+7	NLV	NLV	NLV	NLV	5.9E+6	9.0E+6	1.0E+7	1.0E+7	NA
Copper (B)	7440508	32,000	5.8E+6	5.8E+6	(G)	1.0E+9 (D)	NLV	NLV	NLV	NLV	5.9E+7	7.3E+7	7.9E+7	7.6E+7	NA
Cyanazine	21725462	NA	200	200	1,100 (X)	56,000	NLV	NLV	NLV	NLV	ID	66,000	92,000	77,000	NA
Cyanide (P,R)	57125	390 (total)	4,000	4,000	100	2.5E+5	NLV	NLV	NLV	NLV	2.5E+5	2.5E+5	2.5E+5	2.5E+5	NA
Cyclohexanone	108941	NA	5.2E+6	1.5E+7	NA	2.2E+8 (C)	32,000	1.3E+6	1.1E+7	2.7E+7	2.9E+10	2.2E+8 (C)	2.2E+8 (C)	2.2E+8 (C)	2.2E+8
Dacthal	1861321	NA	50,000	1.4E+5	NA	3.4E+5	NLV	NLV	NLV	NLV	ID	7.3E+6	1.0E+7	8.6E+6	NA
Dalapon	75990	NA	4,000	4,000	NA	5.9E+7 (C)	NLV	NLV	NLV	NLV	ID	5.9E+7 (C)	5.9E+7 (C)	5.9E+7 (C)	5.9E+7
4-4'-DDD	72548	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	5.6E+7	4.0E+5	7.1E+5	5.2E+5	NA
4-4'-DDE	72559	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	4.0E+7	1.9E+5	3.3E+5	2.4E+5	NA
4-4'-DDT	50293	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	4.0E+7	2.8E+5	3.4E+5	3.1E+5	NA

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV
PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS;
PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
Decabromodiphenyl ether	1163195	NA	1.4E+5	1.4E+5	NA	1.4E+5	1.0E+9 (D)	1.0E+8	1.0E+8	1.0E+8	1.0E+9	1.1E+7	2.0E+7	1.5E+7	NA
Di-n-butyl phthalate	84742	NA	7.6E+5 (C)	7.6E+5 (C)	11,000	7.6E+5 (C)	NLV	NLV	NLV	NLV	1.5E+9	7.6E+5 (C)	7.6E+5 (C)	7.6E+5 (C)	7.6E+5
Di(2-ethylhexyl) adipate	103231	NA	9.6E+5 (C)	9.6E+5 (C)	NA	9.6E+5 (C)	NLV	NLV	NLV	NLV	1.2E+10	9.6E+5 (C,DD)	9.6E+5 (C,DD)	9.6E+5 (C,DD)	9.6E+5
Di-n-octyl phthalate	117840	NA	1.0E+8	1.4E+8 (C)	ID	1.4E+8 (C)	NLV	NLV	NLV	NLV	ID	2.0E+7	3.6E+7	2.6E+7	1.4E+8
Diacetone alcohol (I)	123422	NA	ID	ID	NA	ID	NLV	NLV	NLV	NLV	7.1E+10	ID	ID	ID	1.1E+8
Diazinon	333415	NA	95	280	NA	95,000	NLV	NLV	NLV	NLV	ID	70,000 (DD)	1.1E+5 (DD)	86,000 (DD)	3.1E+5
Dibenzo(a,h)anthracene (Q)	53703	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	8,000	16,000	11,000	NA
Dibenzofuran	132649	NA	ID	ID	1,700	ID	ID	ID	ID	ID	ID	ID	ID	ID	NA
Dibromochloromethane	124481	NA	1,600 (W)	1,600 (W)	ID	3.6E+5	21,000	80,000	80,000	98,000	1.6E+8	5.0E+5	6.1E+5 (C)	5.8E+5	6.1E+5
Dibromochloropropane	96128	NA	10 (M); 4.0	10 (M); 4.0	NA	1,200 (C)	1,200 (C)	15,000	15,000	15,000	5.9E+6	1,200 (C)	1,200 (C)	1,200 (C)	1,200
Dibromomethane	74953	NA	1,600	4,600	NA	2.0E+6 (C)	ID	ID	ID	ID	ID	2.0E+6 (C)	2.0E+6 (C)	2.0E+6 (C)	2.0E+6
Dicamba	1918009	NA	4,400	13,000	NA	1.2E+7	NLV	NLV	NLV	NLV	ID	1.7E+7	3.5E+7	2.3E+7	NA
1,2-Dichlorobenzene	95501	NA	14,000	14,000	360	2.1E+5 (C)	2.1E+5 (C)	4.6E+7	4.6E+7	5.5E+7	4.4E+10	2.1E+5 (C)	2.1E+5 (C)	2.1E+5 (C)	2.1E+5
1,3-Dichlorobenzene	541731	NA	170	480	1,100	51,000	ID	ID	ID	ID	ID	1.7E+5 (C)	1.7E+5 (C)	1.7E+5 (C)	1.7E+5
1,4-Dichlorobenzene	106467	NA	1,700	1,700	290	1.4E+5	1.0E+5	2.6E+5	2.6E+5	3.4E+5	5.7E+8	1.9E+6	2.6E+6	2.2E+6	NA
3,3'-Dichlorobenzidine	91941	NA	2,000 (M); 28	2,000 (M); 110	2,000 (M,X); 510	4,600	NLV	NLV	NLV	NLV	8.2E+6	30,000	43,000	36,000	NA
Dichlorodifluoromethane	75718	NA	95,000	2.7E+5	ID	1.0E+6 (C)	1.7E+6	6.3E+7	5.5E+8	1.4E+9	1.5E+12	1.0E+6 (C)	1.0E+6 (C)	1.0E+6 (C)	1.0E+6
1,1-Dichloroethane	75343	NA	18,000	50,000	15,000	8.9E+5 (C)	4.3E+5	2.5E+6	6.0E+6	1.4E+7	1.5E+10	8.9E+5 (C)	8.9E+5 (C)	8.9E+5 (C)	8.9E+5
1,2-Dichloroethane (I)	107062	NA	100	100	7,200 (X)	3.8E+5	11,000	21,000	33,000	74,000	1.5E+8	4.2E+5	5.9E+5	4.9E+5	1.2E+6
1,1-Dichloroethylene (I)	75354	NA	140	140	1,300 (X)	2.2E+5	330	3,700	15,000	37,000	7.8E+7	5.7E+5 (C)	5.7E+5 (C)	5.7E+5 (C)	5.7E+5
cis-1,2-Dichloroethylene	156592	NA	1,400	1,400	12,000	6.4E+5 (C)	41,000	2.1E+5	4.3E+5	1.0E+6	1.0E+9	6.4E+5 (C)	6.4E+5 (C)	6.4E+5 (C)	6.4E+5
trans-1,2-Dichloroethylene	156605	NA	2,000	2,000	30,000	1.4E+6 (C)	43,000	3.3E+5	8.4E+5	2.0E+6	2.1E+9	1.4E+6 (C)	1.4E+6 (C)	1.4E+6 (C)	1.4E+6
2,6-Dichloro-4-nitroaniline	99309	NA	44,000	1.3E+5	NA	1.4E+5	NLV	NLV	NLV	NLV	ID	2.2E+8	3.1E+8	2.6E+8	NA
2,4-Dichlorophenol	120832	NA	1,500	4,200	380	9.6E+5	NLV	NLV	NLV	NLV	2.3E+9	1.8E+6 (C,DD)	1.8E+6 (C,DD)	1.8E+6 (C,DD)	1.8E+6

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
2,4-Dichlorophenoxyacetic acid	94757	NA	1,400	1,400	4,400	2.4E+6	NLV	NLV	NLV	NLV	2.9E+9	8.6E+6	1.0E+7	9.4E+6	NA
1,2-Dichloropropane (I)	78875	NA	100	100	5,800 (X)	3.2E+5	7,400	30,000	51,000	1.2E+5	1.2E+8	5.5E+5 (C)	5.5E+5 (C)	5.5E+5 (C)	5.5E+5
1,3-Dichloropropene	542756	NA	170	700	NA	1.1E+5	5,400	60,000	2.0E+5	4.7E+5	5.9E+8	2.4E+5	3.4E+5	2.9E+5	6.2E+5
Dichlorovos	62737	NA	50 (M); 32	130	NA	1.2E+5	NLV	NLV	NLV	NLV	1.5E+7	47,000	65,000	55,000	2.2E+6
Dicyclohexyl phthalate	84617	NA	ID	ID	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	NA
Dieldrin	60571	NA	NLL	NLL	NLL	NLL	7.2E+5	64,000	64,000	64,000	8.5E+5	4,700	8,300	6,100	NA
Diethyl ether	60297	NA	200	200	ID	7.4E+6 (C)	7.4E+6 (C)	1.0E+8	1.6E+8	3.5E+8	3.5E+11	7.4E+6 (C)	7.4E+6 (C)	7.4E+6 (C)	7.4E+6
Diethyl phthalate	84662	NA	1.1E+5	3.2E+5	2,200	7.4E+5 (C)	NLV	NLV	NLV	NLV	1.5E+9	7.4E+5 (C)	7.4E+5 (C)	7.4E+5 (C)	7.4E+5
Diethylene glycol monobutyl ether	112345	NA	1,800	5,000	NA	8.0E+7	NLV	NLV	NLV	NLV	5.9E+8	8.7E+6	1.2E+7	1.0E+7	1.1E+8
Diisopropyl ether	108203	NA	600	1,300 (C)	ID	1,300 (C)	1,300 (C)	3.2E+6	4.8E+6	1.0E+7	1.1E+10	1,300 (C)	1,300 (C)	1,300 (C)	1,300
Diisopropylamine (I)	108189	NA	110	320	NA	4.2E+5	ID	ID	ID	ID	ID	5.6E+5	7.9E+5	6.6E+5	6.7E+6
Dimethyl phthalate	131113	NA	7.9E+5 (C)	7.9E+5 (C)	NA	7.9E+5 (C)	NLV	NLV	NLV	NLV	1.5E+9	7.9E+5 (C)	7.9E+5 (C)	7.9E+5 (C)	7.9E+5
N,N-Dimethylacetamide	127195	NA	3,600	10,000	82,000 (X)	1.1E+8 (C)	NLV	NLV	NLV	NLV	ID	1.8E+7	2.6E+7	2.1E+7	1.1E+8
N,N-Dimethylaniline	121697	NA	320	920	NA	4.0E+5	8.0E+5 (C)	5.2E+5	5.2E+5	5.2E+5	3.3E+8	8.0E+5 (C)	8.0E+5 (C)	8.0E+5 (C)	8.0E+5
Dimethylformamide (I)	68122	NA	14,000	40,000	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	8.8E+8	7.0E+7	9.8E+7	8.2E+7	1.1E+8
2,4-Dimethylphenol	105679	NA	7,400	20,000	7,600	1.0E+7	NLV	NLV	NLV	NLV	2.1E+9	3.6E+7	5.1E+7	4.3E+7	NA
2,6-Dimethylphenol	576261	NA	330 (M); 88	330 (M); 260	NA	1.3E+5	NLV	NLV	NLV	NLV	ID	4.4E+5	6.1E+5	5.1E+5	NA
3,4-Dimethylphenol	95658	NA	330 (M); 200	580	NA	3.6E+5	NLV	NLV	NLV	NLV	ID	1.0E+6	1.4E+6	1.2E+6	NA
Dimethylsulfoxide	67685	NA	4.4E+6	1.3E+7	3.8E+6	1.8E+7 (C)	NLV	NLV	NLV	NLV	ID	1.8E+7 (C)	1.8E+7 (C)	1.8E+7 (C)	1.8E+7
2,4-Dinitrotoluene	121142	NA	430	640	NA	1.7E+5	NLV	NLV	NLV	NLV	2.0E+7	2.2E+5	3.1E+5	2.6E+5	NA
Dinoseb	88857	NA	300	300	200 (M); 43	1.4E+5 (C)	NLV	NLV	NLV	NLV	ID	1.4E+5 (C,DD)	1.4E+5 (C,DD)	1.4E+5 (C,DD)	1.4E+5
1,4-Dioxane (I)	123911	NA	1,700	7,000	56,000	3.4E+7	NLV	NLV	NLV	NLV	7.1E+8	2.4E+6	3.4E+6	2.9E+6	9.7E+7
Diquat	85007	NA	400	400	NA	1.4E+7	NLV	NLV	NLV	NLV	ID	1.6E+6	2.2E+6	1.9E+6	NA
Diuron	330541	NA	620	1,800	NA	7.4E+5	NLV	NLV	NLV	NLV	2.1E+8	3.1E+6	4.4E+6	3.7E+6	NA

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
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Endosulfan (J)	115297	NA	NLL	NLL	NLL	NLL	ID	ID	ID	ID	ID	4.4E+6	6.1E+6	5.1E+6	NA
Endothall	145733	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	1.0E+9	1.2E+7	1.7E+7	1.5E+7	NA
Endrin	72208	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	1.9E+5	3.4E+5	2.5E+5	NA
Epichlorohydrin (I)	106898	NA	100	100	NA	2.2E+5	1.2E+5	37,000	37,000	37,000	2.9E+7	41,000	58,000	48,000	7.3E+6
Ethanol (I)	64175	NA	3.8E+7	7.6E+7	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	5.6E+11	1.1E+8 (C,DD)	1.1E+8 (C,DD)	1.1E+8 (C,DD)	1.1E+8
Ethyl acetate (I)	141786	NA	1.3E+5	3.8E+5	NA	7.5E+6 (C)	7.5E+6 (C)	5.9E+7	5.9E+7	1.0E+8	9.4E+10	7.5E+6 (C)	7.5E+6 (C)	7.5E+6 (C)	7.5E+6
Ethyl-tert-butyl ether (ETBE)	637923	NA	980	980	ID	ID	6.5E+5 (C)	2.3E+6	4.6E+6	1.1E+7	1.1E+10	ID	ID	ID	6.5E+5
Ethylbenzene (I)	100414	NA	1,500	1,500	360	1.4E+5 (C)	1.4E+5 (C)	2.4E+6	3.1E+6	6.5E+6	1.3E+10	1.4E+5 (C)	1.4E+5 (C)	1.4E+5 (C)	1.4E+5
Ethylene dibromide	106934	NA	20 (M); 1.0	20 (M); 1.0	20 (M); 4.0	500	3,600	5,800	5,800	9,800	1.8E+7	430	600	500	8.9E+5
Ethylene glycol	107211	NA	3.0E+5	8.4E+5	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	2.9E+10	1.1E+8 (C)	1.1E+8 (C)	1.1E+8 (C)	1.1E+8
Ethylene glycol monobutyl ether	111762	NA	74,000	2.0E+5	NA	4.1E+7 (C)	1.4E+6	2.1E+7	1.5E+8	3.6E+8	3.8E+11	4.1E+7 (C)	4.1E+7 (C)	4.1E+7 (C)	4.1E+7
Fluoranthene	206440	NA	7.3E+5	7.3E+5	5,500	7.3E+5	1.0E+9 (D)	8.9E+8	8.8E+8	8.8E+8	4.1E+9	1.3E+8	2.4E+8	1.7E+8	NA
Fluorene	86737	NA	3.9E+5	8.9E+5	5,300	8.9E+5	1.0E+9 (D)	1.5E+8	1.5E+8	1.5E+8	4.1E+9	8.7E+7	1.2E+8	1.0E+8	NA
Fluorine (soluble fluoride) (B)	7782414	NA	40,000	40,000	NA	2.4E+8	NLV	NLV	NLV	NLV	ID	6.7E+7 (DD)	7.4E+7 (DD)	7.0E+7 (DD)	NA
Formaldehyde	50000	NA	26,000	76,000	2,400	6.0E+7 (C)	65,000	43,000	69,000	1.5E+5	3.0E+8	6.0E+7 (C)	6.0E+7 (C)	6.0E+7 (C)	6.0E+7
Formic acid (I,U)	64186	NA	2.0E+5	5.8E+5	ID	1.1E+8 (C)	2.8E+6	2.6E+5	1.6E+5	1.6E+5	5.9E+7	1.1E+8 (C)	1.1E+8 (C)	1.1E+8 (C)	1.1E+8
1-Formylpiperidine	2591868	NA	1,600	4,600	NA	ID	ID	ID	ID	ID	ID	8.0E+6	1.0E+7 (C)	9.4E+6	1.0E+7
Gentian violet	548629	NA	300	1,300	NA	2.0E+7	NLV	NLV	NLV	NLV	ID	4.4E+5	6.2E+5	5.2E+5	NA
Glyphosate	1071836	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	5.7E+7 (DD)	1.2E+8 (DD)	7.8E+7 (DD)	NA
Heptachlor	76448	NA	NLL	NLL	NLL	NLL	1.9E+6	2.1E+5	2.1E+5	2.1E+5	3.0E+6	23,000	42,000	30,000	NA
Heptachlor epoxide	1024573	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	1.5E+6	9,500	17,000	12,000	NA
n-Heptane	142825	NA	2.4E+5 (C)	2.4E+5 (C)	NA	2.4E+5 (C)	2.4E+5 (C)	2.5E+7	4.5E+7	1.0E+8	1.0E+11	2.4E+5 (C)	2.4E+5 (C)	2.4E+5 (C)	2.4E+5
Hexabromobenzene	87821	NA	5,400	5,400	ID	5,400	ID	ID	ID	ID	ID	3.1E+6	5.6E+6	4.1E+6	NA
Hexachlorobenzene (C-66)	118741	NA	1,800	1,800	350	8,200	2.2E+5	56,000	56,000	56,000	8.5E+6	37,000	67,000	49,000	NA

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
Hexachlorobutadiene (C-46)	87683	NA	26,000	72,000	91	3.5E+5 (C)	3.5E+5 (C)	4.6E+5	4.6E+5	4.6E+5	1.8E+8	3.5E+5 (C)	3.5E+5 (C)	3.5E+5 (C)	3.5E+5
alpha-Hexachlorocyclohexane	319846	NA	18	71	NA	2,500	1.6E+5	41,000	86,000	86,000	2.1E+6	12,000	17,000	14,000	NA
beta-Hexachlorocyclohexane	319857	NA	37	150	NA	5,100	NLV	NLV	NLV	NLV	7.4E+6	25,000	35,000	29,000	NA
Hexachlorocyclopentadiene (C-56)	77474	NA	3.2E+5	3.2E+5	ID	7.2E+5 (C)	56,000	60,000	60,000	60,000	5.9E+6	7.2E+5 (C)	7.2E+5 (C)	7.2E+5 (C)	7.2E+5
Hexachloroethane	67721	NA	430	1,200	1,800 (X)	1.1E+5	79,000	6.6E+5	1.4E+6	1.4E+6	1.0E+8	7.3E+5	1.0E+6	8.6E+5	NA
n-Hexane	110543	NA	44,000 (C)	44,000 (C)	NA	44,000 (C)	44,000 (C)	3.5E+6	3.5E+6	6.4E+6	5.9E+9	44,000 (C)	44,000 (C)	44,000 (C)	44,000
2-Hexanone	591786	NA	20,000	58,000	NA	2.5E+6 (C)	1.8E+6	1.3E+6	1.3E+6	1.5E+6	1.2E+9	2.5E+6 (C)	2.5E+6 (C)	2.5E+6 (C)	2.5E+6
Indeno(1,2,3-cd)pyrene (Q)	193395	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	80,000	1.6E+5	1.1E+5	NA
Iron (B)	7439896	1.2E+7	6,000	6,000	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	5.8E+8	6.2E+8	6.0E+8	NA
Isobutyl alcohol (I)	78831	NA	46,000	1.3E+5	NA	8.9E+6 (C)	8.9E+6 (C)	9.5E+7	9.5E+7	9.5E+7	4.4E+10	8.9E+6 (C)	8.9E+6 (C)	8.9E+6 (C)	8.9E+6
Isophorone	78591	NA	15,000	62,000	11,000 (X)	2.4E+6 (C)	NLV	NLV	NLV	NLV	8.2E+9	2.4E+6 (C)	2.4E+6 (C)	2.4E+6 (C)	2.4E+6
Isopropyl alcohol (I)	67630	NA	9,400	26,000	1.1E+6 (X)	1.1E+8 (C)	NLV	NLV	NLV	NLV	6.5E+9	4.7E+7	6.5E+7	5.5E+7	1.1E+8
Isopropyl benzene	98828	NA	91,000	2.6E+5	ID	3.9E+5 (C)	3.9E+5 (C)	2.0E+6	2.0E+6	3.0E+6	2.6E+9	3.9E+5 (C)	3.9E+5 (C)	3.9E+5 (C)	3.9E+5
Lead (B)	7439921	21,000	7.0E+5	7.0E+5	(G,X)	ID	NLV	NLV	NLV	NLV	4.4E+7	9.0E+5 (DD)	4.0E+5	4.0E+5	NA
Lindane	58899	NA	20 (M); 7.0	20 (M); 7.0	20 (M); 0.99	7,100	ID	ID	ID	ID	ID	42,000	49,000	45,000	NA
Lithium (B)	7439932	9,800	3,400	7,000	1,900	1.1E+8	NLV	NLV	NLV	NLV	ID	3.1E+7 (DD)	3.5E+7 (DD)	3.3E+7 (DD)	NA
Magnesium (B)	7439954	NA	8.0E+6	2.2E+7	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	2.9E+9	1.0E+9 (D)	1.0E+9 (D)	1.0E+9 (D)	NA
Manganese (B)	7439965	4.4E+5	1,000	1,000	(G,X)	1.8E+8	NLV	NLV	NLV	NLV	1.5E+6	9.0E+7	9.8E+7	9.4E+7	NA
Mercury (Total) (B,Z)	Varies	130	1,700	1,700	50 (M); 1.2	47,000	89,000	62,000	62,000	62,000	8.8E+6	5.8E+5	6.2E+5	6.0E+5	NA
Methane	74828	NA	ID	ID	NA	ID	8.4E+6 ug/m3 (GG)	ID	ID	ID	ID	ID	ID	ID	ID
Methanol	67561	NA	74,000	2.0E+5	9,600	3.1E+6 (C)	3.1E+6 (C)	3.7E+7	4.6E+7	9.7E+7	9.6E+10	3.1E+6 (C)	3.1E+6 (C)	3.1E+6 (C)	3.1E+6
Methoxychlor	72435	NA	16,000	16,000	NA	18,000	ID	ID	ID	ID	ID	5.6E+6	1.0E+7	7.3E+6	NA
2-Methoxyethanol (I)	109864	NA	150	420	NA	1.7E+7	NLV	NLV	NLV	NLV	5.9E+8	7.3E+5	1.0E+6	8.6E+5	1.1E+8
2-Methyl-4-chlorophenoxyacetic acid	94746	NA	390	1,100	NA	4.9E+5	NLV	NLV	NLV	NLV	ID	7.3E+5	1.0E+6	8.6E+5	NA

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
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2-Methyl-4,6-dinitrophenol	534521	NA	830 (M); 400	830 (M); 400	NA	1.9E+5	NLV	NLV	NLV	NLV	ID	2.6E+5	3.6E+5	3.0E+5	NA
N-Methyl-morpholine (I)	109024	NA	400	1,100	NA	3.0E+7	NLV	NLV	NLV	NLV	ID	2.0E+6	2.8E+6	2.3E+6	1.1E+8
Methyl parathion	298000	NA	46	130	NA	76,000	NLV	NLV	NLV	NLV	ID	1.8E+5	2.6E+5	2.1E+5	NA
4-Methyl-2-pentanone (MIBK) (I)	108101	NA	36,000	1.0E+5	ID	2.7E+6 (C)	2.7E+6 (C)	5.3E+7	5.3E+7	7.0E+7	6.0E+10	2.7E+6 (C)	2.7E+6 (C)	2.7E+6 (C)	2.7E+6
Methyl-tert-butyl ether (MTBE)	1634044	NA	800	800	15,000 (X)	5.9E+6 (C)	5.9E+6 (C)	3.0E+7	4.1E+7	8.9E+7	8.8E+10	5.9E+6 (C)	5.9E+6 (C)	5.9E+6 (C)	5.9E+6
Methylcyclopentane (I)	96377	NA	ID	ID	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	3.5E+5
4,4'-Methylene-bis-2-chloroaniline (MBOCA)	101144	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	1.1E+8	32,000	44,000	37,000	NA
Methylene chloride	75092	NA	100	100	19,000 (X)	2.3E+6 (C)	2.4E+5	7.0E+5	1.7E+6	4.0E+6	8.3E+9	2.3E+6 (C)	2.3E+6 (C)	2.3E+6 (C)	2.3E+6
2-Methylnaphthalene	91576	NA	57,000	1.7E+5	ID	5.5E+6	ID	ID	ID	ID	ID	2.6E+7	3.7E+7	3.1E+7	NA
Methylphenols (J)	1319773	NA	7,400	20,000	1,400	1.6E+7	NLV	NLV	NLV	NLV	2.9E+9	3.6E+7	5.1E+7	4.3E+7	NA
Metolachlor	51218452	NA	4,800	20,000	NA	4.4E+5 (C)	NLV	NLV	NLV	NLV	ID	4.4E+5 (C,DD)	4.4E+5 (C,DD)	4.4E+5 (C,DD)	4.4E+5
Metribuzin	21087649	NA	3,600	10,000	NA	2.40E+07	ID	ID	ID	ID	ID	2.8E+7	5.0E+7	3.6E+7	NA
Mirex	2385855	NA	NLL	NLL	NLL	NLL	ID	ID	ID	ID	ID	40,000	72,000	52,000	NA
Molybdenum (B)	7439987	NA	1,500	4,200	16,000 (X)	1.9E+7	NLV	NLV	NLV	NLV	ID	9.6E+6	1.0E+7	1.0E+7	NA
Naphthalene	91203	NA	35,000	1.0E+5	870	2.1E+6	4.7E+5	3.5E+5	3.5E+5	3.5E+5	8.8E+7	5.2E+7	7.2E+7	6.1E+7	NA
Nickel (B)	7440020	20,000	1.0E+5	1.0E+5	(G)	1.0E+9 (D)	NLV	NLV	NLV	NLV	1.6E+7	1.5E+8	1.6E+8	1.5E+8	NA
Nitrate (B,N)	14797558	NA	2.0E+5 (N)	2.0E+5 (N)	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	ID	ID	ID	NA
Nitrite (B,N)	14797650	NA	20,000 (N)	20,000 (N)	NA	3.8E+8	NLV	NLV	NLV	NLV	ID	ID	ID	ID	NA
Nitrobenzene (I)	98953	NA	330 (M); 68	330 (M); 190	3,600 (X)	2.2E+5	1.7E+5	64,000	64,000	64,000	2.1E+7	3.4E+5	4.7E+5	3.9E+5	4.9E+5
2-Nitrophenol	88755	NA	400	1,200	ID	1.6E+6	NLV	NLV	NLV	NLV	ID	2.0E+6	2.9E+6	2.4E+6	NA
n-Nitroso-di-n-propylamine	621647	NA	330 (M); 100	330 (M); 100	NA	7,200	NLV	NLV	NLV	NLV	2.0E+6	5,400	7,600	6,400	1.5E+6
N-Nitrosodiphenylamine	86306	NA	5,400	22,000	NA	7.0E+5	NLV	NLV	NLV	NLV	ID	7.8E+6	1.1E+7	9.2E+6	NA
Oxamyl	23135220	NA	4,000	4,000	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	2.8E+7	3.9E+7	3.3E+7	NA
Oxo-hexyl acetate	88230357	NA	1,500	4,200	NA	ID	ID	ID	ID	ID	2.4E+9	7.3E+6	1.0E+7	8.6E+6	1.0E+7

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			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
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Pendimethalin	40487421	NA	1.1E+6	1.1E+6	NA	1.1E+6	NLV	NLV	NLV	NLV	ID	1.3E+8	2.4E+8	1.7E+8	NA
Pentachlorobenzene	608935	NA	29,000	81,000	9,500	1.9E+5 (C)	ID	ID	ID	ID	ID	1.9E+5 (C)	1.9E+5 (C)	1.9E+5 (C)	1.9E+5
Pentachloronitrobenzene	82688	NA	37,000	37,000	NA	37,000	2.2E+5	2.8E+5	2.8E+5	2.8E+5	1.5E+8	5.5E+6	7.7E+6	6.4E+6	NA
Pentachlorophenol	87865	NA	22	22	(G,X)	4,300	NLV	NLV	NLV	NLV	1.3E+8	3.2E+5	9.2E+5	4.9E+5	NA
Pentane	109660	NA	ID	ID	NA	ID	1.8E+5	4.4E+7	3.4E+8	6.0E+08	5.3E+11	ID	ID	ID	2.4E+5
2-Pentene (I)	109682	NA	ID	ID	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	2.2E+5
Phenanthrene	85018	NA	56,000	1.6E+5	5,300	1.1E+6	5.1E+6	1.9E+5	1.9E+5	1.9E+5	2.9E+6	5.2E+6	7.2E+6	6.1E+6	NA
Phenol	108952	NA	88,000	2.6E+5	4,200	1.2E+7 (C)	NLV	NLV	NLV	NLV	1.8E+10	1.2E+7 (C,DD)	1.2E+7 (C,DD)	1.2E+7 (C,DD)	1.2E+7
Phosphorus (Total)	7723140	NA	1.3E+6	4.8E+6	(EE)	ID	NLV	NLV	NLV	NLV	ID	1.0E+9 (D)	1.0E+9 (D)	1.0E+9 (D)	NA
Phthalic acid	88993	NA	2.8E+5	8.0E+5	NA	1.7E+6 (C)	NLV	NLV	NLV	NLV	ID	1.7E+6 (C)	1.7E+6 (C)	1.7E+6 (C)	1.7E+6
Phthalic anhydride	85449	NA	3.0E+5	8.8E+5	NA	1.1E+6 (C)	NLV	NLV	NLV	NLV	ID	1.1E+6 (C)	1.1E+6 (C)	1.1E+6 (C)	1.1E+6
Picloram	1918021	NA	10,000	10,000	920	8.6E+6	NLV	NLV	NLV	NLV	ID	5.1E+7	7.1E+7	6.0E+7	NA
Piperidine	110894	NA	64	180	NA	6.8E+5	NLV	NLV	NLV	NLV	4.1E+9	3.2E+5	4.5E+5	3.8E+5	1.2E+8
Polybrominated biphenyls (J)	67774327	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	ID	4,800	8,600	6,300	NA
Polychlorinated biphenyls (PCBs) (J,T)	1336363	NA	NLL	NLL	NLL	NLL	1.6E+7	8.1E+5	2.8E+7	2.8E+7	6.5E+6	(T)	(T)	(T)	NA
Prometon	1610180	NA	4,900	14,000	NA	5.5E+6	NLV	NLV	NLV	NLV	ID	1.6E+7	2.2E+7	1.9E+7	NA
Propachlor	1918167	NA	1,900	5,400	NA	8.8E+6	NLV	NLV	NLV	NLV	ID	9.5E+6	1.3E+7	1.1E+7	NA
Propazine	139402	NA	4,000	11,000	NA	1.7E+5	NLV	NLV	NLV	NLV	ID	2.0E+7	2.8E+7	2.3E+7	NA
Propionic acid	79094	NA	2.4E+5	7.0E+5	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	8.8E+9	1.1E+8 (C)	1.1E+8 (C)	1.1E+8 (C)	1.1E+8
Propyl alcohol (I)	71238	NA	28,000	80,000	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	2.1E+10	7.4E+7 (DD)	1.1E+8 (DD)	9.1E+7(DD)	1.1E+8
n-Propylbenzene (I)	103651	NA	1,600	4,600	NA	3.0E+5	ID	ID	ID	ID	5.9E+8	8.0E+6	1.0E+7 (C)	9.4E+6	1.0E+7
Propylene glycol	57556	NA	3.0E+6	8.4E+6	5.8E+6	1.1E+8 (C)	NLV	NLV	NLV	NLV	1.8E+11	1.1E+8 (C)	1.1E+8 (C)	1.1E+8 (C)	1.1E+8
Pyrene	129000	NA	4.8E+5	4.8E+5	ID	4.8E+5	1.0E+9 (D)	7.8E+8	7.8E+8	7.8E+8	2.9E+9	8.4E+7	1.5E+8	1.1E+8	NA
Pyridine (I)	110861	NA	400	420	NA	37,000 (C)	2,000	9,800	40,000	97,000	1.0E+8	37,000 (C)	37,000 (C)	37,000 (C)	37,000

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
Selenium (B)	7782492	410	4,000	4,000	400	7.8E+7	NLV	NLV	NLV	NLV	5.9E+7	9.6E+6	1.0E+7	1.0E+7	NA
Silver (B)	7440224	1,000	4,500	13,000	100 (M); 27	2.0E+8	NLV	NLV	NLV	NLV	2.9E+6	9.0E+6	9.8E+6	9.4E+6	NA
Silvex (2,4,5-TP)	93721	NA	3,600	3,600	2,200	3.1E+6	NLV	NLV	NLV	NLV	ID	5.5E+6	7.7E+6	6.4E+6	NA
Simazine	122349	NA	80	80	NA	90,000	NLV	NLV	NLV	NLV	ID	3.8E+6	5.3E+6	4.5E+6	NA
Sodium	17341252	NA	2.5E+6	7.0E+6	NA	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	1.0E+9 (D)	1.0E+9 (D)	1.0E+9 (D)	NA
Sodium azide	26628228	NA	1,800	5,000	NA	ID	ID	ID	ID	ID	ID	8.70E+06	1.20E+07	1.00E+07	NA
Strontium (B)	7440246	NA	92,000	2.6E+5	46,000 (X)	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	1.0E+9 (D)	1.0E+9 (D)	1.0E+9 (D)	NA
Styrene	100425	NA	2,700	2,700	2,200	2.7E+5	5.2E+5 (C)	3.3E+6	3.3E+6	4.2E+6	6.9E+9	5.2E+5 (C)	5.2E+5 (C)	5.2E+5 (C)	5.2E+5
Sulfate	14808798	NA	5.0E+6	5.0E+6	NA	ID	NLV	NLV	NLV	NLV	ID	ID	ID	ID	NA
Tebuthiuron	34014181	NA	10,000	30,000	NA	5.0E+7	NLV	NLV	NLV	NLV	ID	2.7E+7 (DD)	4.2E+7 (DD)	3.3E+7 (DD)	NA
2,3,7,8-Tetrabromodibenzo-p-dioxin (O)	50585416	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	(O)	(O)	(O)	(O)	NA
1,2,4,5-Tetrachlorobenzene	95943	NA	1.5E+6	1.5E+6	3,400 (X)	1.5E+6	ID	ID	ID	ID	ID	2.5E+8	3.5E+8	2.9E+8	NA
2,3,7,8-Tetrachlorodibenzo-p-dioxin (O)	1746016	NA	NLL	NLL	NLL	NLL	NLV	NLV	NLV	NLV	89 (O)	0.99 (O)	1.4 (O)	2.9 (O)	NA
1,1,1,2-Tetrachloroethane	630206	NA	1,500	6,400	ID (X)	4.4E+5 (C)	33,000	1.2E+5	2.1E+5	3.3E+5	5.3E+8	4.4E+5 (C)	4.4E+5 (C)	4.4E+5 (C)	4.4E+5
1,1,2,2-Tetrachloroethane	79345	NA	170	700	1,600 (X)	94,000	23,000	34,000	34,000	34,000	6.8E+7	2.4E+5	3.4E+5	2.9E+5	8.7E+5
Tetrachloroethylene	127184	NA	100	100	900 (X)	88,000 (C)	60,000	6.0E+5	1.4E+6	3.3E+6	6.8E+9	88,000 (C)	88,000 (C)	88,000 (C)	88,000
Tetrahydrofuran	109999	NA	1,900	5,400	2.2E+5 (X)	3.2E+7	2.4E+6	1.5E+7	6.7E+7	1.6E+8	1.7E+11	9.5E+6	1.3E+7	1.1E+7	1.2E+8
Tetranitromethane	509148	NA	ID	ID	ID	ID	600	500 (M); 180	ID	ID	2.6E+5	ID	ID	ID	ID
Thallium (B)	7440280	NA	2,300	2,300	4,200 (X)	1.5E+7	NLV	NLV	NLV	NLV	ID	1.3E+5	1.4E+5	1.3E+5	NA
Toluene (I)	108883	NA	16,000	16,000	2,800	2.5E+5 (C)	2.5E+5 (C)	3.3E+6	3.6E+7	3.6E+7	1.2E+10	2.5E+5 (C)	2.5E+5 (C)	2.5E+5 (C)	2.5E+5
p-Toluidine	106490	NA	660 (M); 300	1,200	NA	4.8E+5	NLV	NLV	NLV	NLV	1.3E+8	4.3E+5	6.1E+5	5.1E+5	1.2E+6
Toxaphene	8001352	NA	24,000	24,000	860	3.6E+5	NLV	NLV	NLV	NLV	1.2E+7	85,000	1.5E+5	1.1E+5	NA
Triallate	2303175	NA	95,000	2.5E+5 (C)	NA	2.5E+5 (C)	ID	ID	ID	ID	ID	2.5E+5 (C)	2.5E+5 (C)	2.5E+5 (C)	2.5E+5
Tributylamine	102829	NA	7,800	23,000	ID	1.8E+6	1.1E+6	7.2E+5	7.2E+5	7.2E+5	2.1E+8	2.6E+6	3.6E+6	3.0E+6	3.7E+6

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS; PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
1,2,4-Trichlorobenzene	120821	NA	4,200	4,200	1,800	1.1E+6	1.1E+6 (C)	3.4E+7	3.4E+7	3.4E+7	1.1E+10	1.1E+6 (C,DD)	1.1E+6 (C,DD)	1.1E+6 (C,DD)	1.1E+6
1,1,1-Trichloroethane	71556	NA	4,000	4,000	4,000	4.6E+5 (C)	4.6E+5	4.5E+6	1.5E+7	3.1E+7	2.9E+10	4.6E+5 (C)	4.6E+5 (C)	4.6E+5 (C)	4.6E+5
1,1,2-Trichloroethane	79005	NA	100	100	6,600 (X)	4.2E+5	24,000	57,000	57,000	1.2E+5	2.5E+8	8.4E+5	9.2E+5 (C)	9.2E+5 (C)	9.2E+5
Trichloroethylene	79016	NA	100	100	4,000 (X)	4.4E+5	37,000	2.6E+5	4.4E+5	1.1E+6	2.3E+9	5.0E+5 (C,DD)	5.0E+5 (C,DD)	5.0E+5 (C,DD)	5.0E+5
Trichlorofluoromethane	75694	NA	52,000	1.5E+5	NA	5.6E+5 (C)	5.6E+5 (C)	1.1E+8	1.4E+11	1.4E+11	1.7E+12	5.6E+5 (C)	5.6E+5 (C)	5.6E+5 (C)	5.6E+5
2,4,5-Trichlorophenol	95954	NA	39,000	1.1E+5	NA	9.1E+6	NLV	NLV	NLV	NLV	1.0E+10	7.3E+7	1.0E+8	8.6E+7	NA
2,4,6-Trichlorophenol	88062	NA	2,400	9,400	330 (M); 100	2.0E+5	NLV	NLV	NLV	NLV	1.3E+9	3.3E+6	4.6E+6	3.9E+6	NA
1,2,3-Trichloropropane	96184	NA	840	2,400	NA	8.3E+5 (C)	ID	ID	ID	ID	ID	8.3E+5 (C)	8.3E+5 (C)	8.3E+5 (C)	8.3E+5
1,1,2-Trichloro-1,2,2-trifluoroethane	76131	NA	5.5E+5 (C)	5.5E+5 (C)	1,700	5.5E+5 (C)	5.5E+5 (C)	2.1E+8	8.9E+8	2.1E+9	2.3E+12	5.5E+5 (C)	5.5E+5 (C)	5.5E+5 (C)	5.5E+5
Triethanolamine	102716	NA	74,000	2.0E+5	NA	1.1E+8 (C)	NLV	NLV	NLV	NLV	1.5E+9	1.1E+8 (C)	1.1E+8 (C)	1.1E+8 (C)	1.1E+8
Triethylene glycol	112276	NA	1.1E+5 (C)	1.1E+5 (C)	NA	1.1E+5 (C)	NLV	NLV	NLV	NLV	ID	1.1E+5 (C,DD)	1.1E+5 (C,DD)	1.1E+5 (C,DD)	1.1E+5
3-Trifluoromethyl-4-nitrophenol	88302	NA	1.1E+5	3.1E+5	NA	1.2E+8	NLV	NLV	NLV	NLV	ID	2.4E+8 (DD)	3.7E+8 (DD)	3.0E+8 (DD)	NA
Trifluralin	1582098	NA	1.9E+5	5.7E+5	NA	1.2E+7	ID	ID	ID	ID	ID	5.7E+6	1.0E+7	7.4E+6	NA
2,2,4-Trimethyl pentane	540841	NA	ID	ID	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	19,000
2,4,4-Trimethyl-2-pentene (I)	107404	NA	ID	ID	NA	ID	ID	ID	ID	ID	ID	ID	ID	ID	56,000
1,2,4-Trimethylbenzene (I)	95636	NA	2,100	2,100	570	1.1E+5 (C)	1.1E+5 (C)	2.5E+7	6.0E+8	6.0E+8	3.6E+10	1.1E+5 (C)	1.1E+5 (C)	1.1E+5 (C)	1.1E+5
1,3,5-Trimethylbenzene (I)	108678	NA	1,800	1,800	1,100	94,000 (C)	94,000 (C)	1.9E+7	4.6E+8	4.6E+8	3.6E+10	94,000 (C)	94,000 (C)	94,000 (C)	94,000
Triphenyl phosphate	115866	NA	1.1E+5 (C)	1.1E+5 (C)	NA	1.1E+5 (C)	NLV	NLV	NLV	NLV	ID	1.1E+5 (C)	1.1E+5 (C)	1.1E+5 (C)	1.1E+5
tris(2,3-Dibromopropyl)phosphate	126727	NA	930	930	NA	27,000 (C)	27,000 (C)	60,000	60,000	60,000	7.4E+6	20,000	27,000 (C)	24,000	27,000
Urea	57136	NA	ID (N)	ID (N)	NA	ID	NLV	NLV	NLV	NLV	ID	ID	ID	ID	NA
Vanadium	7440622	NA	72,000	9.9E+5	1.9E+5	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	5.5E+6 (DD)	6.2E+6 (DD)	5.9E+6 (DD)	NA
Vinyl acetate (I)	108054	NA	13,000	36,000	NA	2.4E+6 (C)	1.5E+6	2.0E+6	2.7E+6	5.9E+6	5.9E+9	2.4E+6 (C,DD)	2.4E+6 (C,DD)	2.4E+6 (C,DD)	2.4E+6
Vinyl chloride	75014	NA	40	40	300	20,000	2,800	29,000	1.7E+5	4.2E+5	8.9E+8	34,000	47,000	40,000	4.9E+5
White phosphorus (R)	12185103	NA	2.2	6.0	NA	58,000	NLV	NLV	NLV	NLV	ID	17,000 (DD)	18,000 (DD)	18,000 (DD)	NA

TABLE 3. SOIL: INDUSTRIAL AND COMMERCIAL II, III, AND IV
PART 201 GENERIC CLEANUP CRITERIA AND SCREENING LEVELS;
PART 213 TIER 1 RISK-BASED SCREENING LEVELS (RBSLs)

			Groundwater Protection				Indoor Air	Ambient Air (Y)				Direct Contact			
Guidesheet Number →		#10	#21		#12	#13	#22	#23	#24	#25	#26	#27	#28	#29	#30
Hazardous Substance	Chemical Abstract Service Number	Statewide Default Background Levels	Residential Drinking Water Protection Criteria & RBSLs	Industrial and Commercial Drinking Water Protection Criteria & RBSLs	Groundwater Surface Water Interface Protection Criteria & RBSLs	Groundwater Contact Protection Criteria & RBSLs	Soil Volatilization to Indoor Air Inhalation Criteria & RBSLs	Infinite Source Volatile Soil Inhalation Criteria (VSIC) & RBSLs	Finite VSIC for 5 Meter Source Thickness	Finite VSIC for 2 Meter Source Thickness	Particulate Soil Inhalation Criteria & RBSLs	Industrial and Commercial II	Commercial III	Commercial IV	Soil Saturation Concentration Screening Levels
Xylenes (I)	1330207	NA	5,600	5,600	700	1.5E+5 (C)	1.5E+5 (C)	5.4E+7	6.5E+7	1.3E+8	1.3E+11	1.5E+5 (C)	1.5E+5 (C)	1.5E+5 (C)	1.5E+5
Zinc (B)	7440666	47,000	2.4E+6	5.0E+6	(G)	1.0E+9 (D)	NLV	NLV	NLV	NLV	ID	6.3E+8	6.9E+8	6.6E+8	NA

FOOTNOTES

FOR THE PART 201 CRITERIA/
PART 213 RISK-BASED SCREENING LEVELS
RRD OPERATIONAL MEMORANDUM No. 1

(A) Criterion is the state of Michigan drinking water standard established pursuant to Section 5 of 1976 PA 399, MCL 325.1005.

(B) Background, as defined in R 299.5701(b), may be substituted if higher than the calculated cleanup criterion. Background levels may be less than criteria for some inorganic compounds.

(C) Value presented is a screening level based on the chemical-specific generic soil saturation concentration (C_{sat}) since the calculated risk-based criterion is greater than C_{sat}. Concentrations greater than C_{sat} are acceptable cleanup criteria for this pathway where a site-specific demonstration indicates that free-phase material containing a hazardous substance is not present.

(D) Calculated criterion exceeds 100 percent, hence it is reduced to 100 percent or 1.0E+9 parts per billion (ppb).

(E) Criterion is the aesthetic drinking water value, as required by Section 20120a(5) of the Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (NREPA). A notice of aesthetic impact may be employed as an institutional control mechanism if groundwater concentrations exceed the aesthetic drinking water criterion, but do not exceed the applicable health-based drinking water value provided in the following table:

Hazardous Substance	Chemical Abstract Service Number	Residential Health-Based Drinking Water Value	Industrial-Commercial Health-Based Drinking Water Value
Aluminum	7429905	300	4,100
tertiary Amyl methyl ether	994058	910	2,600
Copper	7440508	1,400	4,000
Diethyl ether	60297	3,700	10,000
Ethylbenzene	100414	700	700
Iron	7439896	2,000	5,600
Manganese	7439965	860	2,500
Methyl-tert-butyl ether (MTBE)	1634044	240	690
Toluene	108883	1,000	1,000
1,2,4-Trimethylbenzene	95636	1,000	2,900
1,3,5-Trimethylbenzene	108678	1,000	2,900
Xylenes	1330207	10,000	10,000

(F) Criterion is based on adverse impacts to plant life and phytotoxicity.

(G) Groundwater surface water interface (GSI) criterion depends on the pH or water hardness, or both, of the receiving surface water. The final chronic value (FCV) for the protection of aquatic life shall be calculated based on

the pH or hardness of the receiving surface water. Where water hardness exceeds 400 mg $CaCO_3$/L, use 400 mg $CaCO_3$/L for the FCV calculation. The FCV formula provides values in units of ug/L or ppb. The generic GSI criterion is the lesser of the calculated FCV, the wildlife value (WV), and the surface water human non-drinking water value (HNDV). The soil GSI protection criteria for these hazardous substances are the greater of the 20 times the GSI criterion or the GSI soil-water partition values using the GSI criteria developed with the procedure described in this footnote.

Hazardous Substance	FCV Formula ug/L	FCV Conversion Factor (CF)	WV ug/L	HNDV ug/L
Acetate	EXP(0.2732*(pH) + 7.0362)	NA	NA	1.3E+6
Barium⊗	EXP(1.0629*(LnH)+1.1869)	NA	NA	1.6E+5
Beryllium	EXP(2.5279*(LnH)-10.7689)	NA	NA	1,200
Cadmium⊗	(EXP(0.7852*(LnH)-2.715))*CF	1.101672-((LnH)*(0.041838))	NA	130
Chromium (III) ⊗	(EXP(0.819*(LnH)+0.6848))*CF	0.86	NA	9,400
Copper	(EXP(0.8545*(LnH)-1.702)) *CF	0.96	NA	64,000
Lead⊗	(EXP(1.273*(LnH)-3.296))*CF	1.46203-((LnH)*(0.14571))	NA	190
Manganese	EXP(0.8784*(LnH)+3.5199)	NA	NA	59,000
Nickel	(EXP(0.846*(LnH)+0.0584))*CF	0.997	NA	2.1E+5
Pentachlorophenol	EXP(1.005*(pH)-5.134)	NA	NA	2.8
Zinc	(EXP(0.8473*(LnH)+0.884))*CF	0.986	NA	22,000

where,

EXP(x) = The base of the natural logarithm raised to power x (e^x).
LnH = The natural logarithm of water hardness in mg $CaCO_3$/L.
* = The multiplication symbol.
⊗ = The GSI criterion developed here may not be protective for surface water that is used as a drinking water source. Refer to footnote (X) for further guidance.

A spreadsheet that may be used to calculate GSI and GSI protection criteria for (G)-footnoted hazardous substances is available on the Department of Environmental Quality (DEQ) internet web site.

(H) Valence-specific chromium data (Cr III and Cr VI) shall be compared to the corresponding valence-specific cleanup criteria. If both Cr III and Cr VI are present in groundwater, the total concentration of both cannot exceed the drinking water criterion of 100 ug/L. If analytical data are provided for total chromium only, they shall be compared to the cleanup criteria for Cr VI. Cr III soil cleanup criterion for protection of drinking water can only be used at sites where groundwater is prevented from being used as a public water supply, currently and in the future, through an approved land or resource use restriction.

(I) Hazardous substance may exhibit the characteristic of ignitability as defined in 40 C.F.R. §261.21 (revised as of July 1, 2001), which is adopted by reference in these rules and is available for inspection at the DEQ, 525 West Allegan Street, Lansing, Michigan. Copies of the regulation may be purchased, at a cost as of the time of adoption of these

rules of $45, from the Superintendent of Documents, Government Printing Office, Washington, DC 20401 (stock number 869-044-00155-1), or from the DEQ, Remediation and Redevelopment Division (RRD), 525 West Allegan Street, Lansing, Michigan 48933, at cost.

(J) Hazardous substance may be present in several isomer forms. Isomer-specific concentrations shall be added together for comparison to criteria.

(K) Hazardous substance may be flammable or explosive, or both.

(L) Criteria for lead are derived using a biologically based model, as allowed for under Section 20120a(10) of the NREPA, and are not calculated using the algorithms and assumptions specified in pathway-specific rules. The generic residential drinking water criterion of 4 ug/L is linked to the generic residential soil direct contact criterion of 400 mg/kg. A higher concentration in the drinking water, up to the state action level of 15 ug/L, may be allowed as a site-specific remedy and still allow for drinking water use, under Section 20120a(2) of the NREPA if soil concentrations are appropriately lower than 400 mg/kg. If a site-specific criterion is approved based on this subdivision, a notice shall be filed on the deed for all property where the groundwater concentrations will exceed 4 ug/L to provide notice of the potential for unacceptable risk if soil or groundwater concentrations increase. Acceptable combinations of site-specific soil and drinking water concentrations are presented in the following table:

Acceptable Combinations of Lead in Drinking Water and Soil

Drinking Water Concentration (ug/L)	Soil Concentration (mg/kg)
5	386-395
6	376-385
7	376-385
8	366-375
9	356-365
10	346-355
11	336-345
12	336-345
13	326-335
14	316-325
15	306-315

(M) Calculated criterion is below the analytical target detection limit, therefore, the criterion defaults to the target detection limit.

(N) The concentrations of all potential sources of nitrate-nitrogen (e.g., ammonia-N, nitrite-N, nitrate-N) in groundwater that is used as a source of drinking water shall not, when added together, exceed the nitrate drinking water criterion of 10,000 ug/L. Where leaching to groundwater is a relevant pathway, soil concentrations of all potential sources of nitrate-nitrogen shall not, when added together, exceed the nitrate drinking water protection criterion of 2.0E+5 ug/kg.

(O) The concentration of all polychlorinated and polybrominated dibenzodioxin and dibenzofuran isomers present at a facility, expressed as an equivalent

concentration of 2,3,7,8-tetrachlorodibenzo-p-dioxin based upon their relative potency, shall be added together and compared to the criteria for 2,3,7,8-tetrachlorodibenzo-p-dioxin. The generic cleanup criteria for 2,3,7,8-tetrachlorodibenzo-p-dioxin are not calculated according to the algorithms presented in R 299.5714 to R 299.5726. The generic cleanup criteria are being held at the values that the DEQ has used since August 1998, in recognition of the fact that national efforts to reassess risks posed by dioxin are not yet complete. Until these studies are complete, it is premature to select a revised slope factor and/or reference dose for calculation of generic cleanup criteria.

(P) Amenable cyanide methods or method OIA-1677 shall be used to quantify cyanide concentrations for compliance with all groundwater criteria. Total cyanide methods or method OIA-1677 shall be used to quantify cyanide concentrations for compliance with soil criteria. Industrial-commercial direct contact criteria may not be protective of the potential for release of hydrogen cyanide gas. Additional land or resource use restrictions may be necessary to protect for the acute inhalation concerns associated with hydrogen cyanide gas.

(Q) Criteria for carcinogenic polycyclic aromatic hydrocarbons were developed using relative potential potencies to benzo(a)pyrene.

(R) Hazardous substance may exhibit the characteristic of reactivity as defined in 40 C.F.R. §261.23 (revised as of July 1, 2001), which is adopted by reference in these rules and is available for inspection at the DEQ, 525 West Allegan Street, Lansing, Michigan. Copies of the regulation may be purchased, at a cost as of the time of adoption of these rules of $45, from the Superintendent of Documents, Government Printing Office, Washington, DC 20401 (stock number 869-044-00155-1), or from the DEQ, RRD, 525 West Allegan Street, Lansing, Michigan 48933, at cost.

(S) Criterion defaults to the hazardous substance-specific water solubility limit.

(T) Refer to the federal Toxic Substances Control Act (TSCA), 40 C.F.R. §761, Subpart D and 40 C.F.R. §761, Subpart G, to determine the applicability of TSCA cleanup standards. Subpart D and Subpart G of 40 C.F.R. §761 (July 1, 2001) are adopted by reference in these rules and are available for inspection at the DEQ, 525 West Allegan Street, Lansing, Michigan. Copies of the regulations may be purchased, at a cost as of the time of adoption of these rules of $55, from the Superintendent of Documents, Government Printing Office, Washington, DC 20401, or from the DEQ, RRD, 525 West Allegan Street, Lansing, Michigan 48933, at cost. Alternatives to compliance with the TSCA standards listed below are possible under 40 C.F.R. §761 Subpart D. New releases may be subject to the standards identified in 40 C.F.R. §761, Subpart G. Use Part 201 soil direct contact cleanup criteria in the following table if TSCA standards are not applicable.

Land Use Category	TSCA, Subpart D Cleanup Standards	Part 201 Soil Direct Contact Cleanup Criteria
Residential & Commercial I	1,000 ppb, or 10,000 ppb if capped	4,000 ppb
Industrial & Commercial II	1,000 ppb, or 10,000 ppb if capped	16,000 ppb
Commercial III	1,000 ppb, or 10,000 ppb if capped	33,000 ppb
Commercial IV	1,000 ppb, or 10,000 ppb if capped	22,000 ppb

(U) Hazardous substance may exhibit the characteristic of corrosivity as defined in 40 C.F.R. §261.22 (revised as of July 1, 2001), which is adopted by reference in these rules and is available for inspection at the DEQ, 525 West Allegan Street, Lansing, Michigan. Copies of the regulation may be purchased, at a cost as of the time of adoption of these rules of $45, from the Superintendent of Documents, Government Printing Office, Washington, DC 20401 (stock number 869-044-00155-1), or from the DEQ, RRD, 525 West Allegan Street, Lansing, Michigan 48933, at cost.

(V) Criterion is the aesthetic drinking water value as required by Section 20120(a)(5) of the NREPA. Concentrations up to 200 ug/L may be acceptable, and still allow for drinking water use, as part of a site-specific cleanup under Section 20120a(2) of the NREPA.

(W) Concentrations of trihalomethanes in groundwater shall be added together to determine compliance with the Michigan drinking water standard of 80 ug/L. Concentrations of trihalomethanes in soil shall be added together to determine compliance with the drinking water protection criterion of 1,600 ug/kg.

(X) The GSI criterion shown in the generic cleanup criteria tables is not protective for surface water that is used as a drinking water source. For a groundwater discharge to the Great Lakes and their connecting waters or discharge in close proximity to a water supply intake in inland surface waters, the generic GSI criterion shall be the surface water human drinking water value (HDV) listed in the table in this footnote, except for those HDV indicated with an asterisk. For HDV with an asterisk, the generic GSI criterion shall be the lowest of the HDV, the WV, and the calculated FCV. See formulas in footnote (G). Soil protection criteria based on the HDV shall be as listed in the table in this footnote, except for those values with an asterisk. Soil GSI protection criteria based on the HDV shall be as listed in the table in this footnote, except for those values with an asterisk. Soil GSI protection criteria for compounds with an asterisk shall be the greater of 20 times the GSI criterion or the GSI soil-water partition values using the GSI criteria developed with the procedure described in this footnote.

Hazardous Substance	Chemical Abstract Service Number	Surface Water Human Drinking Water Values (HDV) (ug/L)	Soil GSI Protection Criteria for HDV (ug/kg)
Acrylonitrile	107131	2.0 (M); 0.87	100 (M); 17
Alachlor	15972608	3.5	91
Antimony	7440360	2	1,400
Arsenic	7440382	50	23,000
Atrazine	1912249	4.3	86
Barium	7440393	1,900*	*
Benzene	71432	12	240
bis(2-Chloroethyl)ether	111444	1 (M); 0.79	100 (M); 20
Bromate	15541454	10 (M); 0.5	200 (M); 10
Butyl benzyl phthalate	85687	6.9	13,000
Cadmium	7440439	2.5*	*
Carbon tetrachloride	56235	5.6	110
Chloride	16887006	50,000	1.0E+6
Chloroform	67663	77	1,500
Chromium (III)	16065831	120*	*
Cyanazine	21725462	2 (M); 0.93	200 (M); 40
3,3'-Dichlorobenzidine	91941	0.3 (M); 0.14	2,000 (M); 7.7
1,2-Dichloroethane	107062	6	120
1,1-Dichloroethylene	75354	24	480
1,2-Dichloropropane	78875	9.1	180
N,N-Dimethylacetamide	127195	700	14,000
1,4-Dioxane	123911	34	680
Ethylene dibromide	106934	0.05 (M); 0.006	20 (M); 1.0
Ethylene glycol	107211	56,000	1.1E+6
Heptachlor	76448	0.01 (M); 0.0017	NLL
beta-Hexachlorocyclohexane	319857	0.024	20 (M)
Hexachloroethane	67721	5.3	310
Isophorone	78591	310	6,200
Isopropyl alcohol	67630	28,000	5.6E+5
Lead	7439921	14*	*
Manganese	7439965	3600	72,000
Methyl-tert-butyl ether (MTBE)	1634044	100	2,000
Methylene chloride	75092	47	940
Mirex	2385855	0.02 (M); 1.6E-5	NLL
Molybdenum	7439987	120	2,400
Nitrobenzene	98953	4.7	330 (M); 94
Pentachlorophenol	87865	1.8*	*
1,2,4,5-Tetrachlorobenzene	95943	2.8	3,300
1,1,1,2-Tetrachloroethane	630206	19	380
1,1,2,2-Tetrachloroethane	79345	3.2	64
Tetrachloroethylene	127184	11	220
Tetrahydrofuran	109999	350	7,000
Thallium	7440280	2.0 (M); 1.2	2,300
1,1,2-Trichloroethane	79005	12	240
Trichloroethylene	79016	29	580

(Y) Source size modifiers shown in the following table shall be used to determine soil inhalation criteria for ambient air when the source size is not one-half acre. The modifier shall be multiplied by the generic soil inhalation criteria shown in the table of generic cleanup criteria to determine the applicable criterion.

Source Size sq. feet or acres	Modifier
400 sq feet	3.17
1000 sq feet	2.2
2000 sq feet	1.76
1/4 acre	1.15
1/2 acre	1
1 acre	0.87
2 acre	0.77
5 acre	0.66
10 acre	0.6
32 acre	0.5
100 acre	0.43

(Z) Mercury is typically measured as total mercury. The generic cleanup criteria, however, are based on data for different species of mercury. Specifically, data for elemental mercury, chemical abstract service (CAS) number 7439976, serve as the basis for the soil volatilization to indoor air criteria, groundwater volatilization to indoor air, and soil inhalation criteria. Data for methyl mercury, CAS number 22967926, serve as the basis for the GSI criterion; and data for mercuric chloride, CAS number 7487947, serve as the basis for the drinking water, groundwater contact, soil direct contact, and the groundwater protection criteria. Comparison to criteria shall be based on species-specific analytical data only if sufficient facility characterization has been conducted to rule out the presence of other species of mercury.

(AA) Comparison to these criteria may take into account an evaluation of whether the hazardous substances are adsorbed to particulates rather than dissolved in water and whether filtered groundwater samples were used to evaluate groundwater.

(BB) The state drinking water standard for asbestos is in units of fibers per milliliter of water (f/mL) longer than 10 millimicrons. Soil concentrations of asbestos are determined by polarized light microscopy.

(CC) Groundwater: The generic GSI criteria are based on the toxicity of unionized ammonia (NH_3); the criteria are 29 ug/L and 53 ug/L for cold water and warm water surface water, respectively. As a result, the GSI criterion shall be compared to the percent of the total ammonia concentration in the groundwater that will become NH_3 in the surface water. This percent NH_3 is a function of the pH and temperature of the receiving surface water and can be estimated using the following table,

taken from Emerson, et al., (Journal of the Fisheries Research Board of Canada, Volume 32(12):2382, 1975).

Percent NH_3 in Aqueous Ammonia Solutions for 0-30 ^oC and pH 6-10

Temp (^oF)	Temp (^oC)	pH 6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0
32.0	0	0.00827	0.0261	0.0826	0.261	0.820	2.55	7.64	20.7	45.3
33.8	1	0.00899	0.0284	0.0898	0.284	0.891	2.77	8.25	22.1	47.3
35.6	2	0.00977	0.0309	0.0977	0.308	0.968	3.00	8.90	23.6	49.4
37.4	3	0.0106	0.0336	0.106	0.335	1.05	3.25	9.60	25.1	51.5
39.2	4	0.0115	0.0364	0.115	0.363	1.14	3.52	10.3	26.7	53.5
41.0	5	0.0125	0.0395	0.125	0.394	1.23	3.80	11.1	28.3	55.6
42.8	6	0.0136	0.0429	0.135	0.427	1.34	4.11	11.9	30.0	57.6
44.6	7	0.0147	0.0464	0.147	0.462	1.45	4.44	12.8	31.7	59.5
46.4	8	0.0159	0.0503	0.159	0.501	1.57	4.79	13.7	33.5	61.4
48.2	9	0.0172	0.0544	0.172	0.542	1.69	5.16	14.7	35.3	63.3
50.0	10	0.0186	0.0589	0.186	0.586	1.83	5.56	15.7	37.1	65.1
51.8	11	0.0201	0.0637	0.201	0.633	1.97	5.99	16.8	38.9	66.8
53.6	12	0.0218	0.0688	0.217	0.684	2.13	6.44	17.9	40.8	68.5
55.4	13	0.0235	0.0743	0.235	0.738	2.30	6.92	19.0	42.6	70.2
57.2	14	0.0254	0.0802	0.253	0.796	2.48	7.43	20.2	44.5	71.7
59.0	15	0.0274	0.0865	0.273	0.859	2.67	7.97	21.5	46.4	73.3
60.8	16	0.0295	0.0933	0.294	0.925	2.87	8.54	22.8	48.3	74.7
62.6	17	0.0318	0.101	0.317	0.996	3.08	9.14	24.1	50.2	76.1
64.4	18	0.0343	0.108	0.342	1.07	3.31	9.78	25.5	52.0	77.4
66.2	19	0.0369	0.117	0.368	1.15	3.56	10.5	27.0	53.9	78.7
68.0	20	0.0397	0.125	0.396	1.24	3.82	11.2	28.4	55.7	79.9
69.8	21	0.0427	0.135	0.425	1.33	4.10	11.9	29.9	57.5	81.0
71.6	22	0.0459	0.145	0.457	1.43	4.39	12.7	31.5	59.2	82.1
73.4	23	0.0493	0.156	0.491	1.54	4.70	13.5	33.0	60.9	83.2
75.2	24	0.0530	0.167	0.527	1.65	5.03	14.4	34.6	62.6	84.1
77.0	25	0.0569	0.180	0.566	1.77	5.38	15.3	36.3	64.3	85.1
78.8	26	0.0610	0.193	0.607	1.89	5.75	16.2	37.9	65.9	85.9
80.6	27	0.0654	0.207	0.651	2.03	6.15	17.2	39.6	67.4	86.8
82.4	28	0.0701	0.221	0.697	2.17	6.56	18.2	41.2	68.9	87.3
84.2	29	0.0752	0.237	0.747	2.32	7.00	19.2	42.9	70.4	88.3
86.0	30	0.0805	0.254	0.799	2.48	7.46	20.3	44.6	71.8	89.0

The generic approach for estimating NH_3 assumes a default pH of 8 and default temperatures of 68°F and 85°F for cold water and warm water surface water, respectively. The resulting percent NH_3 is 3.8 percent and 7.2 percent for cold water and warm water, respectively. This default percentage shall be multiplied by the total ammonia-nitrogen (NH_3-N) concentration in the groundwater and the resulting NH_3 concentration

compared to the applicable GSI criterion. As an alternative, the maximum pH and temperature data from the specific receiving surface water can be used to estimate, from the table in this footnote, a lower percent unionized ammonia concentration for comparison to the generic GSI.

Soil: The generic soil GSI protection criteria for unionized ammonia are 580 ug/kg and 1,100 ug/kg for cold water and warm water surface water, respectively.

(DD) Hazardous substance causes developmental effects. Residential and commercial I direct contact criteria are protective of both prenatal and postnatal exposure. Industrial and commercial II, III and IV direct contact criteria are protective for a pregnant adult receptor.

(EE) The following are applicable generic GSI criteria as required by Section 20120a(15) of the NREPA.

Hazardous Substance	GSI (ug/L)	Notes
Phosphorus	1,000	Criteria applicable unless receiving water is a surface water that has a phosphorus waste load allocation or is an inland lake. In those cases, contact the department for applicable values.
Total dissolved solids (TDS)	5.0E+5	If TDS data are not available, the TDS criterion may be used a screening level for the sum of the concentrations of the following substances: Calcium, Chlorides, Iron, Magnesium, Potassium, Sodium, Sulfate.
Dissolved Oxygen (DO): Cold receiving waters Warm receiving waters	 ≥ 7,000 ≥ 5,000	Since a low level of DO can be harmful to aquatic life, the criterion represents a minimum level that on-site samples must exceed. This is in contrast to other criteria which represent "not to exceed" concentrations. DO criteria are not applicable if groundwater Carbonaceous Biochemical Oxygen Demand (CBOD) is less than 10,000 ug/L and groundwater ammonia concentration is less than 2,000 ug/L.

(FF) The chloride GSI criterion shall be 125 mg/l when the discharge is to surface waters of the state designated as public water supply sources or 50 mg/l when the discharge is to the Great Lakes or connecting waters. Chloride GSI criteria shall not apply for surface waters of the state that are not designated as a public water supply source, however, the total dissolved solids criterion is applicable.

(GG) Risk-based criteria are not available for methane due to insufficient toxicity data. An acceptable soil gas concentration (presented for both residential and commercial/industrial land uses) was derived utilizing 25 percent of the lower explosive level for methane. This equates to 1.25 percent or 8.4E+6 ug/m^3.

"ID" means insufficient data to develop criterion.

"NA" means a criterion or value is not available or, in the case of background and CAS numbers, not applicable.

"NLL" means hazardous substance is not likely to leach under most soil conditions.

"NLV" means hazardous substance is not likely to volatilize under most conditions.

Appendix E
Calculated Soil Gas and Indoor Air Screening Levels

3754 Ranchero drive
Ann Arbor, MI 48108
(734) 971-7080

PROJECT / PROPOSAL NAME / LOCATION: Tecumseh Products Company, Tecumseh, Michigan		PROJECT / PROPOSAL NO.
SUBJECT: Longterm Indoor Air Criteria		8070.06
PREPARED BY: S. Metz	DATE: 2/16/10, rev. 1 - 5/17/10	FINAL ☐
CHECKED BY: K. Saucier, rev. 1 - D. VanAntwerp	DATE: 2/18/10, rev. 1 - 5/17/10	REVISION x

Longterm Indoor Air Criteria (IAC) Calculation for Carcinogens (Residential):

IAC = (TR * AT * AIR) / (IURF * EF * ED)

Target Risk (TR) =	1.00E-05		
Average Time (AT) =	25,550	days	(70 years * 365 days/year)
Adjusted Inhalation Rate (AIR) =	1		(1 = residential, 2 = non-residential)
Inhalation Unit Risk Factor (URF) =	Chemical Specific	$(ug/m^3)^{-1}$	
Exposure Frequency (EF) =	350	days/year	(350 = residential, 250 = non-residential)
Exposure Duration (ED) =	30	years	(30 = residential, 25 = non-residential)

Longterm Indoor Air Criteria (IAC) Calculation for Carcinogens (Non-Residential):

IAC = (TR * AT * AIR) / (IURF * EF * ED)

Target Risk (TR) =	1.00E-05		
Average Time (AT) =	25,550	days	(70 years * 365 days/year)
Adjusted Inhalation Rate (AIR) =	2		(1 = residential, 2 = non-residential)
Inhalation Unit Risk Factor (URF) =	Chemical Specific	$(ug/m^3)^{-1}$	
Exposure Frequency (EF) =	250	days/year	(350 = residential, 250 = non-residential)
Exposure Duration (ED) =	25	years	(30 = residential, 25 = non-residential)

Longterm Indoor Air Criteria (IAC) Calculation for Non-Carcinogens (Residential):

IAC = (HQ * AT * RfC) / (EF * ED)

Hazard Quotient (HQ) =	1		
Average Time (AT) =	10,950	days	(30 years * 365 days/year)
Reference Concentration (RfC) =	Chemical Specific	(ug/m^3)	
Exposure Frequency (EF) =	350	days/year	(350 = residential, 250 = non-residential)
Exposure Duration (ED) =	30	years	(30 = residential, 25 = non-residential)

Longterm Indoor Air Criteria (IAC) Calculation for Non-Carcinogens (Non-Residential):

IAC = (HQ * AT * RfC) / (EF * ED)

Hazard Quotient (HQ) =	1		
Average Time (AT) =	9,125	days	(25 years * 365 days/year)
Reference Concentration (RfC) =	Chemical Specific	(ug/m^3)	
Exposure Frequency (EF) =	250	days/year	(350 = residential, 250 = non-residential)
Exposure Duration (ED) =	25	years	(30 = residential, 25 = non-residential)

RMT
3754 Ranchero drive
Ann Arbor, MI 48108
(734) 971-7080

PROJECT / PROPOSAL NAME / LOCATION: Tecumseh Products Company, Tecumseh, Michigan		PROJECT / PROPOSAL NO.
SUBJECT: Longterm Indoor Air Criteria		8070.06
PREPARED BY: S. Metz	DATE: 2/16/10, rev. 1 - 5/17/10	FINAL ☐
CHECKED BY: K. Saucier, rev. 1 - D. VanAntwerp	DATE: 2/18/10, rev. 1 - 5/17/10	REVISION x

Chemical Specific Values

Compound	Conversion Factor (ug/m^3 to ppbv)	Unit Risk Factor $(ug/m^3)^{-1}$	Reference Concentration (ug/m^3)	Data Source for URF and RfC Values (1)
1,1-Dichloroethane	0.25	1.60E-06	500	RSLs (URF)/ JEM (RfC)
1,2-Dichloroethane	0.25	2.60E-05	2400	IRIS (URF) / RSLs (RfC)
1,1-Dichloroethene	0.25		200	IRIS
cis-1,2-Dichloroethene	0.25		35	JEM
trans-1,2-Dichloroethene	0.25		60	RSLs
Tetrachloroethene	0.15	5.90E-06	270	RSLs
1,1,1-Trichloroethane	0.18		5000	IRIS
Trichloroethene	0.19	2.00E-06		RSLs
Vinyl Chloride	0.39	8.80E-06	100	IRIS
2-Butanone (MEK)	ND		5000	IRIS
Trichlorofluoromethane	ND		700	RSLs

1) See oringinal calculation sheets included in the March 2010 Preliminary Off-Sits Soil Gas Sampling Workplan for a summary of availible toxicity data. IRIS = USEPA Integrated Risk Information System, RSLs = USEPA Regional Screening Levels for Chemical Contaiminants at Superfund Sites, JEM = USEPA Johnson and Ettinger Model Spreadsheet

Calculated Longterm Residential Indoor Air Criteria

Compound	IAC for Carcinogens (ug/m^3)	IAC for Non-Carcinogens (ug/m^3)	Critical IAC (ug/m^3)	Critical IAC (ppbv)
2-Butanone (MEK)	NA	5214	5214	1735
1,1-Dichloroethane	15	521	15	3.8
1,2-Dichloroethane	0.94	2503	0.94	0.23
1,1-Dichloroethene	NA	209	209	52
cis-1,2-Dichloroethene	NA	37	37	9.1
trans-1,2-Dichloroethene	NA	63	63	16
Tetrachloroethene	4.1	282	4.124	0.62
1,1,1-Trichloroethane	NA	5214	5214	939
Trichloroethene	12	NA	12	2.3
Trichlorofluoromethane	NA	730	730	128
Vinyl Chloride	2.8	104	2.8	1.1

Calculated Longterm Non-Residential Indoor Air Criteria

Compound	IAC for Carcinogens (ug/m^3)	IAC for Non-Carcinogens (ug/m^3)	Critical IAC (ug/m^3)	Critical IAC (ppbv)
2-Butanone (MEK)	NA	7300	7300	2430
1,1-Dichloroethane	51	730	51	13
1,2-Dichloroethane	3.1	3504	3.1	0.79
1,1-Dichloroethene	NA	292	292	73
cis-1,2-Dichloroethene	NA	51	51	13
trans-1,2-Dichloroethene	NA	88	88	22
Tetrachloroethene	14	394	14	2.1
1,1,1-Trichloroethane	NA	7300	7300	1314
Trichloroethene	41	NA	41	7.8
Trichlorofluoromethane	NA	1022	1022	179
Vinyl Chloride	9.3	146	9.3	3.6

RMT

3754 Ranchero drive
Ann Arbor, MI 48108
(734) 971-7080

SHEET__1___OF___1__

PROJECT / PROPOSAL NAME / LOCATION: Tecumseh Products Company, Tecumseh, Michigan		PROJECT / PROPOSAL NO.
SUBJECT: Generic Soil Gas Screening Levels		8070.06
PREPARED BY: S. Metz	DATE: 2/22/10, rev. 1 - 5/17/10	FINAL ☐
CHECKED BY: C. Daining, rev. 1 - D. VanAntwerp	DATE: 3/12/10, rev. 1 - 5/17/10	REVISION x

Generic Soil Gas Screening Level (SGSL) Calculation:

SGSL = IAC / α

Indoor Air Criteria (IAC) = Chemical Specific (ug/m^3 or ppbv)
Sub-Slab Attenuation Factor (α) = 0.02 (Default values recommended by MDNRE are 0.02 for sub-slab or 0.002 for deep)
DEEP Attenuation Factor (α) = 0.002

Calculated Generic Residential Sub-Slab Soil Gas Screening Levels

Compound	Residential Indoor Air Criteria (ug/m^3)	Residential Indoor Air Criteria (ppbv)	Residential Sub-Slab Soil Gas Screening Level (ug/m^3)	Residential Sub-Slab Soil Gas Screening Level (ppbv)
1,1-Dichloroethane	15	3.8	760	190
1,2-Dichloroethane	0.94	0.23	47	12
1,1-Dichloroethene	209	52	10,429	2,607
cis-1,2-Dichloroethene	37	9.1	1,825	456
trans-1,2-Dichloroethene	63	16	3,129	782
Tetrachloroethene	4.1	0.62	206	31
1,1,1-Trichloroethane	5,214	939	260,714	46,929
Trichloroethene	12	2.3	608	116
Vinyl Chloride	2.8	1.1	138	54

Calculated Generic Non-Residential Sub-Slab Soil Gas Screening Levels

Compound	Non-Residential Indoor Air Criteria (ug/m^3)	Non-Residential Indoor Air Criteria (ppbv)	Non-Residential Sub-Slab Soil Gas Screening Level (ug/m^3)	Non-Residential Sub-Slab Soil Gas Screening Level (ppbv)
1,1-Dichloroethane	51	13	2,555	639
1,2-Dichloroethane	3.1	0.79	157	39
1,1-Dichloroethene	292	73	14,600	3,650
cis-1,2-Dichloroethene	51	13	2,555	639
trans-1,2-Dichloroethene	88	22	4,380	1,095
Tetrachloroethene	14	2.1	693	104
1,1,1-Trichloroethane	7300	1314	365,000	65,700
Trichloroethene	41	7.8	2,044	388
Vinyl Chloride	9.3	3.6	465	180

Calculated Generic Residential DEEP Soil Gas Screening Levels

Compound	Residential Indoor Air Criteria (ug/m^3)	Residential Indoor Air Criteria (ppbv)	Residential DEEP Soil Gas Screening Level (ug/m^3)	Residential DEEP Soil Gas Screening Level (ppbv)
1,1-Dichloroethane	15	3.8	7,604	1,901
1,2-Dichloroethane	0.94	0.23	468	117
1,1-Dichloroethene	209	52	104,286	26,071
cis-1,2-Dichloroethene	37	9.1	18,250	4,563
trans-1,2-Dichloroethene	63	16	31,286	7,821
Tetrachloroethene	4.1	0.62	2,062	309
1,1,1-Trichloroethane	5,214	939	2,607,143	469,286
Trichloroethene	12	2.3	6,083	1,156
Vinyl Chloride	2.8	1.1	1,383	539

Calculated Generic Non-Residential DEEP Soil Gas Screening Levels

Compound	Non-Residential Indoor Air Criteria (ug/m^3)	Non-Residential Indoor Air Criteria (ppbv)	Non-Residential DEEP Soil Gas Screening Level (ug/m^3)	Non-Residential DEEP Soil Gas Screening Level (ppbv)
1,1-Dichloroethane	51	13	25,550	6,388
1,2-Dichloroethane	3.1	0.79	1,572	393
1,1-Dichloroethene	292	73	146,000	36,500
cis-1,2-Dichloroethene	51	13	25,550	6,388
trans-1,2-Dichloroethene	88	22	43,800	10,950
Tetrachloroethene	14	2.1	6,929	1,039
1,1,1-Trichloroethane	7300	1314	3,650,000	657,000
Trichloroethene	41	7.8	20,440	3,884
Vinyl Chloride	9.3	3.6	4,650	1,800

3754 Ranchero drive
Ann Arbor, MI 48108
(734) 971-7080

SHEET__1___OF___1__

PROJECT / PROPOSAL NAME / LOCATION: Tecumseh Products Company, Tecumseh, Michigan		PROJECT / PROPOSAL NO.
SUBJECT: Site Specific Soil Gas Screening Level		8070.06
PREPARED BY: S. Metz	DATE: 7/9/10	FINAL x
CHECKED BY: D. VanAntwerp	DATE: 7/13/10	REVISION ☐

Site Specific DEEP Soil Gas Screening Level (SGC) Calculation:

SGSL = IAC / α

Indoor Air Criteria (IAC) = Chemical Specific (ug/m^3 or ppbv)
JEM DEEP Attenuation Factor (α) = 0.003 Calculated using Johnson Ettinger Model Spreadsheet (v. 3.1) - Attached

Calculated Site Specific Residential DEEP Soil Gas Screening Levels

Compound	Residential Indoor Air Criteria (ug/m^3)	Residential Indoor Air Criteria (ppbv)	Residential DEEP Soil Gas Screening Level (ug/m^3)	Residential DEEP Soil Gas Screening Level (ppbv)
1,1-Dichloroethane	15	3.8	5,069	1,267
1,2-Dichloroethane	0.94	0.23	312	78
1,1-Dichloroethene	209	52	69,524	17,381
cis-1,2-Dichloroethene	37	9.1	12,167	3,042
trans-1,2-Dichloroethene	63	16	20,857	5,214
Tetrachloroethene	4.1	0.62	1,375	206
1,1,1-Trichloroethane	5,214	939	1,738,095	312,857
Trichloroethene	12	2.3	4,056	771
Vinyl Chloride	2.8	1.1	922	359

Calculated Site Specific Non-Residential DEEP Soil Gas Screening Levels

Compound	Non-Residential Indoor Air Criteria (ug/m^3)	Non-Residential Indoor Air Criteria (ppbv)	Non-Residential DEEP Soil Gas Screening Level (ug/m^3)	Non-Residential DEEP Soil Gas Screening Level (ppbv)
1,1-Dichloroethane	51	13	17,033	4,258
1,2-Dichloroethane	3.1	0.79	1,048	262
1,1-Dichloroethene	292	73	97,333	24,333
cis-1,2-Dichloroethene	51	13	17,033	4,258
trans-1,2-Dichloroethene	88	22	29,200	7,300
Tetrachloroethene	14	2.1	4,619	693
1,1,1-Trichloroethane	7300	1314	2,433,333	438,000
Trichloroethene	41	7.8	13,627	2,589
Vinyl Chloride	9.3	3.6	3,100	1,200

SG-SCREEN
Version 3.1; 02/04

Reset to Defaults

Soil Gas Concentration Data

ENTER Chemical CAS No. (numbers only, no dashes)	ENTER Soil gas conc., C_g ($\mu g/m^3$)	OR	ENTER Soil gas conc., C_g (ppmv)	Chemical
79016			8.00E-01	Trichloroethylene

MORE ↓

ENTER Depth below grade to bottom of enclosed space floor, L_F (15 or 200 cm)	ENTER Soil gas sampling depth below grade, L_s (cm)	ENTER Average soil temperature, T_S (^oC)	ENTER Vadose zone SCS soil type (used to estimate soil vapor permeability)	OR	ENTER User-defined vadose zone soil vapor permeability, k_v (cm^2)
200	200	10	S		

MORE ↓

ENTER Vadose zone SCS soil type (Lookup Soil Parameters)	ENTER Vadose zone soil dry bulk density, ρ_b^A (g/cm^3)	ENTER Vadose zone soil total porosity, n^V (unitless)	ENTER Vadose zone soil water-filled porosity, θ_w^V (cm^3/cm^3)	ENTER Average vapor flow rate into bldg. (Leave blank to calculate) Q_{soil} (L/m)
S	1.66	0.375	0.054	

MORE ↓

ENTER Averaging time for carcinogens, AT_C (yrs)	ENTER Averaging time for noncarcinogens, AT_{NC} (yrs)	ENTER Exposure duration, ED (yrs)	ENTER Exposure frequency, EF (days/yr)
70	30	30	350

END

CHEMICAL PROPERTIES SHEET

Diffusivity in air, D_a (cm^2/s)	Diffusivity in water, D_w (cm^2/s)	Henry's law constant at reference temperature, H (atm-m^3/mol)	Henry's law constant reference temperature, T_R (^oC)	Enthalpy of vaporization at the normal boiling point, $\Delta H_{v,b}$ (cal/mol)	Normal boiling point, T_B (^oK)	Critical temperature, T_C (^oK)	Unit risk factor, URF ($\mu g/m^3)^{-1}$	Reference conc., RfC (mg/m^3)	Molecular weight, MW (g/mol)
7.90E-02	9.10E-06	1.03E-02	25	7,505	360.36	544.20	2.0E-06	0.0E+00	131.39

END

INTERMEDIATE CALCULATIONS SHEET

Source-building separation, L_T (cm)	Vadose zone soil air-filled porosity, θ_a^V (cm^3/cm^3)	Vadose zone effective total fluid saturation, S_{te} (cm^3/cm^3)	Vadose zone soil intrinsic permeability, k_i (cm^2)	Vadose zone soil relative air permeability, k_{rg} (cm^2)	Vadose zone soil effective vapor permeability, k_v (cm^2)	Floor-wall seam perimeter, X_{crack} (cm)	Soil gas conc. ($\mu g/m^3$)	Bldg. ventilation rate, $Q_{building}$ (cm^3/s)
1	0.321	0.003	9.92E-08	0.998	9.91E-08	4,000	4.52E+03	2.54E+04

Area of enclosed space below grade, A_B (cm^2)	Crack-to-total area ratio, η (unitless)	Crack depth below grade, Z_{crack} (cm)	Enthalpy of vaporization at ave. soil temperature, $\Delta H_{v,TS}$ (cal/mol)	Henry's law constant at ave. soil temperature, H_{TS} (atm-m^3/mol)	Henry's law constant at ave. soil temperature, H'_{TS} (unitless)	Vapor viscosity at ave. soil temperature, μ_{TS} (g/cm-s)	Vadose zone effective diffusion coefficient, D^{eff}_V (cm^2/s)	Diffusion path length, L_d (cm)
1.80E+06	2.22E-04	200	8,557	4.78E-03	2.06E-01	1.75E-04	1.28E-02	1

Convection path length, L_p (cm)	Source vapor conc., C_{source} ($\mu g/m^3$)	Crack radius, r_{crack} (cm)	Average vapor flow rate into bldg., Q_{soil} (cm^3/s)	Crack effective diffusion coefficient, D^{crack} (cm^2/s)	Area of crack, A_{crack} (cm^2)	Exponent of equivalent foundation Peclet number, exp(Pe^f) (unitless)	Infinite source indoor attenuation coefficient, α (unitless)	Infinite source bldg. conc., $C_{building}$ ($\mu g/m^3$)
200	4.52E+03	0.10	6.84E+01	1.28E-02	4.00E+02	1.55E+58	2.69E-03	1.21E+01

Unit risk factor, URF $(\mu g/m^3)^{-1}$	Reference conc., RfC (mg/m^3)
2.0E-06	NA

END

Site specific attenuation factor is 0.00269, round up to 0.003

INCREMENTAL RISK CALCULATIONS:

Incremental risk from vapor intrusion to indoor air, carcinogen (unitless)	Hazard quotient from vapor intrusion to indoor air, noncarcinogen (unitless)
1.0E-05	NA

MESSAGE SUMMARY BELOW:

MESSAGE: Risk/HQ or risk-based soil concentration is based on a route-to-route extrapolation.

END

Appendix F
Soil Sampling and Preservation Methods

EPA Method 5035

Field Preservation Kit

samplesmart

Collecting soil samples for volatile organic compounds can become very expensive and cumbersome. The **o2si** 5035 **samplesmart** kit provides significant cost savings. How? The **o2si samplesmart** kit saves time and money by reducing collection times and increasing holding times. For example, **o2si**'s 5035 **samplesmart** kit provides holding times up to 14 days. This simplifies work schedules and shipping requirements. Unlike other soil sampling kits, the need to weigh methanol in the field is eliminated through the introduction of methanol provided in a uniquely sealed Teflon® tube. Finally, the design of the **o2si** 5035 **samplesmart** kit requires only one label for each kit, thereby reducing collection time.

Provide Significant Cost Savings

- Meets CLP Sample Collecting Guidelines Options 1 and 2
- Meets EPA Method 5035 Sampling Requirements
- Eliminates Field Weighing
- Decreases Collection Time
- Increases Productivity in the Field and Laboratory
- Increases Holding Times
- Eliminates Multiple Labeling
- Reduces Laboratory Prep Cost

o2si smart solutions®

P.O. Box 30712 • Charleston, SC 29417
www.o2si.com • Phone: 843.763.4884 • Fax: 843.766.9182

o2si smart solutions is an ISO 9001:2000 certified company.

EPA Method 5035
Field Preservation Kit

Historically, the disadvantages of field preservation has been handling the vials, field weighing the methanol vial, methanol loss, and packaging the vials for shipping. The **o2si** 5035 **samplesmart** kit overcomes these problems!

Each Kit Contains:

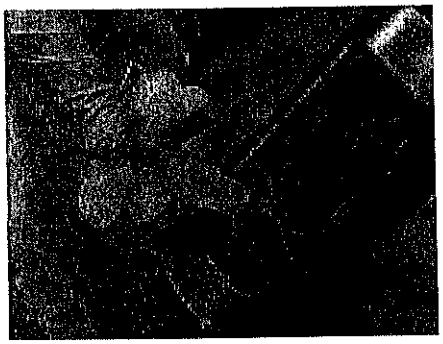
- Disposable syringe sampling device, two pre-weighed low-level vials, one pre-weighed high-level vial, and an evaporative loss vial.
- Methanol preservative in a sealed Teflon® tube.
  - Eliminates the need for field weighing.
  - No chance for methanol loss prior to sampling.
  - Methanol can be supplied with surrogates to provide the most rigorous surrogate criteria.
  - Weight of methanol already added to methanol vial weight.
- Plastic rack which allows access to vials without having to remove them. The weatherproof rack holds the vials upright and allows all the lids to be easily removed and easily filled.
- Bar coded label with a unique number with corresponding numbers on each vial. A pull tab allows the bar code to be put on the chain-of-custody form.
  - Complete only one label for each kit instead of labeling each container.
- Sampling instructions printed on each kit.

Confidence Factor:

- All 5035 **samplesmart** kits include certified pre-cleaned vials that are reanalyzed by EPA Method 5035 heated-purge specifications following preservation to ensure cleanliness. A certificate of analysis is available upon request.
- Methanol is purge-and-trap grade that has been analyzed after packaging the tubes.
- All vials are tared with a calibrated balance checked against NIST traceable weights each day before use.
- Plastic rack provides excellent protection against breakage during shipping.

Kit Instructions

5035 samplesmart T-Handle



Step 1:
With the plunger seated in the handle, push the syringe into the soil until the sample chamber is full. (This is equivalent to approximately 5 grams of soil.)

Step 2:
Remove the caps from all four vials.

Step 3:
Rotate the plunger, which is located in the handle, 90 degrees until it is aligned with the slots in the body. Place the end of the syringe into the vial to be filled and push the plunger down to remove the soil inside the syringe. Repeat for each vial.

Step 4:
Using scissors, cut the Teflon® methanol tube and pour into the high-level amber vial (vial number 3).

Step 5:
Tightly cap all four vials.

Step 6:
Complete the collection information on the label provided.

Step 7:
Place the kit upright in a cooler of ice for shipment to the laboratory.

o2si
smart solutions

P.O. Box 30712 • Charleston, SC 29417
www.o2si.com • Phone: (843) 763-4884 • Fax: (843) 766-9182

Disposable En Core® Sampler

En Novative Technologies, Inc.
1795 Industrial Drive
Green Bay, WI 54302
Phone: 920-465-3960 • Fax: 920-465-3963
Toll Free: 888-411-0757
www.ennovativetech.com

Sampling Procedures

Using The En Core® T-Handle

NOTE:

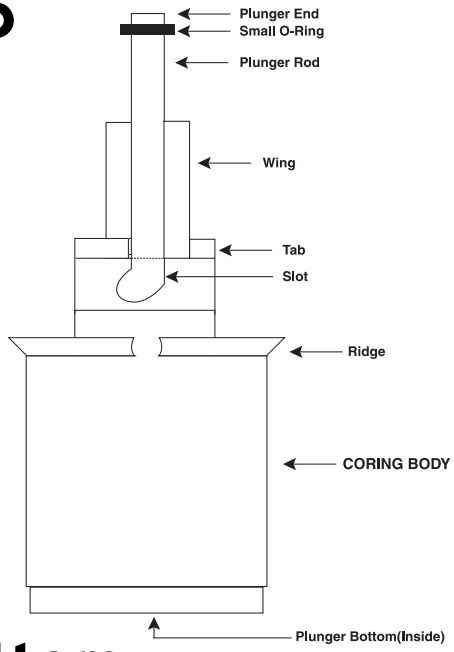
1. En Core® Sampler is a SINGLE USE device. It cannot be cleaned and/or reused.

2. En Core® Sampler is designed to store soil. Do not use En Core Sampler to store solvent or free product!

3. En Core® Sampler must be used with En Core® T-Handle and/or En Core® Extrusion Tool exclusively. (These items are sold separately.)

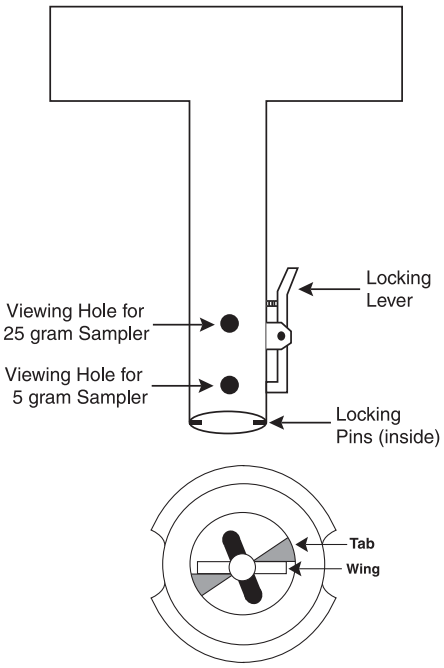
En Core®

Top



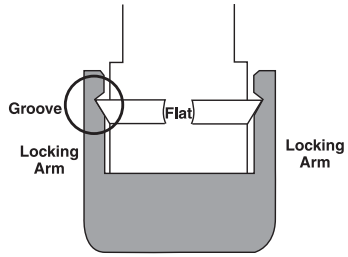
Bottom

En Core® T-Handle



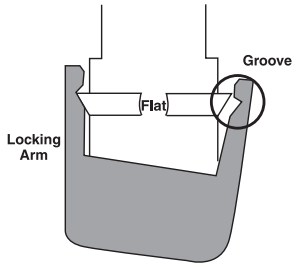
Sampler Correctly Capped

(Locking arm grooves seated over coring body ridge.)



Sampler Incorrectly Capped

(Cap appears crooked; locking arm grooves not fully seated over coring body ridge.)



BEFORE TAKING SAMPLE:

1. Hold **coring body** and push **plunger rod** down until **small o-ring** rests against **tabs**. This will assure that plunger moves freely.

2. Depress **locking lever** on En Core T-Handle. Place coring body, **plunger end first,** into open end of T-Handle, *aligning the (2)* ***slots*** *on the coring body with the (2)* ***locking pins*** *in the T-Handle*. Twist coring body clockwise to lock pins in slots. Check to ensure Sampler is locked in place. Sampler is ready for use.

TAKING SAMPLE:

3. Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body (ensure that plunger bottom is in position). Using T-Handle, push Sampler into soil until coring body is completely full. When full, small o-ring will be centered in T-Handle **viewing hole**. Remove Sampler from soil. Wipe excess soil from coring body exterior.

4. Cap coring body while it is still on T-handle. *Push* **cap** over **flat** area of **ridge** *and twist* to lock **cap** in place. **CAP MUST BE SEATED TO SEAL SAMPLER (see diagram)**.

PREPARING SAMPLER FOR SHIPMENT:

5. Remove the capped Sampler by depressing locking lever on T-Handle while twisting and pulling Sampler from T-Handle.

6. Lock plunger by rotating extended plunger rod fully counter-clockwise until **wings** rest firmly against tabs (see plunger diagram).

7. Attach completed tear-off label (from En Core Sampler bag) to cap on coring body.

8. Return full En Core Sampler to zipper bag. Seal bag and put on ice.

Disposable En Core® Sampler

EXTRUSION PROCEDURES

USING THE En Core® EXTRUSION TOOL

CAUTION! Always use the Extrusion Tool to extrude soil from the En Core Sampler. If the Extrusion Tool is not used, the Sampler may fragment, causing injury.

1. To attach En Core Sampler to En Core Extrusion Tool: Depress locking lever on Extrusion Tool and place Sampler, plunger end first, into open end of Extrusion Tool, aligning slots on coring body with pins in Extrusion Tool. Turn coring body clockwise until it locks into place. Release locking lever.

2. Rotate and gently push Extrusion Tool plunger knob clockwise until plunger slides over wings of coring body. (When properly positioned plunger will not rotate further.)

3. Hold Extrusion Tool with capped Sampler pointed upward so soil does not fall out when cap is removed. Remove cap from Sampler by rotating cap until locking arms are aligned with the flat area of ridge and pull cap off. To release soil core push down on plunger knob of En Core Extrusion Tool. Remove and properly dispose of En Core Sampler.

Warranty and Disclaimers

IMPORTANT: FAILURE TO USE THE EN CORE® SAMPLER IN COMPLIANCE WITH THE WRITTEN INSTRUCTIONS PROVIDED HEREIN VOIDS ALL EXPRESS AND IMPLIED WARRANTIES, INCLUDING WARRANTY OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

PRINCIPLE OF USE. The En Core Sampler Cartridge System is a volumetric sampling system designed to collect, store and deliver a soil sample. The En Core Sampler comes in two sizes for sample volumes of approximately 25 or 5 grams. There are four components: the cartridge with a movable plunger; a cap with two locking arms; a T-handle (purchased separately); and an extrusion handle (purchased separately). NOTE: The En Core Sampler is designed to store soil. It is not designed to store solvent or free product.

The soil is stored in a sealed headspace-free state. The seals are achieved by three special Viton® * o-rings, two located on the plunger and one on the cap of the Sampler. At no time and under no condition should these o-rings be removed or disturbed.

QUALITY CONTROL. The cartridge is sealed in an airtight package to prevent contamination prior to use. Due to the stringent quality control requirements associated with the use of this system, the disposable cartridge is designed to be used only once.

WARRANTY. En Novative Technologies, Inc. ("En Novative Technologies") warrants that the En Core Sampler shall perform consistent with the research conducted under En Novative Technologies' approval, within thirty (30) days from the date of delivery, provided that the Customer gives En Novative Technologies prompt notice of any defect or failure to perform and satisfactory proof thereof. THIS WARRANTY DOES NOT APPLY TO THE FOLLOWING, AS SOLELY DETERMINED BY EN NOVATIVE TECHNOLOGIES: (a) Damage caused by accident, abuse, mishandling or dropping; (b)Samplers that have been opened, taken apart or mishandled; (c)Samplers not used in accordance with the directions; and (d)Damages exceeding the cost of the sampler. Seller warrants that all En Core Samplers shall be free from defects in title. THE FOREGOING WARRANTIES ARE IN LIEU OF ALL OTHER WARRANTIES, WHETHER ORAL, WRITTEN, EXPRESSED, IMPLIED OR STATUTORY, INCLUDING ANY INFORMATION PROVIDED BY SALES REPRESENTATIVES OR IN MARKETING LITERATURE. IMPLIED WARRANTIES OF FITNESS AND MERCHANTABILITY SHALL NOT APPLY. En Novative Technologies' warranty obligations and Customer's remedies, except as to title, are solely and exclusively as stated herein.

LIMITATION OF LIABILITY. IN NO EVENT SHALL EN NOVATIVE TECHNOLOGIES BE LIABLE FOR ANTICIPATED PROFITS, INCIDENTAL, SPECIAL OR CONSEQUENTIAL DAMAGES, INCLUDING, BUT NOT LIMITED TO, DAMAGES FOR LOSS OF REVENUE, DOWN TIME, REMEDIATION ACTIVITIES, REMOBILIZATION OR RESAMPLING, COST OF CAPITAL, SERVICE INTERRUPTION OR FAILURE OF SUPPLY, LIABILITY OF CUSTOMER TO A THIRD PARTY, OR FOR LABOR, OVERHEAD, TRANSPORTATION, SUBSTITUTE SUPPLY SOURCES OR ANY OTHER EXPENSE, DAMAGE OR LOSS, INCLUDING PERSONAL INJURY OR PROPERTY DAMAGE. En Novative Technologies' liability on any claim of any kind shall be replacement of the En Core Sampler or refund of the purchase price. En Novative Technologies shall not be liable for penalties of any description whatsoever. In the event the En Core Sampler will be utilized by Customer on behalf of a third party, such third party shall not occupy the position of a third-party beneficiary of the obligation or warranty provided by En Novative Technologies, and no such third party shall have the right to enforce same. All claims must be brought within one (1) year of shipment, regardless of their nature.

En Novative Technologies, Inc.
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Green Bay, WI 54302
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Toll Free: 888-411-0757
www.ennovativetech.com

* Viton® is a registered trademark of DuPont Dow Elastomers.

Soil Sampling Tools 5035

Lock N' Load™ Handles and Syringes

The Lock N' Load™ handle and soil syringe system is the easiest way to collect undisturbed, measured soil cores for field preservation techniques for EPA Method 5035.

Lock N' Load™ Handle and Syringes can also be used in the lab. They are great for subsampling from brass sleeves or soil jars to 40 mL vials or auto-sampler extraction.

FEATURES

- **Handle locks at 5 and 10 gram settings with one easy turn.**
- **Beveled edge syringe is stronger than cut syringes and fits in the neck of 40 mL glass vials.**
- **Turn handle and push to dispense soil into the vial. Soil dispensing is done without having to remove the syringe.**
- **Strong, Accurate, Easy and Economical**
- **Handles and Syringes sold separately**
- **Lock N' Load™ Syringes are packaged 50/case**

Lock N' Load handle and soil syringe

OPERATION

S.O.P. For taking field soil samples or sub-sampling in the lab:

1. Insert Lock N' Load™ Syringe into Lock N' Load™ Handle at base opening. Turn the locking portion of the syringe into the O gram setting. Remove end cap from the Lock N' Load™ Syringe. Position the Lock N' Load™ Handle to the desired soil sample volume (5 grams or 10 grams)*. To do this, slide the slot portion of the handle down the fitted track, then turn the handle ¼ turn right at the desired setting. Push the syringe into the soil until the plunger portion of the syringe makes contact with the base of the Lock N' Load™ Handle. Transfer the soil from the syringe into a 40 ml vial** by turning the Lock N' Load™ Handle ¼ turn left (back to the fitted track) and pushing down
2. Cap the vial and store the sample at 4°C until time of analysis.

*USEPA Method 5035 recommends 5 grams of soil for low level analysis (with 5 mL of sodium solution) and 10 grams of soil for medium to high level analysis (with 10 mL of methanol). Sample amounts and preservative types may vary between regulatory agencies.

CALL GEOTECH TODAY (800) 833-7958

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email: sales@geotechenv.com website: www.geotechenv.com

Lock N Load.indd 11/20/08

Appendix G
Standard Field Forms

RMT

ENVIRONMENT • ENERGY • ENGINEERING

PROJECT NAME: TPC -

PROJECT NUMBER: 00-08070.

PROJECT MANAGER: Graham Crockford

SITE LOCATION: Tecumseh, Michigan

DATES OF FIELDWORK: TO

PURPOSE OF FIELDWORK:

WORK PERFORMED BY:

SIGNED DATE CHECKED BY DATE

RMT

GENERAL NOTES

PROJECT NAME: TPC -	DATE:	TIME ARRIVED:
PROJECT NUMBER: 00-08070.	AUTHOR:	TIME LEFT:

WEATHER		
TEMPERATURE: _____ °F	WIND: _____ MPH	VISIBILITY: _____

WORK / SAMPLING PERFORMED

PROBLEMS ENCOUNTERED	CORRECTIVE ACTION TAKEN

COMMUNICATION		
NAME	REPRESENTING	SUBJECT / COMMENTS

INVESTIGATION DERIVED WASTE SUMMARY		
WASTE MATRIX	QUANTITY	COMMENTS

SIGNED DATE

CHECKED BY DATE

RMT

EQUIPMENT SUMMARY

PROJECT NAME:	TPC -	SAMPLER NAME:
PROJECT NO.:	00-08070.	

WATER LEVEL MEASUREMENTS COLLECTED WITH:

NAME AND MODEL OF INSTRUMENT

SERIAL NUMBER (IF APPLICABLE)

PRODUCT LEVEL MEASUREMENTS COLLECTED WITH:

NA

NAME AND MODEL OF INSTRUMENT

SERIAL NUMBER (IF APPLICABLE)

DEPTH TO BOTTOM OF WELL MEASUREMENTS COLLECTED WITH:

NA

NAME AND MODEL OF INSTRUMENT

SERIAL NUMBER (IF APPLICABLE)

PURGING METHOD

NAME AND MODEL OF PUMP OR TYPE OF BAILER

SERIAL NUMBER (IF APPLICABLE)

SAMPLING METHOD

NAME AND MODEL OF PUMP OR TYPE OF BAILER

SERIAL NUMBER (IF APPLICABLE)

NAME AND MODEL OF FILTERATION DEVICE

FILTER TYPE AND SIZE

☐ LOW-FLOW SAMPLING EVENT

TUBING TYPE

PURGE WATER DISPOSAL METHOD

☐ GROUND ☐ DRUM ☐ POTW ☐ POLYTANK ☐ OTHER _____

DECONTAMINATION AND FIELD BLANK WATER SOURCE

STORE BOUGHT

POTABLE WATER SOURCE

STORE BOUGHT

DI WATER SOURCE

SIGNED DATE

CHECKED BY DATE

RMT

WATER QUALITY METER CALIBRATION LOG

PROJECT NAME:	TPC -	MODEL:	SAMPLER: SN
PROJECT NO.:	00-08070.	SERIAL #:	DATE:

PH CALIBRATION CHECK

pH 7 **(LOT #):** **(EXP. DATE):** POST-CAL. READING / STANDARD	pH 4 / 10 **(LOT #):** **(EXP. DATE):** POST-CAL. READING / STANDARD	CAL. RANGE	TIME
/	/	☐ WITHIN RANGE	
/	/	☐ WITHIN RANGE	
/	/	☐ WITHIN RANGE	
/	/	☐ WITHIN RANGE	

SPECIFIC CONDUCTIVITY CALIBRATION CHECK

CAL. READING **(LOT #):** **(EXP. DATE):** POST-CAL. READING / STANDARD	TEMPERATURE (°CELSIUS)	CAL. RANGE	TIME
/		☐ WITHIN RANGE	
/		☐ WITHIN RANGE	
/		☐ WITHIN RANGE	
/		☐ WITHIN RANGE	

ORP CALIBRATION CHECK

CAL. READING **(LOT #):** **(EXP. DATE):** POST-CAL. READING / STANDARD	TEMPERATURE (°CELSIUS)	CAL. RANGE	TIME
/		☐ WITHIN RANGE	
/		☐ WITHIN RANGE	
/		☐ WITHIN RANGE	
/		☐ WITHIN RANGE	

D.O. CALIBRATION CHECK

CALIBRATION READING (mg/L)	CAL. RANGE	TIME
	☐ WITHIN RANGE	
	☐ WITHIN RANGE	
	☐ WITHIN RANGE	
	☐ WITHIN RANGE	

TURBIDITY CALIBRATION CHECK

CALIBRATION READING (NTU) **(LOT #):** **(EXP. DATE):** POST-CAL. READING / STANDARD	CALIBRATION READING (NTU) **(LOT #):** **(EXP. DATE):** POST-CAL. READING / STANDARD	CAL. RANGE	TIME
/	/	☐ WITHIN RANGE	
/	/	☐ WITHIN RANGE	
/	/	☐ WITHIN RANGE	
/	/	☐ WITHIN RANGE	

NOTES

COMMENTS

☐ **AUTOCAL SOLUTION** **(LOT #):** **(EXP. DATE):**	☐ **STANDARD SOLUTION (S)** LIST LOT NUMBERS AND EXPIRATION DATES UNDER CALIBRATION CHECK
CALIBRATED PARAMETERS	**CALIBRATION RANGES [1]**
☐ pH	pH: +/- 0.2 S.U.
☐ COND	COND: +/- 1% OF CAL. STANDARD
☐ ORP	ORP: +/- 25 mV
☐ D.O.	D.O.: VARIES
☐ TURB	TURB: +/- 5% OF CAL. STANDARD
☐ _____	[1] CALIBRATION RANGES ARE SPECIFIC TO THE MODEL OF THE WATER QUALITY METER
☐ _____	

PROBLEMS ENCOUNTERED	CORRECTIVE ACTIONS

SIGNED DATE

CHECKED BY DATE

RMT

PID FIELD CALIBRATION LOG

PROJECT NAME:	TPC -	MODEL:	
PROJECT NUMBER.:	00-08070.	LAMP VOLTAGE:	
SAMPLER NAME:		SERIAL NO.:	

PID CALIBRATION CHECK

	DATE: TIME: INITIALS:	DATE: TIME: INITIALS:	DATE: TIME: INITIALS:	DATE: TIME: INITIALS:	DATE: TIME: INITIALS:
BATTERY CHECK	☐	☐	☐	☐	☐
ZERO GAS	/	/	/	/	/
SPAN GAS	/	/	/	/	/
AUDIBLE FAN MOTOR CHECK	☐	☐	☐	☐	☐
RESPONSE CHECK	☐	☐	☐	☐	☐

NOTES

PROBLEMS ENCOUNTERED	CORRECTIVE ACTION

SIGNED DATE CHECKED DATE

REVISED 03/2008

RMT

WATER LEVEL DATA

PROJECT NAME: TPC -	DATE:
PROJECT NUMBER: 00-08070.	AUTHOR:

WELL LOCATION	TIME	REFERENCE	DEPTH TO WATER (FEET)	DEPTH TO BOTTOM (FEET)	DEPTH TO PRODUCT (FEET)	WATER ELEVATION

ALL WATER LEVELS MUST INCLUDE REFERENCE POINT AND TAPE CORRECTION FACTOR (E.G., 1.1 + 0.00 T/PVC).

SIGNED DATE

CHECKED DATE

RMT

WATER SAMPLE LOG

PROJECT NAME: TPC -	PREPARED		CHECKED	
PROJECT NUMBER: 00-08070.	BY	DATE:	BY:	DATE:

SAMPLE ID:	WELL DIAMETER: ☐ 2" ☐ 4" ☐ 6" ☐ OTHER _____
WELL MATERIAL: ☐ PVC ☐ SS ☐ IRON ☐ GALVANIZED STEEL ☐ OTHER _____	
SAMPLE TYPE: ☐ GW ☐ WW ☐ SW ☐ DI ☐ LEACHATE ☐ OTHER _____	

PURGING	TIME:	DATE:	SAMPLE	TIME:	DATE:
PURGE METHOD: ☐ PUMP _____ ☐ BAILER _____			PH: _____ SU	CONDUCTIVITY: _____ umhos/cm	
			ORP: _____ mV	DO: _____ mg/L	
DEPTH TO WATER: _____ T/ PVC			TURBIDITY: _____ NTU ☐ NONE ☐ SLIGHT ☐ MODERATE ☐ VERY		
DEPTH TO BOTTOM: _____ T/ PVC					
WELL VOLUME: _____ ☐ LITERS ☐ GALLONS			TEMPERATURE: _____ °C	OTHER: _____	
VOLUME REMOVED: _____ ☐ LITERS ☐ GALLONS			COLOR: _____	ODOR: _____	
COLOR: _____ ODOR: _____			FILTRATE (0.45 um) ☐ YES ☐ NO		
TURBIDITY ☐ NONE ☐ SLIGHT ☐ MODERATE ☐ VERY			FILTRATE COLOR: _____	FILTRATE ODOR: _____	
			QC SAMPLE: ☐ MS/MSD ☐ DUP- _____		
DISPOSAL METHOD: ☐ GROUND ☐ DRUM ☐ OTHER			COMMENTS:		

TIME	PURGE RATE (ML/MIN)	PH (SU)	CONDUCTIVITY (umhos/cm)	ORP (mV)	D.O. (mg/L)	TURBIDITY (NTU)	TEMPERATURE (°C)	WATER LEVEL (FEET)	CUMULATIVE PURGE VOLUME (GAL OR L)
									INITIAL

NOTE: STABILIZATION TEST IS COMPLETE WHEN 3 SUCCESSIVE READINGS ARE WITHIN THE FOLLOWING LIMITS:

pH: +/- **10 %** COND.: +/- **10 %** ORP: +/- **10 %** D.O.: +/- **10 %** TURB: +/- **10 %** or </= **5** TEMP.: +/- **0.5°C**

BOTTLES FILLED	PRESERVATIVE CODES A - NONE B - HNO3 C - H2SO4 D - NaOH E - HCL F - _____								
NUMBER	SIZE	TYPE	PRESERVATIVE	FILTERED		NUMBER	SIZE	TYPE	PRESERVATIVE	FILTERED	
				☐ Y	☐ N					☐ Y	☐ N
				☐ Y	☐ N					☐ Y	☐ N
				☐ Y	☐ N					☐ Y	☐ N
				☐ Y	☐ N					☐ Y	☐ N
				☐ Y	☐ N					☐ Y	☐ N

SHIPPING METHOD: _____	DATE SHIPPED: _____	AIRBILL NUMBER: _____
COC NUMBER: _____	SIGNATURE: _____	DATE SIGNED: _____

RMT

WATER SAMPLE LOG (CONTINUED FROM PREVIOUS PAGE)

PROJECT NAME: TPC -	PREPARED		CHECKED	
PROJECT NUMBER: 00-08070.	BY:	DATE:	BY:	DATE:

SAMPLE ID:

TIME	PURGE RATE (ML/MIN)	PH (SU)	CONDUCTIVITY (umhos/cm)	ORP (mV)	D.O. (mg/L)	TURBIDITY (NTU)	TEMPERATURE (°C)	WATER LEVEL (FEET)	CUMULATIVE PURGE VOLUME (GAL OR L)

SIGNATURE: _____ DATE SIGNED: _____

RMT SEDIMENT / SOIL GRAB SAMPLE LOG

PROJECT NAME: TPC -	PREPARED		CHECKED	
PROJECT NUMBER: 00-08070.	BY:	DATE:	BY:	DATE:

SAMPLE ID:	COLLECTED BY:
DATE COLLECTED:	SAMPLE TYPE: ☐ SEDIMENT ☐ SOIL ☐ OTHER _____
TIME COLLECTED:	QC SAMPLE: ☐ MS/MSD ☐ DUP- _____

SAMPLE LOCATION	SAMPLE COORDINATES
	NORTHING / LATITUDE:
	EASTING / LONGITUDE:

SAMPLE CONTAINERS:

SAMPLE EQUIPMENT:

SAMPLE SCREENING EQUIPMENT: ☐ PID ☐ GAMMA DETECTOR ☐ FID ☐ OTHER _____ NOTES: _____

SAMPLE SCREENING RESULTS: _____ ☐ PPM ☐ OTHER _____

SAMPLE DESCRIPTION:

ADDITIONAL NOTES:

SHIPPING METHOD: _____	DATE SHIPPED: _____	AIRBILL NUMBER: _____
COC NUMBER: _____	SIGNATURE: _____	DATE SIGNED: _____

RMT

AIR / VAPOR SAMPLE LOG

PROJECT NAME: TPC -	PREPARED	CHECKED
PROJECT NUMBER: 00-08070.	BY: DATE:	BY: DATE:

SAMPLE INFORMATION		
SAMPLE TYPE: ☐ COMPOSITE ☐ GRAB	**SAMPLE ID:**	
SAMPLE MEDIA ☐ INDOOR AIR ☐ SOIL VAPOR ☐ SYSTEM PERFORMANCE ☐ OTHER	LOCATION:	LOCATION COORDINATES: N: E:
SAMPLE DURATION:	SAMPLE HEIGHT / (DEPTH):	
SAMPLE CONTAINER TYPE: ☐ SUMMA CANISTER ☐ TEDLAR BAG ☐ OTHER:		
FLOW VALVE ID / SERIAL NUMBER:	CANISTER SERIAL NUMBER:	

READING	TIME	VACUUM (INCHES - Hg / PSIG)	DATE	INITIALS	COMMENTS
INITIAL VACUUM CHECK					
INITIAL FIELD VACUUM					
FINAL FIELD VACUUM					

SAMPLE START TIME:	SAMPLE STOP TIME:

NOTES AND OBSERVATIONS
MOTORIZED VEHICLE STORAGE :
MOTORIZED VEHICLE TRAFFIC:
OPERATIONS (e.g., painting, oil recovery):
CLEANERS / SOLVENTS IN USE:
MATERIAL STORAGE (e.g., paint, gasoline):
NOTICEABLE ODORS:
AUDIBLE OR NEARBY HVAC OPERATION:
OTHER:

ADDITIONAL COMMENTS:

SHIPPING METHOD: _____	DATE SHIPPED: _____	AIRBILL NUMBER: _____
COC NUMBER: _____	SIGNATURE: _____	DATE SIGNED: _____

RMT

PASSIVE SOIL GAS SAMPLE LOG

		PREPARED		CHECKED	
PROJECT NAME:	TPC -				
PROJECT NUMBER:	00-08070.	BY:	DATE:	BY:	DATE:

Sample ID	Grid Location	Boring Depth (inches)	PID Reading (ppm)	Installation		Removal		Additional Notes
				Date	Time	Date	Time	

RMT

LOG OF SOIL BORING

PROJECT NAME: TPC -		**SOIL BORING ID:**	
PROJECT NUMBER: 00-08070.		LOCATION:	SHEET 1 OF
LOGGED BY:			SURFACE ELEV.:
PROJECT LOCATION: Tecumseh, Michigan		N: E:	DATE STARTED:
DRILLED BY:	DRILLER NAME:		DATE COMPLETED:

NO.	TYPE	%	BLOWS	PID	DEPTH	VISUAL CLASSIFICATION AND OBSERVATIONS	COMMENT

DRILLING METHOD
DRILL RIG
BORING DIAMETER

WATER LEVEL OBSERVATIONS			
FIRST OCCURRENCE:			
DATE	TIME	DEPTH TO WATER	DEPTH TOBOTTOM

SIGNED DATE

CHECKED DATE

REVISED 03/2008

RMT

LOG OF SOIL BORING

SHEET 2 OF

PROJECT NAME: TPC -

SOIL BORING ID:

NO.	TYPE	%	BLOWS	PID	DEPTH	VISUAL CLASSIFICATION AND OBSERVATIONS	COMMENT

SIGNED DATE

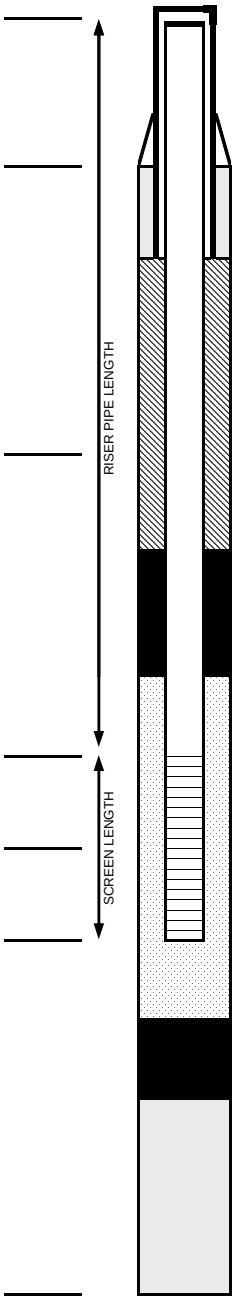
CHECKED DATE

RMT

WELL CONSTRUCTION DIAGRAM

PROJ. NAME: TPC -		WELL ID:	
PROJ. NO: 00-08070.	DATE INSTALLED:	INSTALLED BY:	CHECKED BY:

ELEVATION (BENCHMARK: USGS)	DEPTH BELOW OR ABOVE GROUND SURFACE (FEET)



_____ TOP OF CASING

0.0 GROUND SURFACE

_____ CEMENT SURFACE PLUG

GROUT/BACKFILL MATERIAL

GROUT/BACKFILL METHOD

_____ GROUT

BENTONITE SEAL MATERIAL

_____ BENTONITE SEAL

_____ TOP OF SCREEN

FILTER PACK MATERIAL

_____ BOTTOM OF SCREEN

_____ BOTTOM OF FILTER PACK

_____ BENTONITE PLUG

BACKFILL MATERIAL

_____ HOLE BOTTOM

NOTES:

CASING AND SCREEN DETAILS

TYPE OF RISER: _____

PIPE SCHEDULE: _____

PIPE JOINTS: _____

SOLVENT USED? _____

SCREEN TYPE: _____

SCR. SLOT SIZE: _____

BOREHOLE DIAMETER: _____ IN. FROM _____ TO _____ FT.
_____ IN. FROM _____ TO _____ FT.

SURF. CASING DIAMETER: _____ IN. FROM _____ TO _____ FT.
_____ IN. FROM _____ TO _____ FT.

WELL DEVELOPMENT

DEVELOPMENT METHOD: _____

TIME DEVELOPING: _____ HOURS

WATER REMOVED: _____ GALLONS

WATER ADDED: _____ GALLONS

WATER CLARITY BEFORE / AFTER DEVELOPMENT

CLARITY BEFORE: _____

COLOR BEFORE: _____

CLARITY AFTER: _____

COLOR AFTER: _____

ODOR (IF PRESENT): _____

WATER LEVEL SUMMARY

MEASUREMENT (FEET)			DATE	TIME
DTB BEFORE DEVELOPING:		T/PVC		
DTB AFTER DEVELOPING:		T/PVC		
SWE BEFORE DEVELOPING:		T/PVC		
SWE AFTER DEVELOPING:		T/PVC		
OTHER SWE:		T/PVC		
OTHER SWE:		T/PVC		

PROTECTIVE CASING DETAILS

PERMANENT, LEGIBLE WELL LABEL ADDED? ☐ YES ☐ NO

PROTECTIVE COVER AND LOCK INSTALLED? ☐ YES ☐ NO

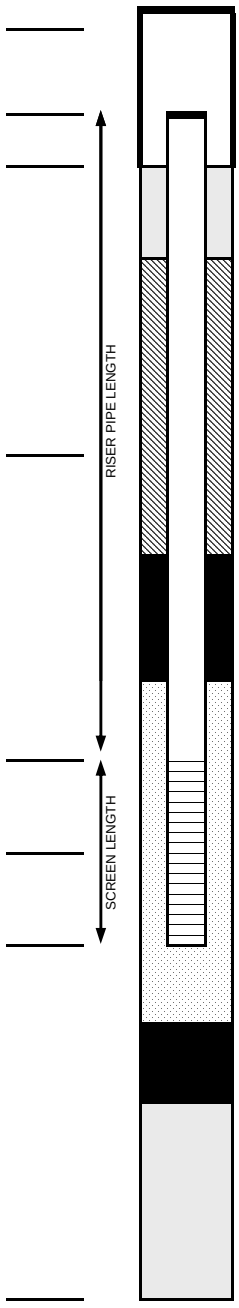
LOCK KEY NUMBER: _____

RMT

WELL CONSTRUCTION DIAGRAM

PROJ. NAME: TPC -		WELL ID:	
PROJ. NO: 00-08070.	DATE INSTALLED:	INSTALLED BY:	CHECKED BY:

ELEVATION (BENCHMARK: USGS)	DEPTH BELOW OR ABOVE GROUND SURFACE (FEET)



0.0 GROUND SURFACE

TOP OF CASING

CEMENT SURFACE PLUG

GROUT/BACKFILL MATERIAL

GROUT/BACKFILL METHOD

GROUT

BENTONITE SEAL MATERIAL

BENTONITE SEAL

TOP OF SCREEN

FILTER PACK MATERIAL

BOTTOM OF SCREEN

BOTTOM OF FILTER PACK

BENTONITE PLUG

BACKFILL MATERIAL

HOLE BOTTOM

NOTES:

CASING AND SCREEN DETAILS

TYPE OF RISER: _____

PIPE SCHEDULE: _____

PIPE JOINTS: _____

SOLVENT USED? _____

SCREEN TYPE: _____

SCR. SLOT SIZE: _____

BOREHOLE DIAMETER: _____ IN. FROM _____ TO _____ FT.
_____ IN. FROM _____ TO _____ FT.

SURF. CASING DIAMETER: _____ IN. FROM _____ TO _____ FT.
_____ IN. FROM _____ TO _____ FT.

WELL DEVELOPMENT

DEVELOPMENT METHOD: _____

TIME DEVELOPING: _____ HOURS

WATER REMOVED: _____ GALLONS

WATER ADDED: _____ GALLONS

WATER CLARITY BEFORE / AFTER DEVELOPMENT

CLARITY BEFORE: _____

COLOR BEFORE: _____

CLARITY AFTER: _____

COLOR AFTER: _____

ODOR (IF PRESENT): _____

WATER LEVEL SUMMARY

MEASUREMENT (FEET)			DATE	TIME
DTB BEFORE DEVELOPING:		T/PVC		
DTB AFTER DEVELOPING:		T/PVC		
SWE BEFORE DEVELOPING:		T/PVC		
SWE AFTER DEVELOPING:		T/PVC		
OTHER SWE:		T/PVC		
OTHER SWE:		T/PVC		

PROTECTIVE CASING DETAILS

PERMANENT, LEGIBLE WELL LABEL ADDED? ☐ YES ☐ NO

PROTECTIVE COVER AND LOCK INSTALLED? ☐ YES ☐ NO

LOCK KEY NUMBER: _____

Appendix H
Pace Instructions of Time Weighted Sampling with Air Canisters

Pace Analytical®

AIR CANISTERS

Instructions for Canister Time Weighted Sampling

(Tools needed: two open ended 9/16" wrenchs)

1. INSPECTION – Inspect your canister shipment upon arrival. Compare the contents with the packing slip and notify Pace Analytical of any discrepancy or damage. Familiarize yourself with the contents you received by comparing them to the pictures on the right. Do not open the valve until you are ready to sample. Even a small loss of vacuum will compromise your sample.

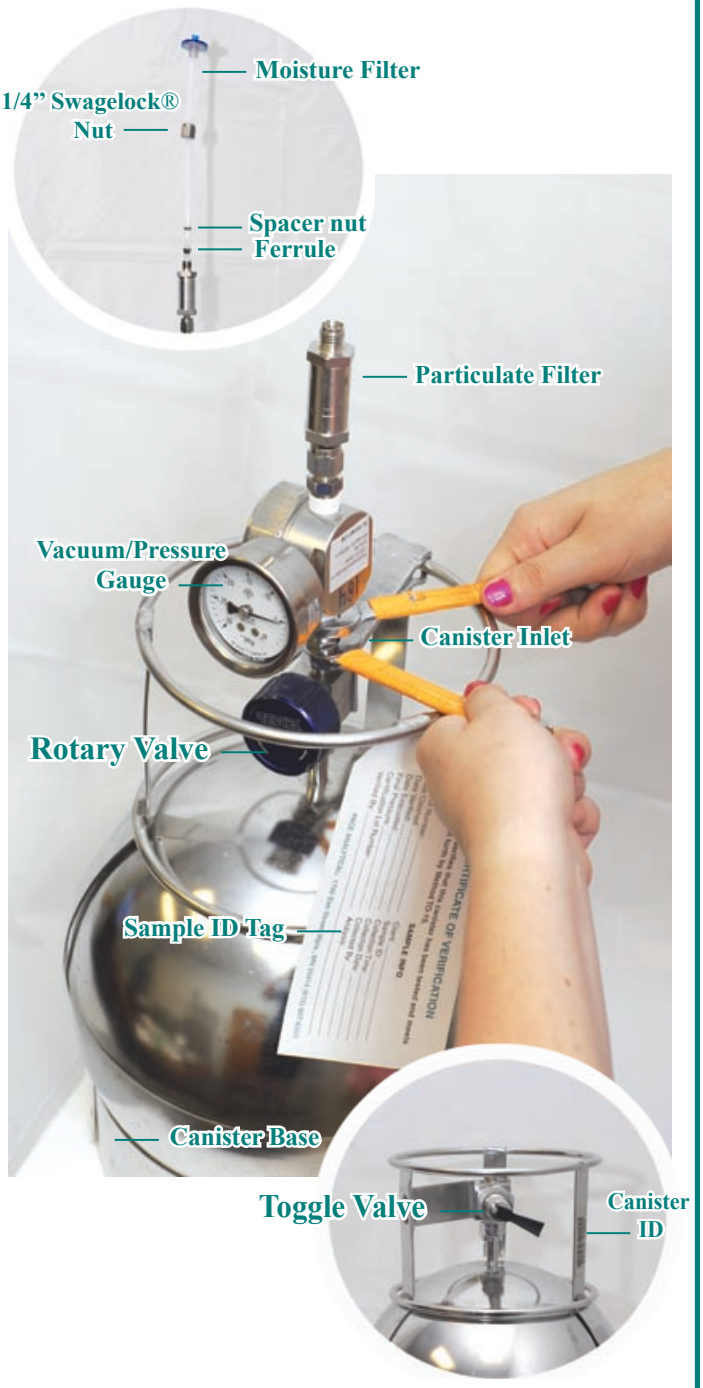
2. CONNECTION – Remove the brass caps with a 9/16" wrench. Connect the flow controller to the valve where the cap was. The connection is made by hand tightening the Swagelock® connection to the canister. There is only one way to connect the flow controller, so you will not be able to put it on backwards or incorrectly. The particulate filter points away from the can. Once connected, use two open ended 9/16 inch wrenches to further tighten this connection. It has been found that using two open ended wrenches makes the most reliable connection. Make sure that the connection is firmly tightened. The final connection must be leak tight recognizing also that over-tightening can cause leaks as well. Do not use pliers or adjustable-end wrenches to tighten this Swagelock® connection. Use only open ended wrenches for tightening. If you did not request extra media (tubing, fittings and moisture filters etc...) the canister is now ready for sampling. If you are connecting to a predetermined sampling point you may have received the following: 6 inches of ¼ inch OD Teflon tubing, ¼ inch Swagelock® nut, ferrule, spacer nut and moisture filter (if requested). Connect these items in series using the pictures on the right as a guide. The spacer fits between the nut and the f errule. The ferrule must be pointed down toward the flow controller connection.

3. SAMPLING – To begin sampling simply open the canister valve (you may have either a rotary valve or a toggle valve). One full turn counter clockwise for the rotary valve is sufficient. The toggle valve will open by flipping upward. Record the initial vacuum gauge reading. Depending on atmospheric conditions; it should be between -26 and -30 inches of Hg. Watch the vacuum decline. The vacuum rate decline is directly proportional to collection set time.

4. COMPLETION – After sampling is complete, close the canister valve and record the ending vacuum gauge reading. Disassemble the components and return them in the original shipping package they were received in. Verify the contents for return to the laboratory. Complete the Chain-of-Custody form and return with the samples to the laboratory. Please reference the canister ID on the Chain-of-Custody.

Time Weighted Sample Configuration

Sampling Point Connection



www.pacelabs.com

Questions? Please call the Air Technical phone number 612-607-6386

Appendix I
Laboratory Chain-of-Custody Forms

TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court SE Grand Rapids, MI 49512
Phone (616) 975-4500 Fax (616) 942-7463
www.trimatrixlabs.com

Chain of Custody Record

COC No.

For Lab Use Only

Cart

VOA Rack/Tray

Receipt Log No.

Project Chemist

Work Order No.

Client Name

Address

Phone

Fax

Project Name

Client Project No. / P.O. No.

Invoice To
(●) Client ( ) Other (comments)

Contact/Report To

Analyses Requested

⇦ PRESERVATIVES

A NONE pH~7
B HNO_3 pH<2
C H_2SO_4 pH<2
D 1+1 HCl pH<2
E NaOH pH>12
F ZnAc/NaOH pH>9
G MeOH
H Other (note below)

Container Type (corresponds to Container Packing List)

Number of Containers Submitted

Schedule	Matrix Code	Laboratory Sample Number		Sample ID	Cooler ID	Sample Date	Sample Time	Comp / Grab	Matrix	Total	Sample Comments
			1								
			2								
			3								
			4								
			5								
			6								
			7								
			8								
			9								
			10								

Sampled By (print)

Sampler's Signature

Company

How Shipped?

Tracking No.

Comments

1. Relinquished By Date Time

1. Received By Date Time

2. Relinquished By Date Time

2. Received By Date Time

3. Relinquished By Date Time

3. Received For Lab By Date Time

AIR: CHAIN-OF-CUSTODY / Analytical Request Document

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

Page: of

Section A
Required Client Information:

Company:	
Address:	
Email To:	
Phone:	Fax:
Requested Due Date/TAT:	

Section B
Required Project Information:

Report To:
Copy To:
Purchase Order No.:
Project Name:
Project Number:

Section C
Invoice Information:

Attention:
Company Name:
Address:
Pace Quote Reference:
Pace Project Manager/Sales Rep.
Pace Profile #:

Program

☐ UST ☐ Superfund ☐ Emissions ☐ Clean Air Act
☐ Voluntary Clean Up ☐ Dry Clean ☐ RCRA ☐ Other____

Location of Sampling by State _____

Reporting Units
ug/m³___ mg/m³ ____
PPBV ___ PPMV ____
Other ___

Report Level II.___ III.___ IV.___ Other_____

'Section D Required Client Information

AIR SAMPLE ID
Sample IDs MUST BE UNIQUE

Valid Media Codes

MEDIA	CODE
Tedlar Bag	TB
1 Liter Summa Can	1LC
6 Liter Summa Can	6LC
Low Volume Puff	LVP
High Volume Puff	HVP
Other	PM10

ITEM #	MEDIA CODE	PID Reading (Client only)	COLLECTED COMPOSITE START END/GRAB DATE	COLLECTED COMPOSITE START END/GRAB TIME	COLLECTED COMPOSITE - DATE	COLLECTED COMPOSITE - TIME	Canister Pressure (Initial Field - psig)	Canister Pressure (Final Field - psig)	Summa Can Number	Flow Control Number	Method: PM10	3C- Fixed Gas (%)	TO-3	TO-3M (Methane)	TO-4 (PCBs)	TO-13 (PAH)	TO-14	TO-15	TO15 Short List*	Pace Lab ID
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				
9																				
10																				
11																				
12																				

Comments :

RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME

SAMPLE CONDITIONS

	Y/N	Y/N	Y/N	Y/N
	Y/N	Y/N	Y/N	Y/N
	Y/N	Y/N	Y/N	Y/N
	Y/N	Y/N	Y/N	Y/N
Temp in °C	Received on Ice	Custody Sealed Cooler	Samples Intact	

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER:

SIGNATURE of SAMPLER: DATE Signed (MM / DD / YY)

CHAIN-OF-CUSTODY
PASSIVE SOIL-GAS SAMPLES

Project Information	
Beacon Project No.:	
Site Name:	
Site Location:	
Analytical Method:	EPA Method 8260B
Target Compounds:	

BEACON ENVIRONMENTAL SERVICES, INC.
323 Williams Street, Suite D, Bel Air, MD 21014 (800) 878-5510

Client Information	
Company Name:	
Office Location:	
Samples Submitted By:	
Contact Phone No.:	

Field Sample ID	Lab Sample ID (for lab use only)	Comments (only necessary if problem or discrepancy)			
		Condition of sample or vial	Date	Time	Initial

Shipment of Field Kit to Site — Custody Seal # **Intact? Y N**

Relinquished by:	Date/Time	Courier	Received by:	Date/Time

Shipment of Field Kit to Laboratory — Custody Seal # **Intact? Y N**

Relinquished by:	Date/Time	Courier	Received by:	Date/Time

Environmental Chemistry Consulting Services, Inc.
2525 Advance Road
Madison, WI 53718
608-221-8700 (phone)
608-221-4889 (fax)

CHAIN OF CUSTODY

Lab Work Order #:

Mail Report To:
Company:
Address:
E-mail Address:
Invoice To:
Company:
Address:

Project Number:
Project Name:
Project Location:
Turn Around (circle one): Normal Rush
If Rush, Report Due Date:
Sampled By (Print):

Analyses Requested
Preservation Codes

Sample Description	Collection Date	Collection Time	Matrix	Total # of Containers							Comments	Lab ID	Lab Receipt Time

Preservation Codes
A=None B=HCL C=H_2SO_4
D=HNO_3 E=EnCore F=Methanol
G=NaOH O=Other (Indicate)

Matrix Codes
A=Air S=Soil W=Water O=Other

Relinquished By:		Date:	Time:	Received By:	Date:	Time:
Relinquished By:		Date:	Time:	Received By:	Date:	Time:
Custody Seal: Present/Absent	Intact/Not Intact	Seal #'s		Receipt Temp:		
Shipped Via:				Temp Blank Y N		