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DRAFT
QUALITY ASSURANCE PROJECT PLAN
EPA GRANT NO. 00A00826
FORMER DANIEL'S MILL
98 East Main Street
Vernon, Connecticut

August 2023
File No. 05.0045441.12

PREPARED FOR:
Town of Vernon
Vernon, Connecticut

GZA GeoEnvironmental, Inc.
95 Glastonbury Boulevard | Glastonbury, CT 06033
860-286-8900

31 Offices Nationwide
www.gza.com

INTRODUCTION

GZA GeoEnvironmental, Inc. (GZA), on behalf of the Town of Vernon, has prepared this *Quality Assurance Project Plan* (QAPP) for the Former Daniel's Mill property located in Vernon, Connecticut (herein referred to as the Site). As described further herein, the primary objective of this QAPP is to establish data collection and quality assurance/quality control (QA/QC) procedures associated with the planned Site remedial and abatement work. The QAPP was prepared generally consistent with the following two Environmental Protection Agency (EPA) guidance documents: (1) *EPA New England Quality Assurance Project Plan Program Guidance* dated January 9, 2010 (EPAQAPP-2005PG2) and (2) *EPA Requirements for Quality Assurance Project Plans* dated March 2001 (EPA QA/R-5).

The four primary elements of this QAPP and their intent are as follows:

1. **Group A – Project Management:** These elements cover the basic area of project management, including the project history, project objectives, and roles and responsibilities of the participants.

2. **Group B –Data Acquisition:** These elements outline the data collection phase of the project and includes a summary of sampling methodologies, laboratory analyses, field screening activities, the QC measures employed, and data collection documentation procedures.

3. **Group C – Data Assessment:** These elements address the activities for assessing the effectiveness of the implementation of the project and associated QA and QC activities. The purpose of assessment activities is to ensure that the QAPP is implemented as prescribed.

4. **Group D – Data Usability:** These elements cover the QA activities that occur after the data collection phase of the project is completed. Implementation of these elements ensures that the data conform to the specified criteria, thus achieving the project objectives.

GROUP A PROJECT MANAGEMENT

A1 TITLE AND APPROVAL SHEET

GZA Project and Quality Assurance Manager: Anthony Trani

August__, 2023

Signature ***Date***

GZA Principal-In-Charge: David Rusczyk

August __, 2023

Signature ***Date***

EPA Project Officer: Lorraine Byrne

August__, 2023

Signature ***Date***

EPA Quality Regional Manager: Anthony Pepe

August__, 2023

Signature ***Date***

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APPENDICES

A3 DISTRIBUTION LIST

ANTHONY TRANI	GZA PROJECT AND QUALITY ASSURANCE MANAGER
ETHAN LEIGHTON	ALPHA ANALYTICAL (LABORATORY SERVICES)
BOBBI ALOISA	PHOENIX ENVIRONMENTAL LABORATORY (LABORATORY SERVICES)
SHAUN GATELY	TOWN OF VERNON
BRIAN DRAKE	EPA REGION 1: RCRA CORRECTIVE ACTION & TSCA SECTION
LORRAINE BYRNE	EPA REGION 1: PROJECT OFFICER
ANTHONY PEPE	EPA REGION 1: QUALITY ASSURANCE REGIONAL MANAGER
AMBER TRAHAN	CT DEEP: BUREAU OF MATERALS MANAGEMENT AND COMPLIANCE ASSURANCE
CLAIRE QUINN	CT DEEP: BUREAU OF WATER PROTECTION AND LAND REUSE
MEENA MORTAZAVI	CT DEEP: BROWNSFIELD COORDINATOR

A4 Project Organization and Responsibility

The Project Coordinator and Principal-in-Charge for this project is David Rusczyk of GZA. The Project and Quality Assurance (QA) Manager is Anthony Trani of GZA. Mr. Trani will be responsible for the overall management of the project, ensuring that established protocols and procedures are utilized, confirming that data quality documentation is appropriate, and that the QA goals have been met.

The organizational chart included in **Appendix A** illustrates the individuals involved in the project and their affiliations. Contact information for EPA, CT DEEP, the Town of Vernon representative, the GZA Principal-in-Charge, the GZA Project and QA Manager, and the GZA H&S Coordinator is also included in **Appendix A**.

A5 PROBLEM DEFINITION/BACKGROUND

Site Location and Description

The Site is located at 98 East Main Street in an industrial zone of Vernon, Connecticut and consists of an approximate 1-acre parcel of land. The Site is abutted by East Main Street to the north, the former Amerbelle Textile Mill to the east, American Mill Pond to the south and west, and by a former industrial

facility (Anocoil) to the southwest. A Site Locus Plan is provided as **Figure 1** and a Site plan is included as **Figure 2**.

The Site is the location of the former Daniel's Mill, which was built in approximately 1855. The Site is improved with a six-story (including basement and attic) historical mill building with a footprint measuring approximately 9,000 square-feet. Areas to the west of the Site building are currently predominantly asphalt paved and a narrow-grassed area is located to the east of the building. The Hockanum River runs from east to west through the abutting former Amerbelle Textile Mill property in a stone lined raceway and discharges to the American Mill Pond located adjacent to the south and west of the Site. Historically, a portion of the river was diverted through the Daniel's Mill building via a raceway pipe to provide power to the former mill facility.

The Site is serviced by municipal water and sanitary sewer, natural gas, and electric services; however, the Site utilities have been shut-off since the building has been vacant since 2014.

Site History and Problem Definition

The Site was reportedly developed as a textile mill which manufactured cotton, stockinet, and woolen products between 1855 and 1951. After the Site ceased textile operations, it was used to produce fire retardant paints, mastics, and insecticides. Other past tenants/Site uses include a salvage company, outboard motor center, furnace brokers, music studio rentals, an electrical contractor, a sheet metal workshop, and storage units.

In 2015, the Town received a grant from the Connecticut Department of Economic and Community Development (DECD) to assess environmental conditions and facilitate future redevelopment and reuse of the Site. GZA, on behalf of the Town, completed several rounds of investigations to assess environmental conditions and the presence of hazardous building materials. Based on the results and observations made during these investigations, remedial activities will be necessary within certain Areas of Concern (AOCs) to achieve compliance with the Connecticut Remediation Standard Regulations (RSRs). These AOCS include the following:

- AOC-3: Loading Dock Area;
- AOC-6 (Exterior): Historical Use of Site Building - East of the Site Building; and,
- AOC-6 (Interior): Historical Use of Basement.

In addition, polychlorinated biphenyls (PCBs) were detected within interior building materials at concentrations up to 254 mg/kg, in soils beneath the basement concrete floor in localized areas at concentrations greater than 50 mg/kg, and in soils in certain exterior areas adjacent to the building at concentrations up to 26 mg/kg. These PCB impacted materials are regulated under the *Toxic Substances Control Act* (TSCA; 40 CFR 761). Weston & Sampson Engineers, Inc. (W&S), on behalf of the Town of Vernon, submitted a remediation plan to address the PCB-impacted building materials to the EPA and the Connecticut Department of Energy and Environmental Protection (CTDEEP) in December 2021 and, based on subsequent discussions with EPA and CTDEEP, a risk assessment will be performed to support

a Risk-Based Cleanup for the building materials under 40 CFR 761.61(c). The remedial activities outlined in the building materials RAP included:

- Removal and disposal of all temporary walls as PCB bulk product waste.
- Demolition of the painted wooden loading dock on the west side of the building and disposal of the debris as PCB bulk product waste.
- Removal of the top-layer on all floors and the second layer (3rd floor and above) of wood flooring and disposal as a PCB bulk product waste. After removal of the upper layers of flooring, a vapor barrier will be applied to the surface of the remaining wood floor and a continuous, 3-inch-thick layer of concrete will be installed to prevent direct exposure to any PCBs remaining following abatement.
- Removal of paint and plaster from walls, ceilings, and other structural members on all levels of the building through media blasting until there are no visual remnants of paint and disposal of the paint and blasting wastes as PCB bulk product waste. After removal, three coats[1] of varying colors of an epoxy-containing material will be applied to the ceilings, walls, and wood structural members. For aesthetic reasons, barriers may also be constructed over some walls, ceilings, or columns within the structure.
- Removal of doors and windows along with any caulk and disposal as PCB bulk product waste.
- Removal of the elevator cab and elevator doors on each floor and disposal as PCB bulk product waste.

In addition, in January 2023, GZA submitted a *Notification of Self-Implementing Cleanup* (the "Notification") to EPA and CTDEEP to remove the PCB impacted concrete flooring in the basement, soils beneath the basement floor slab, and soils in certain exterior areas to meet the levels for unrestricted, high occupancy use (less than 1 mg/kg) under CFR 761.61(a)(4)(i)(A).

Following a 30-day public notice and comment period, GZA also submitted a May 2023 Remedial Action Plan (RAP) to the CTDEEP describing the remedial activities that will be implemented to address the identified exterior and sub-slab soil impacts at the Site consistent with the RSRs.

The Site is an integral part of a larger proposed mixed residential and commercial development that includes the former Amerbelle Textile Mill (which is owned by the Town of Vernon) located to the east of the Site and the former Anocoil Mill located to the south/southwest of the Site. To facilitate this development, the Town of Vernon applied for Brownfield Municipal Liability Relief Protection (BMLRP) in September 2020 and acquired the Site in 2021. The Town also received DECD and EPA grant funding to remediate the impacted soils and perform the hazardous building material abatement work at the Site.

A6 PROJECT/TASK DESCRIPTION

In order to facilitate redevelopment of the former mill property and to demonstrate compliance with the RSRs and the federal PCB regulations (40 CFR part 761) after completion of the remedial and

[1] Note. The December 2021 PCB Building Materials RAP proposed the application of two coats of epoxy. An additional coat will be conservatively applied to encapsulate residual PCB impacted materials.

abatement activities outlined in the December 2021 PCB Building Materials RAP and the May 2023 RAP, the following sampling programs will be implemented:

- Underground Storage Tank (UST) Closure Sampling – A UST was identified east of the building (AOC-6: Exterior). The liquids within this UST and any interconnected piping will be removed and the interior of the UST cleaned. After liquid removal and UST cleaning, the UST will be removed, and any impacted soil (if any) excavated and disposed off-Site. Verification soil samples will be collected from the tank grave to demonstrate compliance with the Residential Direct Exposure Criteria (R-DEC) and the GB Pollutant Mobility Criteria (GB-PMC) within the RSRs.

- Verification Concrete Sampling – PCB impacted concrete within the basement of the building (AOC-6: Interior) and within the limited areas of the overlying floors will be removed and/or scarified and disposed off-Site to a chemical waste or Subtitle C landfill until residual concentrations within the remaining concrete are less than the unrestricted, high occupancy use threshold of 1 mg/kg under CFR 761.61(a)(4)(i)(A).

- Verification Soil Sampling – Soils containing PCB levels greater than 1 mg/kg (AOC-3, AOC-6: Interior, and AOC-6: Exterior) and/or other constituents above the R-DEC (AOC-6: Exterior) will be excavated and disposed off-Site. Upon achieving the anticipated excavation limits, verification soil samples will be collected from the base and sidewalls of the excavations.

- Baseline Indoor Air Sampling – Ambient air samples will be collected from within the vacant mill building prior to any abatement and renovation activities to evaluate static/equilibrated PCB concentrations and potential worst case exposure risks for future residents/occupants of the building.

- Reoccupancy Indoor Air Sampling – Following the completion of the PCB impacted building material abatement activities and the building renovation work, reoccupancy air samples will be collected from each residential unit and from one common or maintenance area on each floor to evaluate the effectiveness of the abatement activities.

- Long Term Indoor Air Sampling – Ambient air samples will be collected and analyzed for PCBs to evaluate the effectiveness of the abatement activities. The indoor air samples will be collected on the following frequency following completion of the building renovation activities:
  - Annually for the first 5 years from the same common or maintenance area on each floor sampled during the reoccupancy sampling round; and,
  - 5 years after the initial reoccupancy air sampling round from each residential unit unless a tenant moves out of the unit before the 5-year window in which case an air sample will be collected prior to the next tenant occupying the unit.

- Wipe Sampling – Wipe sampling of exposed encapsulant surfaces within each residential unit will be performed on an annual basis to evaluate whether residual PCBs within the substrates have diffused

into the encapsulant layers. At least 2 representative wipe samples will be collected from each residential unit.

- Supplemental Groundwater Monitoring – An additional round of groundwater samples will be collected from the two Site monitoring wells and the samples analyzed for pesticides, polyaromatic hydrocarbons (PAHs), arsenic, lead, and copper to demonstrate compliance with the RSRs.

- Groundwater Compliance Monitoring - The proposed soil remedial activities are primarily driven by exceedances of the DEC rather than the PMC. If verification soil sampling indicates compliance with the GB-PMC, compliance groundwater monitoring will not be required to demonstrate compliance with the RSRs.

These sampling programs are summarized in **Table B-1** included in **Appendix B**.

A7 DATA QUALITY OBJECTIVES AND CRITERIA

Data Quality Objectives (DQOs) are qualitative or quantitative statements that specify the quality of the data needed to support specific decisions. The DQOs are the starting point in designing a sampling program. The DQO development process matches sampling and analytical capabilities to the specific uses and ensures that the quality of the data meets the project requirements.

Phoenix Environmental Laboratories (Phoenix) of Manchester, Connecticut is the primary subcontract laboratory for general analytical services for this project. Certain specialty laboratory analyses will also be provided by Alpha Analytical (Alpha) of Westborough, Massachusetts. The reporting detection limits (RDLs) established by the two laboratories for the project are contained in **Table B-2** included in **Appendix B**.

Project Action Limits (PALs), which are presented in **Table B-2** in **Appendix B**, are target levels driven by the regulatory project requirements. PALs are provided for comparison to the laboratory's RDLs to ensure they are sufficient for comparison to the applicable regulatory criteria. The PALs for this project include the following:

- Residential Direct Exposure Criteria (R-DEC) and the (GB-PMC) for soil (non-PCB)
- Unrestricted, High Occupancy limit for PCBs in concrete and soil (1 mg/kg)
- Surface Water Protection Criteria (SWPC) for groundwater samples
- Non-Detect for PCBs in wipe samples
- Less than 5.0 nanograms per cubic meter (ng/m^3) of PCBs in reoccupancy indoor air samples
- Less than 5.0 ng/m^3 of PCBs in long term indoor air samples

Performance acceptance criteria for the data will be based on principal DQOs including precision, accuracy, completeness, representativeness, and comparability. When the DQOs are not met, corrective actions will take place as described in Section C1 of this QAPP.

When laboratory QA/QC objectives are not met, corrective actions will take place per the laboratory's Quality Assurance Manual (QAM) and Standard Operating Procedures (SOPs) included in **Appendix C**. The assessment methods used to evaluate data quality are summarized below.

Data Precision

Precision is an indication of the reproducibility of measurements under a given set of conditions, or a quantitative measure of the variability of a group of measurements compared to their average values.

Laboratory precision will be evaluated by comparing laboratory duplicate analyses (laboratory control samples and laboratory control sample duplicates). Duplicate measurements of a specific parameter spiked into aliquots (or subsamples) of the same sample are obtained by the same analytical method under identical conditions. Precision of analytical measurements will be expressed as Relative Percent Difference (RPD), calculated as follows:

$$\text{Relative Percent Difference (RPD)} = \frac{|C_1 - C_2| \times 100}{(C_1 + C_2)/2}$$

Where: C_1 = analyte concentration of first analysis
C_2 = analyte concentration of replicate analysis

Acceptable limits for laboratory data precision are defined by the laboratory and included in the applicable SOPs included in **Appendix C**. For some analyses, each analyte has separate, specific acceptance ranges based on statistical parameters defined by the analytical method. According to the laboratory SOP, these statistical limits are updated annually.

Accuracy

Accuracy is the measure of agreement between an analytical result and the true value of the parameter measured. Potential errors include those associated with sample collection, sample preservation, sample handling, matrix effects, sample analysis and data reduction. Accuracy will be evaluated from the analysis of blanks, which detect positive biases, and from the analyses of spiked laboratory control samples, which will be expressed as percent recovery of spiked analytes. Laboratory QC for determining analytical accuracy will consist of: laboratory control samples, which are known quantities of target analytes added to representative samples prior to sample extraction (or digestion) and analysis; surrogate spikes, which are known quantities of organic compounds similar to the analytes of interest added to all samples and QC check samples prior to extraction and analysis; and check standards, which are known quantities of organic compounds similar to the compounds of interest added to blank samples (i.e., DI water) and analyzed every 12 hours or run of 20 samples.

Analytical laboratory accuracy is determined by comparing results from the analysis of matrix spikes, surrogates, or check standards to their known value. Accuracy results are expressed as percent recovery.

$$\text{Accuracy, as Percent Recovery} = \frac{(C_s - C_u) \times 100}{(C_k)}$$

Where: C_s = results of spiked sample
C_u = results of unspiked sample
C_k = amount of spike added

Accuracy is critical when the data are compared to "absolute" criteria (e.g., Permit discharge limits).

Acceptable limits for accuracy are defined by the laboratory and are included in the applicable SOPs included in **Appendix C**. For some analyses, each analyte has separate, specific acceptance ranges based on statistical parameters defined by the analytical method. According to the laboratory SOP, these statistical limits are updated annually.

Completeness

Completeness of data collection is a measure of the amount of valid data obtained as a percentage of the amount that was specified or expected to be obtained under normal conditions. For 100 percent completeness to be achieved, the valid data must be equal in quantity to the amount expected to be collected. Completeness will generally be less than 100 percent due to difficulties in sample collection and analysis of environmental samples. Sources of error affecting completeness include, but are not limited to: missing scheduled sampling events; submitting improper quantities of samples; sample leakage or breakage during transport or handling; improper sample preservation; missing prescribed holding times; losing a sample during analysis; improper documentation compromising traceability; or failure to conform to QC criteria specifications. If these problems occur, the Project Manager will be notified to make a determination as to the appropriate corrective action as discussed in Section C1 of this QAPP.

Completeness must be viewed on a relative basis because the required amount of valid data anticipated or specified prior to sampling may overstate the amount of data necessary to render a correct decision. In general, a higher completeness rate (approaching 100 percent) is required when only a limited amount of data is planned to be collected for making a decision. For this project, substantial data will be collected, so completeness is considered an important but not critical parameter. Therefore, an overall completeness rate of 90 percent will be considered generally acceptable and will be the standard applied to combined field and laboratory procedures for this project.

$$\text{Percent Completeness} = \frac{\text{(Number of Valid Results)} \times 100}{\text{Number of Expected Results}}$$

Representativeness

Representativeness is a qualitative parameter that assesses the suitability of the design of the sampling program for obtaining samples that are truly representative of site conditions. Both the sampling program and the analysis program are measured for representativeness. Ideally, samples

represent a characteristic of a population; in this application, an environmental condition. Adequate representativeness is achieved by properly selecting sampling locations, adhering to adequate sampling techniques, and collecting a sufficient number of samples. For this project, sampling locations were previously selected to represent the range of conditions at the Site.

Comparability

Comparability is the extent to which data from one dataset can be compared directly to similar or related datasets and/or decision-making standard based on the parameters of precision, accuracy, representativeness, and completeness. Data comparability will be achieved by continuity of laboratory practices, method analysis, sample collection procedures and sample handling. It is only when these data sets are known that data can be compared with confidence. This is achieved by adhering to recognized protocols for the collection and analysis of samples that are representative and reporting the analytical results in standardized units. The qualitative analytical results (i.e., specific compounds identified) to be obtained are expected to be within a similar range as to available data for samples collected previously from this Site.

The goal of the laboratory QA/QC is to achieve the acceptance criteria for a given analytical method when samples are analyzed. The quality of the data is indicated by the parameters of precision, accuracy, representativeness, and completeness. Thus, if each method for the analyses meets the QA/QC criteria, the results from these methods can be compared.

Analytical and reporting procedures of the laboratories support the comparability of analytical measurements: standard analytical methods with similar QC standards are used; traceable calibration standards are used; results are expressed in units consistent with each matrix and with standard industry practice; and QA and QC procedures are applied uniformly to all samples.

A8 SPECIAL TRAINING/CERTIFICATION

Personnel responsible for the performance of remediation and sample collection activities for this project are required to maintain Occupational Safety and Health Administration (OSHA) 40-hour training and subsequent annual 8-hour refresher training. Documentation of training will be maintained by the designated Health and Safety Coordinator, Mr. Richard Ecord of GZA.

It is GZA's corporate policy that appropriate and effective health and safety practices will be integrated into all daily operations. In accordance with this policy, GZA implements individual programs and procedures to promote safe and healthful working conditions for its employees, meet OSHA and other applicable local, State, and federal laws and regulations, and curtail workplace injuries and illnesses.

A9 DOCUMENTS AND RECORDS

This QAPP will be maintained by the Project Manager, Mr. Anthony Trani. The Project Manager will be responsible for maintenance and distribution of the QAPP and any revisions to those people included on the distribution list.

Field Data

Field data may include written field notes, soil classification, excavation depths and limits, soil sampling depths, depth to groundwater measurements, field sampling sheets, photos, and laboratory chains of custody.

Field notes will include, at a minimum:

- Site location;
- Date of field activities;
- Names of GZA personnel on Site and/or collecting samples;
- Names of other personnel or subcontractors present, if any;
- Field observations;
- Sampling equipment used (including make model and serial number) and equipment calibration documentation;
- Field screening methods, if used;
- Field screening results;
- Fixed laboratory sample identification number, sample type (composite, grab), time of sampling, sample analysis;
- Sample handling, packaging, labeling, and shipping information (including destination); and
- Other pertinent information.

Soil, air, concrete, wipe, and groundwater sampling information will be recorded directly on applicable field sampling sheets or project specific sampling logbooks.

Project Reports

Remedial Action Reports will be prepared for soil to demonstrate compliance with the RSRs and for the PCB impacted materials that are regulated under 761.61 following the completion of the remedial and/or the building renovation activities. If groundwater monitoring is warranted, a Groundwater Monitoring Report will be submitted following four quarters of sampling. Electronic versions of the reports will be submitted to EPA and CTDEEP.

The laboratories will provide the analytical reports along with a copy of pertinent QC data to the Project Manager. All field reports and field data will be provided to the Project Manager and maintained electronically for use in preparing the abovementioned reports.

Quality Assurance/Quality Control Reporting

The Remedial Action Reports and Groundwater Monitoring Report (if necessary) will include a section and table summarizing whether the quality control criteria in this QAPP were met in the field and in the laboratory. The report will include a discussion of any QA/QC problems and how they were resolved. GZA will note anything unusual that is anticipated to affect the quality or usability of the data.

GROUP B DATA ACQUISITION

B1 SAMPLING PROGRAM DESIGN

Consistent with GZA's *Remedial Action Plan* (May 2023), Weston & Sampson's *Building Materials PCB Remediation Plan* (December 2021), and GZA's *Notification of Self-Implementing Cleanup PCB Impacted Concrete and Soil* Report (January 2023), the sampling programs covered by this QAPP are designed to meet the following objectives:

- Demonstrate compliance with the R-DEC and GB-PMC for soil;
- Demonstrate residual PCB concentrations in soil and concrete are below the unrestricted, high occupancy level of 1 mg/kg;
- Demonstrate compliance with the SWPC for groundwater;
- Determine baseline indoor air PCB concentrations prior to the performance of abatement activities to allow performance of an assessment of potential exposure risks of future building tenants;
- Demonstrate indoor air PCB concentrations are below 5.0 ng/m^3 following the building restoration activities and prior to occupancy of the building; and
- Evaluate the long-term effectiveness of the encapsulants and barriers at mitigating potential exposure risks to the residual PCB impacts.

B2 SAMPLING METHODS

The primary sampling methods for the collection of solid, air, and aqueous samples to achieve the project objectives include the following:

- Verification samples for PCBs in soil and concrete will be extracted using EPA Method 3546 (Microwave extraction) and analyzed using EPA Method 8082 on an accelerated turnaround. Verification sampling will be performed on a modified Subpart O basis. Samples will be collected on a 1.5-meter grid pattern either from the upper 0.5-inches of concrete or the upper 3-inches of soil. Up to 4 adjacent samples will be composited. If the results of the composited sample are equal to or above 0.25 mg/kg (1/4 of the remedial target of 1 mg/kg), then the individual samples comprising the composite will be analyzed to determine where additional removal of concrete or soil is required. Additional remediation will be performed until all individual verification sample results meet the established remedial goal of less than 1 mg/kg or the results of composite samples are below the remedial goal divided by the number of samples (e.g., the target residual concentration for a composite sample consisting of 2 individual samples will be 0.5 mg/kg [1 mg/kg divided by 2 individual samples]).

- Verification sampling for other contaminants will be collected and analyzed based on the following:
  - At least one soil sample for every 20 feet of excavation sidewall and one sample every 400 square feet of excavation footprint will be analyzed for PAHs, arsenic, and lead; and,
  - Soil samples will be collected within AOC-6 from the bottom and the four sidewalls of the tank grave. The tank grave samples will be analyzed for extractable total petroleum hydrocarbons (ETPH), aromatic volatile organic compounds (VOCs) via EPA Method 8260, and semi-volatile organic compounds (SVOCs) via EPA Method 8270.

- Groundwater Sampling – Groundwater samples will be collected using low flow sampling procedures consistent with EPA's *Standard Operating Procedure Low-Stress/Minimal Drawdown Ground-Water Sample Collection* dated November 2022.

- Wipe Sampling – Wipe samples will be collected using a 10 centimeter (cm) by 10 cm template to outline the sample area and a gauze pad saturated with hexane. The hexane-saturated wipe will be used to thoroughly swab the area inside the 100 cm^2 template.

- Air Sampling

  - Baseline indoor air samples will be collected within containment units constructed with timber and polyethylene sheeting walls to isolate each unit from the rest of the building. The containment units will be approximately 25 feet long by 25 feet wide; however, the actual size of each containment unit will be adjusted based on access and logistical constraints. Approximately 1 week after construction of the containments, indoor air samples will be collected from within each containment unit using low volume polyurethane foam (PUF) samplers over an 8-hour period and then analyzed by Method 8270E/680.

  - Reoccupancy Air Sampling and Long Term Air Samples will be collected using EPA Method TO-10A from a height of approximately 3 feet above the floor surface and analyzed for PCBs by homologs using EPA Method 680.

These sampling programs are summarized in **Table B-1** included in **Appendix B**.

B3 SAMPLE HANDLING AND CUSTODY

The following describes the general sample handling and custody procedures for this project. **Table B-3** included in **Appendix B** indicates the maximum hold time for each applicable analytical test method.

Appropriate sample containers and preservatives will be provided by the laboratory as summarized in **Table B-3** in **Appendix B**. Soi, groundwater, concrete, and wipe samples will be stored on ice within plastic bags or ice packs in coolers during the sampling activities and shipment. If temporary storage is necessary, samples will be transferred from coolers to refrigerators temporarily, and back to coolers with ice within plastic bags or ice packs for subsequent transport or shipment to the laboratory. Once

sample custody is transferred to the laboratory, the laboratory will assume responsibility for maintaining sample temperatures at 4°C plus or minus 2°C.

Upon laboratory arrival, the samples will be inspected by the Sample Custodian, or other qualified laboratory personnel, who will record any breakage, the number of containers received, preservatives, temperature of the cooler, and other factors that may affect quality. The samples will be compared to their descriptions on the Chain-of-Custody (COC) form; discrepancies in the number or the designations of the samples will be noted on the form, brought to the attention of the Project Manager and the sampling personnel, and resolved at their instruction. The COC form will be signed, and the date and time recorded to formally accept the samples into laboratory custody. Sample shipments will be logged in and tracked by job number, date received, sample type and number, and analysis requested.

With the exception of air samples, samples will be stored by the laboratory in secure refrigerators before and after analysis. Refrigerator temperatures are maintained at 4°C plus or minus 2°C and are monitored each business day.

Sample Labels

Labels will be affixed to each sample container with at least the following information: Sample number/ID, date and time of sample collection, sample depth (soil and concrete samples only), the laboratory analysis, preservation type (if any), and name of sampler.

COC Procedures

Samples will remain in the sample collector's view at all times, unless locked in a vehicle or other secure place. It is the sampler's responsibility to ensure that the samples are not tampered with prior to their delivery to the analytical laboratory. The COC form will be completed to provide documentation tracking sample possession and handling from the time of collection through delivery to the analytical laboratory and will accompany the samples at all times.

The original COC form will be retained in the laboratory files and copies will be retained by project personnel. Photocopies or photographs of the completed COC form must be taken each day by the sample collector prior to relinquishing the samples to any other entity.

Sample Shipping

Sample custody will be documented to support the DQOs. With the exception of air samples, samples will be packed into a cooler with loose ice cubes for delivery to the laboratory or shipping facility. The temperature requirement during all sample handling is 4° ±2° C (allowing for a reasonable cool-down period after sample collection). The cooler and the COC will be transported to the laboratory either by common carrier, a laboratory courier, or delivered directly to the laboratory by the sampler.

Laboratory Sample Management

The samples will be inspected by the Laboratory Sample Controller, or other qualified laboratory personnel. A Sample Receipt Checklist will be used to document the receipt of the samples and will include a check for breakage, correct container and preservative, temperature of the cooler, holding times, and for other factors that may affect quality. The samples will be compared to their description on the COC form. Discrepancies in the number or the designations of the samples will be noted on the form, brought to the attention of the Project Manager, and resolved at the Project Manager's instruction. The COC form will be signed, and the date and time recorded to formally accept the samples into laboratory custody.

With the exception of air samples, once samples have been labeled with unique laboratory identification numbers, they will be placed in the laboratory refrigerator.

B4 ANALYTICAL METHODS

Table B-1 included in **Appendix B** outlines the analytical test methods for this project.

B5 ANALYTICAL QUALITY CONTROL

The quality objectives and criteria for measurement data include the following:

- Obtain a minimum of 90% data completeness for data analysis samples;
- Achieve the laboratory's precision and accuracy targets specified in the SOPs included in **Appendix C**; and
- Achieve reporting limits consistent with the PALs.

Laboratory Control Samples

Control samples include all QA/QC samples required by the methods performed including method detection limit studies, check standards, laboratory control samples, laboratory control sample duplicates, surrogate spiked samples, and method blanks. The Data Validator will have access to the method detection limit studies. In addition, all calibration data will be included in the data package. The results of surrogate spikes, laboratory control samples, and laboratory control sample duplicates will be used to evaluate accuracy and precision of laboratory analyses.

Surrogate Spiked Samples

Known quantities of organic compounds similar to target compounds will be added by the laboratory to all samples that are analyzed for ETPH, VOCs, PCBs, PAHs, and pesticides. Percent recovery of these compounds will be taken as a measure of recovery of analytes of interest in the sample. Note that it is not

appropriate to employ surrogate spikes as a QC measure for the analysis of inorganic or "indicator" (e.g., TOC) analyses.

Matrix Spiked Samples

Known quantities of analytes of interest will be added to duplicate fractions of certain samples. One matrix-spiked and duplicate matrix-spiked sample will be collected at a rate of one per 20 samples collected during the sampling events. Percent recovery of these compounds will be taken as a measure of analytical accuracy. Relative percent differences between the duplicate matrix spike samples will be taken as a measure of the precision of the analysis.

Calibration

Calibration procedures for each of the laboratory instruments are provided in the Laboratory SOPs included in **Appendix C**. Check standards will be analyzed every 12 hours or as required by the method.

Method Blanks

Method blanks will be prepared in the laboratory from analyte-free water and analyzed in the same manner as environmental samples. As required by analytical protocols, one method blank will be run each day or for each analytical batch of 20 or fewer samples (whichever is more frequent) to document laboratory sources of contamination.

Trip Blanks

Trip blanks will be prepared by the laboratory using containers identical to those pre-prepared containers sent to the field for analyses. For each day during which samples are collected and shipped for VOC analyses, at least one trip blank will accompany each shipment of sample containers to the Site and back to the laboratory. Trip blanks will be analyzed for VOCs to identify any potential contamination resulting from the handling, sampling, and analytical processes.

Equipment Blanks

Equipment blanks will be collected daily during the soil and concrete verification sampling. Equipment blanks will be collected by running laboratory provided water through the decontaminated field sampling equipment and directly into sample containers.

Equipment blanks will not be collected for the wipe sampling, air sampling, or groundwater sampling activities since these sampling activities utilize dedicated sampling equipment.

Blind Field Duplicates

Blind field duplicate samples will be collected at a rate of one per 20 samples during soil and concrete sampling events. In addition, one blind duplicate sample will be collected during the reoccupancy air

sampling. Results of the original sample and field duplicate will be compared, and the Relative Percent Difference will be calculated as follows:

$$\text{Relative Percent Difference (RPD)} = \frac{|C_1 - C_2| \times 100}{(C_1 + C_2)/2}$$

Where: C_1 = analyte concentration of first analysis
C_2 = analyte concentration of replicate analysis

Acceptable limits for the RPD for the blind field duplicates are less than 30%.

Matrix Spike/Matrix Spike Duplicate

A project specific matrix spike/matrix spike duplicate (MS/MSD) sample will be analyzed for each 20 samples obtained during the concrete and soil verification sampling.

Corrective Action Procedures - Field & Laboratory

Field technical personnel will report nonconformance or deficiencies, along with recommendations for feasible corrective action to the Project Manager. Activities using suspected equipment or procedures will be suspended until the deficiencies are corrected or explained. The Project Manager will be responsible for evaluating the incident and implementing and approving corrective action. A report on each incident will be kept on file by the Project Manager.

The report will address the following:

- The nature and extent of the nonconformance or deficiency;
- A review for errors and omissions of the applicable records, such as field notes, log sheets, chain-of-custody forms, maintenance and calibration records, and other records as deemed necessary;
- The effect of the nonconformance or deficiencies on data quality and integrity;
- An analysis of recommended corrective actions; and,
- The corrective action taken, its justification, and any lasting effects of the initial incident or the corrective action taken.

Potential corrective actions for field activities include:

- Correcting errors on chain-of-custody forms;
- Re–assigning analysis (if holding time criterion permits);
- Examination of calculation procedures; and
- Reassignment of sampling and analyses using a different batch of containers if Trip Blank contamination is suspected or reported.

If the results of this review identify that a sample or sample set does not meet Project QA criteria, the sample set will be disregarded, and the level of uncertainty or inaccuracy will be acknowledged by flagging the data and providing an explanation of the qualifications.

In addition, laboratory corrective actions will comply with the requirements of the laboratory's QAM (**Appendix C**) and will be reported to the Project Manager. Examples of potential Laboratory corrective action initiators include:

- Accepting data but qualifying the results;
- Rerunning analysis (if holding time criterion permits);
- Recalibration with fresh standards; and
- Examination of calculation procedure.

B6 FIELD SCREENING METHODS

Field screening with portable hand-held equipment will be performed during the field sampling events. The following table outlines the objectives of these field screening activities, the parameters to be measured, and the equipment used.

Field Screening Program	Objective	Measured Parameters	Equipment
Screening of emissions during performance of remedial excavation and during verification soil sampling	Monitor VOC levels	Organic Vapors	Photo-Ionization Detector
Screening of purge water during groundwater sampling events	Evaluate when parameters have stabilized and samples can be collected	pH, Specific Conductance, Oxygen Reduction Potential, and Dissolved Oxygen	Multi-Parameter Water Quality Meter
Screening of purge water during groundwater sampling events	Evaluate when parameters have stabilized and samples can be collected	Turbidity	Turbidity Meter

B7 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The table below provides a summary of preventive maintenance steps to ensure proper functioning of field equipment. The Project Manager will be responsible for ensuring the instrument inspection and maintenance schedule is achieved.

Field Equipment - Preventive Maintenance

Instrument	Activity	Frequency
Photo-Ionization Detector	Battery check Inspect vapor element Calibrate	Daily
Multi-Parameter Water Quality Meter	Battery check Calibrate	Daily
Water Level Indicator	Battery check	Daily
YSI Multiparameter Sonde with Flow Cell	Battery check Calibrate	Daily
Turbidity Meter	Battery check Calibrate	Daily

In the case of field equipment failure, backup equipment will be provided.

All non-dedicated sampling equipment including water levels and electrical wires will be decontaminated between samples and at the end of the day.

B8 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

This section provides a summary of performance requirements for field screening equipment; required corrective actions should equipment fail; and references to relevant SOPs for the project. The procedures and acceptance criteria outlined in this section and the SOPs included in **Appendix D** are generally consistent with EPA's Region 1 SOPS outlined within the March 2017 *Standard Operating Procedure Calibration of Field Instruments* guidance document.

Field Equipment - Calibration and Corrective Action

Instrument	Calibration Frequency	Calibration and Calibration Check Standards	Acceptance Criteria (both daily Calibration and End of Day Check	Corrective Action	SOP Reference
Photo-Ionization Detector	Daily Calibration and check at the end of the day/1	100 ppm isobutylene-in-air and zero air	+/- 10%	Re-analyze standard. If it is still outside the acceptance criteria, then replace with a different meter.	SOP D-11

Instrument	Calibration Frequency	Calibration and Calibration Check Standards	Acceptance Criteria (both daily Calibration and End of Day Check	Corrective Action	SOP Reference
YSI Multiparameter Sonde with Flow Cell					
Dissolved Oxygen & temperature	Daily calibration at the beginning and calibration check at the end of the day[/1]	Calibrate to 100% water saturated air and check in 0 mg/L DO solution	Less than 0.5 mg/L in the 0 mg/L DO solution (Negative readings are not acceptable)	**Morning Calibration Check –** If outside the criteria during the morning calibration, replace the appropriate calibration standards and recalibrate/check. If recalibration is unsuccessful, replace the unit. **End of the day Calibration Check –** If outside the criteria at the end of the day, the data will be qualified by GZA	SOP D-19
Oxygen Reduction Potential		Zobell solution (calibration and check)	+/- 5%		SOP D-19
Specific Conductance		Calibrate to 1,413 µS/cm and use 718 µS/cm to check. Use 718 µS/cm to check at end of day.	+/- 5% of standard or +/- 10 µS/cm (whichever is greater)		SOP D-19
pH		Calibrate to pH 4, 7, and 10 and use pH 7 to check	+/-0.3 units in pH 7 buffer		SOP D-19
Turbidity Meter	Daily calibration at the beginning and calibration check at the end of the day[/1]	Calibrate to 1000, 10, and 0.02 NTUs and use 10 NTUs to check	+/- 10% (9 to 11 NTUs)		SOP D-19

Note:

1. The objective of the end of the day check is to compare readings/measurements of the instrument against the calibration standards. This is not recalibration but rather a check.

Daily instrument calibration and checks will be documented on the form included in **Appendix D**.

B9 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

All field sampling collection supplies and standard materials such as calibration fluids, sampling tools, personal protective equipment, and other consumables for collecting samples will be provided and inspected by GZA's Field Team Leader prior to mobilizing to the Site. All analytical sampling containers, preservatives, deionized water, reagents, etc., will be provided by the laboratory and will be inspected by GZA's assigned Field Team leader prior to mobilizing to the Site.

B10 DATA MANAGEMENT

GZA assigns a project number under which all electronic and paper records will be stored. Project plans, analytical data, laboratory reports, tabulation of results and copies of field logbooks and field data forms will be maintained in the GZA file and reviewed by the Project Manager to evaluate the usability of the data and assure compliance with the elements of this QAPP. Electronic project files shall be located on GZA's computer network server, which is backed up on a regular basis by GZA's IT Department.

Retention and archiving of records shall be completed in accordance with the current GZA policy. We anticipate that GZA will maintain electronic records for this project for five (5) years following completion of the project at a minimum. To the extent that records retention is served, GZA reserves the right to adopt new storage systems which may become standard practices.

Alpha and Phoenix will retain analytical results and related records in accordance with their record keeping policies as noted in their quality manuals.

GROUP C DATA ASSESSMENT

C1 ASSESSMENT AND RESPONSE ACTIONS

Field Sampling Assessment

The assigned Field Team Leader will oversee field sampling activities and sample handling so that activities are conducted consistent with this QAPP, GZA's *Remedial Action Plan* (May 2023), Weston & Sampson's *Building Materials PCB Remediation Plan* (December 2021), and GZA's *Notification of Self-Implementing Cleanup PCB Impacted Concrete and Soil* Report (January 2023). The assigned Field Team leader will report to the Project Manager with regard to the assessment of QAPP implementation. The Project Manager and Field Team Leader have the authority to stop work should a deviation be identified at which time appropriate corrective response actions will be taken to remedy the situation.

Corrective Response Actions

Any suspected problems will be brought to the attention of the Project Manager. Immediate corrective action steps to be taken by the Field Team Leader and Project Manager will include:

- Identification and definition of the problem;
- Investigation of the problem;
- Determination of the cause of the problem and appropriate corrective action;
- Verification that the problem has been corrected;
- Modification of procedures, as necessary, to prevent recurrence; and
- Documentation of the events.

Laboratory Assessments

The following will be examined when evaluating analytical data: container handling, sample transport, adherence to holding times, calibration documentation, field and laboratory sample identification, raw data, QC limits, method detection limits, calculations, units of measurement, and sample documentation.

Lab Analyst Review

The Lab Analyst will review his/her work and report the data. This data will be reviewed for final acceptance by the Laboratory Supervisor and/or Lab Quality Control Officer. Factors to be considered in assessing analyte acceptability and validity will include initial condition of the sample; matrix spike recoveries; percent differences between matrix spike duplicates; surrogate spike recoveries; instrument QC;

and method blank precision and accuracy results from the batch. Precision will be measured as the relative percentage difference for each analyte in matrix spiked duplicates.

Lab Management Review

If the data is acceptable, the Laboratory Supervisor will release it and a final report will be generated. The Laboratory Supervisor will determine corrective action if the data is not acceptable. Any problems that are encountered would be handled by corrective action and an additional review of the data would occur.

C2 REPORTS TO MANAGEMENT

The Project Manager will be responsible for communicating with the Principal-In-Charge regarding project status, performance evaluations, periodic data quality assessments, and will report any significant quality assurance problems, should they occur. Routine and frequent email and telephone communication is anticipated, with meetings as needed.

GROUP D DATA USABILITY

D1 DATA REVIEW AND USABILITY

The CTDEEP Quality Assurance/Quality Control (QA/QC) Work Group finalized Reasonable Confidence Protocols (RCPs) in November 2007 which provided guidelines for QA/QC procedures for analytical methods and reporting. Each Alpha and Phoenix laboratory data package will be prepared consistent with the requirements of the RCPs.

The Laboratory Supervisor will be responsible for reporting to the Project Manager any deficiencies in the data quality. The Laboratory Supervisor, the Laboratory Quality Assurance Officer, and the Project Manager will decide upon the action to be taken with respect to questionable data. Data that fail to meet the project requirements and that cannot be readily rectified or qualified as estimated may be rejected. Data qualifiers include the following:

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

NJ - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

D2 VERIFICATION METHODS

The Project Manager will the data quality according to the CTDEEP *Data Quality Assessment and Data Usability Evaluation Guidelines* (May 2009; revised December 2010). This assessment will include the following:

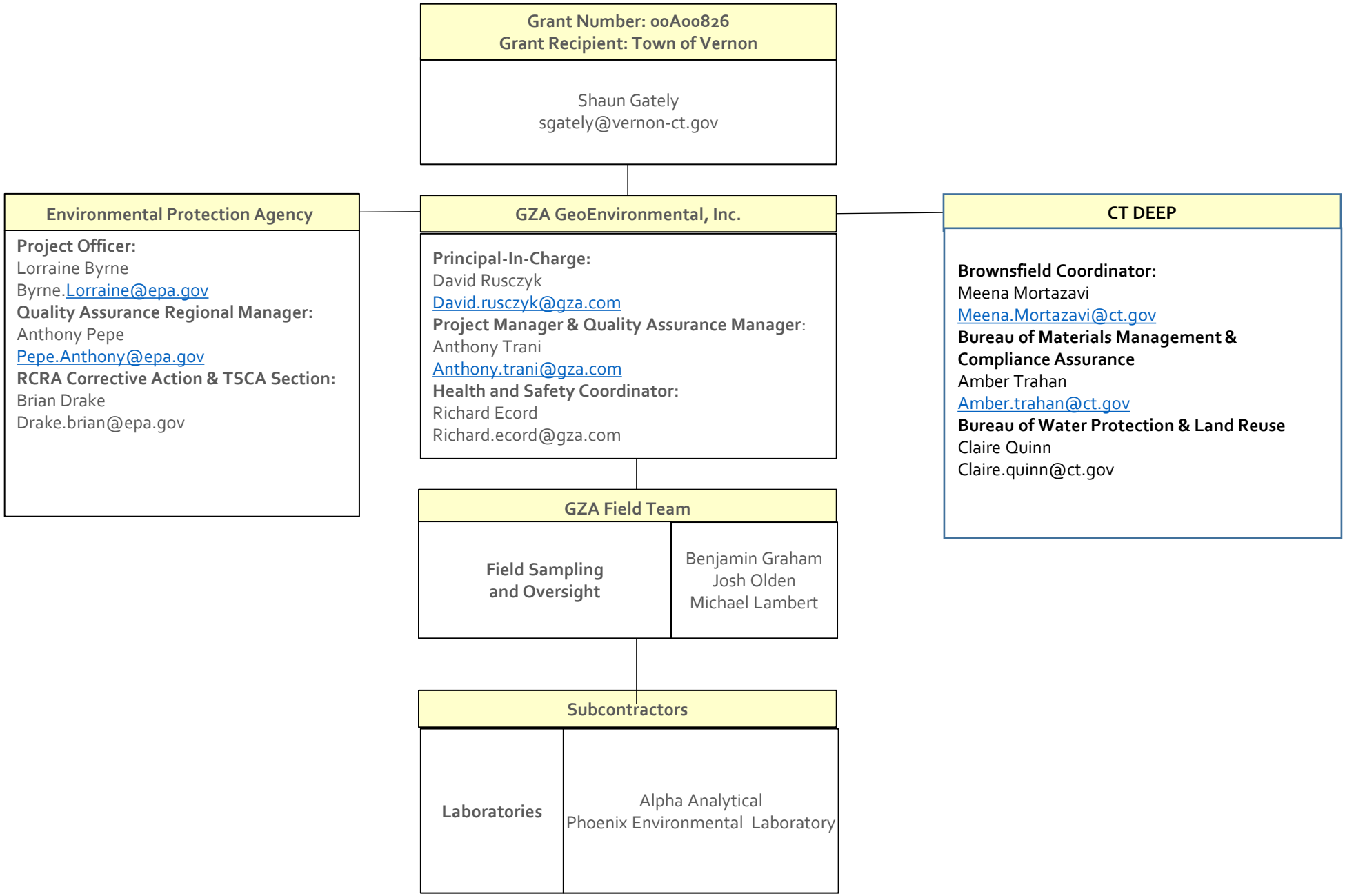
- Review of sample holding times, sample preservation methods, and sample integrity;
- Review of laboratory method blanks and trip blanks
- Review of surrogate recoveries;
- Review of matrix spike, matrix spike duplicate and laboratory control sample data;
- Review of laboratory and field duplicate data;
- Assessment of compound identification, quantification, and detection limits; and
- Review of sample documentation including chain of custodies and sample receipts.

FIGURES

APPENDIX A

ORGANIZATIONAL CHART AND PERSONNEL CONTACT INFORMATION

Appendix A
Organizational Chart and Personnel Contact Information



APPENDIX B

MONITORING PROGRAMS, PROJECT ACTION LEVELS AND LABORATORY REQUIREMENTS

Table B-1
Summary of Sampling Programs
Former Daniel's Mill
Vernon, Connecticut

Sampling Program	Area of Concern (AOC)	Matrix	PCBs	Aromatic VOCs	SVOCs	PAHs	ETPH	Pesticides	Metals	SPLP	Notes
Verification Sampling	AOC-3	Soil	X							X	SPLP PCBs on representative sample with higher PCB concentration to evaluate need to perform groundwater compliance monitoring
Underground Storage Tank Closure/Verification Sampling	AOC-6 (Exterior)	Soil		X	X		X				
Verification Sampling	AOC-6 (Exterior)	Soil	X			X			X (See Note 1)	X	SPLP PCBs on representative sample with higher PCB concentration to evaluate need to perform groundwater compliance monitoring. SPLP metals will be analyzed (if necessary) to determine compliance with GB-PMC based on mass results
Verification Sampling	AOC-6 (Interior)	Soil	X							X	SPLP PCBs on representative sample with higher PCB concentration to evaluate need to perform groundwater compliance monitoring
Verification Sampling	AOC-6 (Interior)	Concrete	X								
Baseline Indoor Sampling	Interior of Building	Air	X								
Reoccupancy Indoor Sampling	Interior of Building	Air	X								
Long Term Indoor Sampling	Interior of Building	Air	X								
Wipe Sampling	Interior of Building	Wipes	X								
Supplemental Groundwater Monitoring	Site-Wide	Aqueous	X			X		X	X (See Note 2)		
Groundwater Compliance Monitoring	AOC-3/AOC-6 (Interior)/AOC-6 (Exterior)	Aqueous	X								Groundwater compliance monitoring not required if SPLP PCB results are less GB-PMC

Legend:
PCBs = Polychlorinated biphenyls
VOCs = volatile organic compounds
SVOCs = semi-volatile organic compounds
PAHs = polyaromatic hydrocarbons
ETPH = Extractable total petroleum hydrocarbons
SPLP = Synthetic Precipitation Leaching Procedure
Notes:
1. Metals = Arsenic and Lead
2. Metals = Arsenic, Lead, and Copper

Table B-2
QAPP Worksheet
Former Daniel's Mill
Vernon, Connecticut

Laboratory Performing Analysis	Analysis Group	Method Description	Analyte	CAS Number	Project Action Limits (PALs)				Reporting Limit (RL)	Units
					R-DEC	GB-PMC	Air Related PAL	SWPC		
Soil Sampling										
Phoenix	Soil - VOC 8260 - 8020 List	Volatile Organic Compounds (GC/MS)	1,1,1,2-Tetrachloroethane	630-20-6	24	0.2	-	-	0.054	mg/Kg
			1,1,2,2-Tetrachloroethane	79-34-5	3.1	0.1	-	-	0.032	mg/Kg
			1,1-Dichloropropene	563-58-6	NE	NE	-	-	0.054	mg/Kg
			1,2,3-Trichlorobenzene	87-61-6	NE	NE	-	-	0.054	mg/Kg
			1,2,3-Trichloropropane	96-18-4	NE	NE	-	-	0.054	mg/Kg
			1,2,4-Trichlorobenzene	120-82-1	*21*	*14*	-	-	0.054	mg/Kg
			1,2,4-Trimethylbenzene	95-63-6	*500*	*28*	-	-	0.054	mg/Kg
			1,2-Dibromo-3-chloropropane	96-12-8	*0.09*	*0.04*	-	-	0.05	mg/Kg
			1,2-Dichloroethane	107-06-2	6.7	*0.2*	-	-	0.054	mg/Kg
			1,3,5-Trimethylbenzene	108-67-8	*500*	*28*	-	-	0.054	mg/Kg
			1,3-Dichloropropane	142-28-9	NE	NE	-	-	0.054	mg/Kg
			1,4-Dichlorobenzene	106-46-7	26	15	-	-	0.054	mg/Kg
			2,2-Dichloropropane	594-20-7	NE	NE	-	-	0.054	mg/Kg
			2-Chlorotoluene	95-49-8	*500*	*28*	-	-	0.054	mg/Kg
			2-Hexanone	591-78-6	*340*	*7*	-	-	0.27	mg/Kg
			2-Isopropyltoluene	527-84-4	NE	NE	-	-	0.054	mg/Kg
			4-Chlorotoluene	106-43-4	*500*	*28*	-	-	0.054	mg/Kg
			4-Methyl-2-pentanone	108-10-1	500	14	-	-	0.27	mg/Kg
			Acetone	67-64-1	500	140	-	-	2.7	mg/Kg
			Acrylonitrile	107-13-1	1.1	0.1	-	-	0.054	mg/Kg
			Benzene	71-43-2	21	0.2	-	-	0.054	mg/Kg
			Bromobenzene	108-86-1	NE	NE	-	-	0.054	mg/Kg
			Bromochloromethane	74-97-5	NE	NE	-	-	0.054	mg/Kg
			Bromodichloromethane	75-27-4	*18*	*0.21*	-	-	0.054	mg/Kg
			Carbon Disulfide	75-15-0	*500*	*8*	-	-	0.054	mg/Kg
			Carbon tetrachloride	56-23-5	4.7	1	-	-	0.054	mg/Kg
			Chloromethane	74-87-3	*180*	*3.6*	-	-	0.054	mg/Kg
			cis-1,3-Dichloropropene	10061-01-5	NE	NE	-	-	0.054	mg/Kg
			Dibromochloromethane	124-48-1	7.3	0.1	-	-	0.032	mg/Kg
			Dibromomethane	74-95-3	NE	NE	-	-	0.054	mg/Kg
			Ethylbenzene	100-41-4	500	10.1	-	-	0.054	mg/Kg
			Hexachlorobutadiene	87-68-3	*130*	*1.5*	-	-	0.054	mg/Kg
			Isopropylbenzene	98-82-8	*500*	*5*	-	-	0.054	mg/Kg
			m&p-Xylene	179601-23-1	500	19.5	-	-	0.054	mg/Kg
			Methyl Ethyl Ketone	78-93-3	500	80	-	-	0.32	mg/Kg
			Methyl t-butyl ether (MTBE)	1634-04-4	500	20	-	-	0.11	mg/Kg
			Methylene chloride	75-09-2	82	1	-	-	0.11	mg/Kg
			Naphthalene	91-20-3	1000	56	-	-	0.054	mg/Kg
			n-Butylbenzene	104-51-8	*500*	*70*	-	-	0.054	mg/Kg
			n-Propylbenzene	103-65-1	*500*	*10*	-	-	0.054	mg/Kg
			o-Xylene	95-47-6	500	19.5	-	-	0.054	mg/Kg
			p-Isopropyltoluene	99-87-6	*500*	*5*	-	-	0.054	mg/Kg
			sec-Butylbenzene	135-98-8	*500*	*70*	-	-	0.054	mg/Kg
			Styrene	100-42-5	500	20	-	-	0.054	mg/Kg
			tert-Butylbenzene	98-06-6	*500*	*70*	-	-	0.054	mg/Kg
			Tetrahydrofuran (THF)	109-99-9	*61*	*0.8*	-	-	0.11	mg/Kg
			Toluene	108-88-3	500	67	-	-	0.054	mg/Kg
			Total Xylenes	1330-20-7	500	19.5	-	-	0.054	mg/Kg
			trans-1,4-dichloro-2-butene	110-57-6	NE	NE	-	-	0.11	mg/Kg
	Soil - ETPH CTDPH	Extractable Total Petroleum Hydrocarbons (GC/FI)	Ext. Petroleum Hydrocarbons (C9-C36)	PHNX - TPH	500	2,500	-	-	51	mg/Kg
	Soil - PAH 8270 - SW8270D	Polycyclic Aromatic Hydrocarbons (GC/MS)	2-Methylnaphthalene	91-57-6	*270*	*5.6*	-	-	0.4	mg/Kg
			Acenaphthene	83-32-9	*1000*	*84*	-	-	0.4	mg/Kg
			Acenaphthylene	208-96-8	1000	84	-	-	0.4	mg/Kg
			Anthracene	120-12-7	1000	400	-	-	0.4	mg/Kg
			Benz(a)anthracene	56-55-3	1	1	-	-	0.4	mg/Kg
			Benzo(a)pyrene	50-32-8	1	1	-	-	0.4	mg/Kg
			Benzo(b)fluoranthene	205-99-2	1	1	-	-	0.4	mg/Kg
			Benzo(ghi)perylene	191-24-2	*8.4*	*1*	-	-	0.4	mg/Kg
			Benzo(k)fluoranthene	207-08-9	8.4	1	-	-	0.4	mg/Kg
			Chrysene	218-01-9	*84*	*1*	-	-	0.4	mg/Kg
			Dibenzo(a,h)anthracene	53-70-3	*1*	*1*	-	-	0.4	mg/Kg
			Fluoranthene	206-44-0	1000	56	-	-	0.4	mg/Kg
			Fluorene	86-73-7	1000	56	-	-	0.4	mg/Kg
			Indeno(1,2,3-cd)pyrene	193-39-5	*1*	*1*	-	-	0.4	mg/Kg
			Naphthalene	91-20-3	1000	56	-	-	0.4	mg/Kg
			Phenanthrene	85-01-8	1000	40	-	-	0.4	mg/Kg
			Pyrene	129-00-0	1000	40	-	-	0.4	mg/Kg
	Soil - PCB 8082A	Polychlorinated Biphenyl Aroclors (GC-ECD)	PCB-1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268	Various	1	0.005	-	-	0.06	mg/Kg
	Soil - 6010C	Total Metals (ICP)	Arsenic	7440-38-2	10	-	-	-	0.63	mg/Kg
	Soil - 6010C	Total Metals (ICP)	Lead	7439-92-1	400	-	-	-	0.32	mg/Kg
	Soil - 1312/6010C	SPLP Metals	Arsenic	7440-38-2	-	0.5	-	-	0.025	mg/L
	Soil - 1312/6010C	SPLP Metals	Lead	7439-92-1	-	0.15	-	-	0.01	mg/L
	Soil - SPLP PCB	Polychlorinated Biphenyl Aroclors (SPLP)	PCB-1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268	Various	-	0.005	-	-	0.005	mg/L
Air Sampling										
Alpha	Air -8270E-SIM/680M	Polychlorinated Biphenyl Congeners	Various	Various	-	-	5	-	4.2***	ng/m^3
Phoenix	Air -TO-10/680	Polychlorinated Biphenyl Aroclors (GC-ECD)	PCB-1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268	Various	-	-	5	-	5	ng/m^3
Concrete Sampling										
Phoenix	Solid - PCB 8082A	Polychlorinated Biphenyl Aroclors (GC-ECD)	PCB-1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268	Various	1**	-	-	-	1.7	mg/Kg
Wipe Sampling										
Phoenix	Solid - PCB 8082A	Polychlorinated Biphenyl Aroclors (GC-ECD)	PCB-1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268	Various	Non-Detect	-	-	-	1	$ug/100\ cm^2$

Table B-2
QAPP Worksheet
Former Daniel's Mill
Vernon, Connecticut

Laboratory Performing Analysis	Analysis Group	Method Description	Analyte	CAS Number	Project Action Limits (PALs)				Reporting Limit (RL)	Units
					R-DEC	GB-PMC	Air Related PAL	SWPC		
Groundwater Sampling										
Phoenix	Water - PCB 8082A	Polychlorinated Biphenyl Aroclors (GC-ECD)	PCB-1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268	Various	-	-	-	0.5	0.5	ug/L
	Water - PAH 8270 - SW8270D	Polycyclic Aromatic Hydrocarbons (SIM)	2-Methylnaphthalene	91-57-6	-	-	-	*62*	0.1	ug/L
			Acenaphthene	83-32-9	-	-	-	*150*	0.1	ug/L
			Acenaphthylene	208-96-8	-	-	-	0.3	0.1	ug/L
			Anthracene	120-12-7	-	-	-	1,100,000	0.1	ug/L
			Benz(a)anthracene	56-55-3	-	-	-	0.3	0.02	ug/L
			Benzo(a)pyrene	50-32-8	-	-	-	0.3	0.02	ug/L
			Benzo(b)fluoranthene	205-99-2	-	-	-	0.3	0.02	ug/L
			Benzo(ghi)perylene	191-24-2	-	-	-	*150*	0.1	ug/L
			Benzo(k)fluoranthene	207-08-9	-	-	-	0.3	0.02	ug/L
			Chrysene	218-01-9	-	-	-	*0.54*	0.02	ug/L
			Dibenzo(a,h)anthracene	53-70-3	-	-	-	*0.3*	0.01	ug/L
			Fluoranthene	206-44-0	-	-	-	3,700	0.1	ug/L
			Fluorene	86-73-7	-	-	-	140,000	0.1	ug/L
			Indeno(1,2,3-cd)pyrene	193-39-5	-	-	-	0.54	0.02	ug/L
			Naphthalene	91-20-3	-	-	-	*210*	0.1	ug/L
			Phenanthrene	85-01-8	-	-	-	14	0.07	ug/L
			Pyrene	129-00-0	-	-	-	110,000	0.1	ug/L
	Water - Total Metals-7010	Total Metals (ICP)	Arsenic	7440-38-2	-	-	-	4	4	ug/L
			Copper	7440-50-8	-	-	-	48	5	ug/L
			Lead	7439-92-1	-	-	-	13	2	ug/L
	Water - Pesticides 8081	Pesticides (GC-ECD)	Alachlor	1572-60-8	-	-	-	NE	0.075	ug/L
			Aldrin	309-00-2	-	-	-	*0.05*	0.002	ug/L
			Alpha-BHC	319-84-6	-	-	-	NE	0.025	ug/L
			Beta-BHC	319-85-7	-	-	-	NE	0.005	ug/L
			Delta-BHC	319-86-8	-	-	-	NE	0.025	ug/L
			Gamma-BHC	58-89-9	-	-	-	NE	0.025	ug/L
			Chlordane	57-74-9	-	-	-	*0.3**	0.3	ug/L
			4,4'-DDD	72-54-8	-	-	-	*0.05**	0.05	ug/L
			4,4'-DDE	72-55-9	-	-	-	*0.05**	0.05	ug/L
			4,4'-DDT	50-29-3	-	-	-	*0.05**	0.05	ug/L
			Dieldrin	60-57-1	-	-	-	0.1	0.002	ug/L
			Endosulfan I	959-98-8	-	-	-	*0.56**	0.05	ug/L
			Endosulfan II	203716-99-8	-	-	-	*0.56**	0.05	ug/L
			Endosulfan sulfate	1031-07-8	-	-	-	*0.56**	0.05	ug/L
			Endrin	72-20-8	-	-	-	*0.1**	0.05	ug/L
			Endrin Aldehyde	7421-93-4	-	-	-	*0.1**	0.15	ug/L
			Endrin Ketone	53494-70-5	-	-	-	*0.1**	0.05	ug/L
			Heptachlor	76-44-8	-	-	-	0.05	0.025	ug/L
			Heptachlor Epoxide	1024-57-3	-	-	-	0.05	0.025	ug/L
			Methoxychlor	72-43-5	-	-	-	*0.5*	0.1	ug/L
			Toxaphene	8001-35-2	-	-	-	1	1	ug/L

Notes
1. Residential Direct Exposure Criteria (R-DEC)
2. GB-Pollutant Mobility Criteria (GB-PMC)
3. Surface Water Protection Criteria (SWPC)
Note about 10x GWPC is applicable for PMC for SPLP values
Italics = No RSR crietia; APS Criteria can be used with site-specific approval
* = indicates criteria applies to sum of chlordane, DDT, endosulfan, and endrin compounds
** = indicated unrestricted, high occupancy use level
*** = Reporting limit dependent on sample volume

Legend
"-" = Not Applicable
NE = None Established
PAL = Project Action Limit
RL = Reporting Limit

Table B-3
Sample Containers, Preservation Requirements, and Holding Times
Former Daniel's Mill
Vernon, Connecticut

Matrix	Parameter	Analytical Method	Containers	Preservation Requirements	Maximum Holding Time
Soil	VOCs	8260	2 - water VOA 1 - methanol VOA	4°C	14 days to extract, 40 days to analyze. Water VOAs must be submitted to lab within 48 hours of collection, or frozen within 48
	ETPH	CT ETPH	1 - 4 oz Glass Jar	4°C	14 days to extract, 40 days to analyze
	PAHs	8270	1 - 4 oz Glass Jar	4°C	14 days to extract, 40 days to analyze
	PCBs	8082A/3546	1 - 4 oz Glass Jar	4°C	14 days to extract, 40 days to analyze
	Metals (arsenic & lead)	6010C	1 - 4 oz Glass Jar	4°C	6 months
Air	PCBs	8270E-SIM/680M	1 - Low Volume PUF Sampler/Cartridge	4°C +/- 2°C	7 days to extract, 40 days to analyze
	PCBs	TO-10A/680	1 - Low Volume PUF Sampler/Cartridge	4°C	14 days to extract, 40 days to analyze
Concrete	PCBs	8082A/3546	1 - 4 oz Glass Jar	4°C	14 days to extract, 40 days to analyze
Wipe	PCBs	8082A/3546	1 - 4 oz Glass Jar	4°C	14 days to extract, 40 days to analyze
Aqueous	Pesticides	8081B	1 - 1L Amber Glass	4°C	7 days to extract, 40 days to analyze
	PAHs	8270	2 - 1L Amber Glass	4°C	7 days to extract, 40 days to analyze
	PCBs	8082A	1 - 1L Amber Glass	4°C	7 days to extract, 40 days to analyze
	Metals (arsenic, copper, lead)	7010	1 - 250mL Plastic	HNO_3, 4°C	6 months

Notes:
1. Blind duplicate samples will be collected as specified in the text.
2. Equipment/field blanks will be collected as specified in the text.
3. Matrix Spike/Matrix Spike Duplicates will be collected as specified in the text.

APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES AND QUALITY ASSURANCE MANUALS

Phoenix Environmental Laboratories, Inc.

Quality Manual

Document Control No.: 22-0912-1
Date Issued: 09/12/2022

Table of Contents

1.0 Quality Manual Identification Form

Document Title:	Quality Manual Phoenix Environmental Laboratories, Inc.
Document Control Number:	22-0912-1
Organization Title:	Phoenix Environmental Laboratories, Inc.
Address:	587 Middle Turnpike East Manchester, CT 06040
Responsible Official:	Phyllis Shiller
Title:	Laboratory Director
Phone:	(860) 645-1102
Quality Assurance Officer:	Kathleen Cressia
Title:	Director of Quality Assurance
Phone:	(860) 645-1102

Manual Coverage: Environmental Laboratories Including:

- Project Planning and Control
- Glassware Preparation and Laboratory Supplies
- Sample Receipt and Control
- Sample Extraction and Preparation Laboratory
- Inorganic Laboratory
- GC/MS Laboratory
- GC Laboratory
- Data Entry and Report Preparation
- Data Technical Review
- Customer Inquiry
- Quality Assurance

Concurrences

(1) Name: Kathleen Cressia
Title: Quality Assurance Officer

Signature: Kathleen Cressia

Date: 09/12/22

(2) Name: Phyllis Shiller
Title: Laboratory Director

Signature: Phyllis Shiller

Date: 09/12/22

2.0 Introduction

Phoenix Environmental Laboratories, Incorporated is committed to providing the highest quality laboratory services and data available. All laboratory analyses are performed in full compliance within applicable State, or Federal Quality Control guidelines. The Quality Assurance (QA) program and Quality Control (QC) procedures are defined by the Quality Manual and the Laboratory Standard Operating Procedure (SOP) Manual. The QA program meets or exceeds EPA recommended guidelines with quality control samples accounting for at least 20% of the total number of samples analyzed. Data from the analysis of these samples can be used to update control limits, or in the case of projects with defined control limits, the data serves to demonstrate the overall lab performance. Data which exceed control limits are considered suspicious and shall initiate specific actions as defined in this Manual and the SOP Manual. The Quality Assurance Office ensures that facilities, equipment, personnel, methods, records, and Quality Control procedures are in conformance with Phoenix Environmental Standard Operating Procedures (SOPs) as well as with applicable EPA Quality Control guidelines.

Each laboratory project is monitored through application of a QA/QC program, which includes the following elements:

- Centralized Project files
- Written Standard Operating Procedures
- Rigorous Chain-of-Custody procedures
- Documentation of nonconformance events and corrective actions taken
- Quality Control of data is assessed by analysis of reference samples, spiked samples, duplicates and surrogate spikes
- Periodic inspections of projects in progress
- Frequent equipment calibration and maintenance inspections
- Archiving of project records under controlled access

3.0 Quality Assurance Policy Statement

Statement of Authority and Responsibility

This document is the Quality Assurance Manual for Phoenix Environmental Laboratories, Incorporated. This Manual describes the activities necessary to meet or exceed the data quality objectives of Phoenix Environmental Laboratories, Inc.'s clients.

The management of Phoenix Environmental Laboratories, Incorporated is dedicated to the quality assurance program described in this Manual, and procedures as defined in the SOP manual. Each Director, and Supervisor as well as their staff members, as assigned in accordance with this Manual, are obligated to comply with its stated requirements, responsibilities, and objectives.

The Quality Assurance Program shall be maintained and expanded or modified as necessary to ensure all reportable data are of uncompromising quality.

The Quality Assurance Officer is responsible for the contents of the Manual and is committed to assuring that the stated requirements are met.

Kathleen Cressia — 09/12/22
Kathleen Cressia — Date
Quality Assurance Officer

Phyllis Shiller — 09/12/22
Phyllis Shiller — Date
Laboratory Director

4.0 Quality Assurance Management

4.1 Introduction

The management of the Quality Assurance Unit at Phoenix Environmental Laboratories, Incorporated is headed by the Director of Quality Assurance. The Quality Assurance Unit is independent of the data generating and Project Management groups and reports directly to the Board of Directors of the Company through the General Manager.

4.2 Assignment of Responsibilities

The Quality Assurance Office operates independently of the data generating areas for which they have quality assurance oversight. The Quality Assurance Director reports directly to the Corporate Management.

The goal of the Quality Assurance Program is to assure that data generated by Phoenix Environmental Laboratories, Incorporated is of the highest quality available. To reach this goal the program seeks to develop policies and procedures to monitor, maintain and improve data quality, and maintain the necessary documentation of laboratory performance. A listing of Quality Assurance personnel responsibilities is detailed below.

Director of Quality Assurance

The Director of Quality Assurance has the responsibility for the development and administration of the Quality Assurance Program. This effort is supported by the Laboratory Director and Assistant Laboratory Director.

Additionally, the Director of Quality Assurance's duties include:

- Preparation of written documents defining Quality Assurance and Quality Control Procedures.
- Maintaining current knowledge of approved methods and other regulatory requirements.
- Serving as a liaison to regulatory agencies in Quality Assurance matters.
- Reviewing Nonconformance Reports and corrective actions to assure that operations have been appropriately corrected.
- Employee training in Quality Control procedures and Quality Assurance practices.
- Preparation of reports of lab inspections and data reviews for the QA office and the Laboratory Director.

- Reviewing and approving performance evaluation sample results prior to submission to regulatory agencies.
- Assistance in preparation, review and approval of SOPs.
- Maintaining copies of all current procedures.
- Scheduling and performance of quality audits.
- Performance of inspections of lab operations and records to assess compliance with SOPs and contract requirements.
- Informing management of the status of the Quality Assurance Program.
- Continually assessing the Quality Assurance Program

.

The Director of Quality Assurance has the final authority to stop or change any incorrect or improper sampling or analytical procedure to assure data quality.

Quality Assurance Specialists

In addition to the Director of Quality Assurance, the Quality Assurance Office consists of technical specialists who have the responsibility and authority to monitor all phases of laboratory operations. Their functions include:

- Preparing and submitting blind QC check samples to the lab and evaluating lab performance.
- Reviewing the outcome of QC samples on a routine basis to assure that control limits are being met and internal SOPs for control chart analyses are followed.
- Immediately notifying the QA office of nonconformance events.
- Ensuring that all standards are traceable to NBS or EPA provided materials.

Quality Control Staff

An analytical quality control program is conducted to ensure the production of valid data. The QA office supervises the analytical Quality Control Program and interacts with the project staff in determining corrective action procedures. Duties of the Quality Control Staff include:

- Maintenance quality control charts for each area of the laboratory.
- Preparation of current tabular results of control charts.
- Posting of these control chart tables at each instrument and/or bench.

Laboratory Management

The laboratory management has the responsibility for directing that the laboratory sections follow the Quality Assurance Program. This obligation is met through the following steps:

- Recruiting, hiring, and training of suitably qualified personnel.
- Allocation of sufficient resources including staff, time, materials and equipment to complete required tasks.
- Integration of Quality Control measures into the Job Descriptions of laboratory personnel so that each employee is responsible for the quality of the work they produce.
- Effective response to corrective action requirements identified by the Quality Assurance Office.
- Assignment of Standard Operating Procedure development as required by Quality Assurance.
- Review and approval of SOPs.
- Evaluation of the lab performance of policies outlined in this manual including the ethics policy, the conflict of interest policy, and the client confidentiality policy. Review of the labworks audit trail is one of the mechanisms to these evaluate these policies.

Laboratory Section Supervisors

Laboratory Section Supervisors are an integral part of the implementation of the Quality Assurance Program. Each Supervisor is responsible for the quality of the data generated by their group. All activities performed in the lab section must comply with the internal Standard Operating Procedures and individual contract requirements. It is the responsibility of the Section Supervisor to train analytical personnel, prepare and update SOPs for each operation, and instruct analysts to perform QC checks at the appropriate intervals. The Section Supervisor reviews data and assures that all QC criteria for each data set have been met before releasing results for reporting. Additionally, it is the responsibility of the Section Supervisor to document nonconformance events and corrective action taken.

Chemists and Lab Technicians

It is the responsibility of the individual analysts to follow the appropriate methods, documenting their activities and results concisely, and implementing the QC checks as required by the contract and/or the Standard Operating Procedures. The analysts are expected to produce data of measurable quality and, therefore, must evaluate the outcome of QC samples as part of the regular analytical procedure. Individual analysts, as the first line of quality control, must identify quality problems and initiate a Nonconformance Report.

Coverage during Absence

In the absence of the QA Director, the QA specialists with assistance from the Lab Director cover the duties of the QA Director. In the absence of the Lab Director, the Assistant Laboratory Director with assistance from the QA Director covers the duties of the Lab Director.

4.3 Communications

The Quality Assurance Office communicates with other laboratory sections in two predominant methods, by regular staff meetings and by memorandum or report.

Management Meetings are held regularly between the Laboratory Directors, Project Managers, Laboratory Management, and the Director of Quality Assurance. In addition to production planning, marketing efforts, and laboratory management issues, Quality Assurance concerns are discussed. This forum provides immediate access to responsible individuals for the resolution of Quality Assurance concerns. Decisions made are documented in memoranda following the meeting.

Reports are issued to document findings of audits, inspections, and data reviews performed by the Quality Assurance Office. Findings and recommendations are documented in a report issued by Quality Assurance. Reports are issued to supervisors responsible for the work reviewed, and to management. The Supervisor responds to each of the findings and documents corrective actions. The report is then circulated to management for review. Quality Assurance verifies that corrective actions have been implemented and then files the report in Quality Assurance Office files.

Memoranda are generally issued to communicate results of Performance Testing (P.T.) studies or interlaboratory studies, to document problems brought to the attention of Quality Assurance, and as a form of written communication to keep laboratory staff and management informed of activities related to Quality Assurance.

In addition, reports are issued to the President and Laboratory Section Supervisors/Directors to summarize activities of the Quality Assurance Office. Quality Assurance Audit Reports and Corrective Action Reports are other forms of written communication between laboratory staff, management and the Quality Assurance Office.

4.4 Document Control

Quality Assurance Reports are maintained in locked file cabinets which are separate from other study records. Quality Assurance records are often direct and forthright in addressing problems and to allow these records to become public knowledge would hinder the performance of the Quality Assurance Office. Thus, these records are considered most confidential and are not available for inspection by persons outside the company.

Procedures described in Section 1.4.1 of the Quality Assurance Handbook for Air Pollution Measurement Systems. Volume I (EPA-600/9-76-005) and Chapter 3 of the Manual for the Certification of Laboratories Analyzing Drinking Water 5th edition, January 2005 are used in the publication of this Quality Manual and the laboratory's Standard Operating Procedure Manual.

Original copies of Standard Operating Procedure documents are maintained in the Quality Assurance Office. All SOPs are available to all employees in PDF format in the Phoenix LIMS system. SOP distribution lists are maintained by the Quality Assurance Office for those departments that also keep a hard copy SOP Manual.

Document control of this Quality Assurance Manual is basically the same as that described for the SOP documentation described above. A current and historical file system, distribution list, and limited copies of the document are used in the production of the Quality Manual to maintain its integrity. The Quality Manual is printed on Ivory paper and the current version is always available in electronic format in the LIMS system for all employees.

4.5 Quality Assurance Program Assessment

The Director of Quality Assurance and the staff of the Quality Assurance Office conduct periodic assessments of the total Quality Assurance Program. Based upon these assessments, and an annual review of the Quality Manual, an annual status report of Quality Assurance activities and progress is presented during the annual management review meeting. This report is used to define areas of focus for the coming year and will determine changes required in the Quality Manual. This report shall include such information as:

A. Status of or changes to the Quality Manual.

B. Status of Quality Assurance Project Plans (QAPjP), if any.

C. Measures of Data Quality.

D. Significant Quality Assurance problems, accomplishments, and recommendations.

E. Results of Performance Audits.

F. Results of Systems Audits.

G. Status of Quality Assurance requirements for contracts.

H. Summary of Quality Assurance Training.

4.6 Review of Requests, Tenders, and Contracts

All tentative environmental testing work that is presented to Phoenix Environmental Laboratories that may lead to a contract is reviewed to ensure that the laboratory can complete the work in a timely manner. A contract may be any written or oral agreement to provide a client with environmental testing services. The Client Services Staff along with the Quality Assurance Officer ensure that the laboratory is certified for all analyses/methods being requested along with ensuring that the laboratory is adequately staffed and equipped.

Any work that Phoenix Environmental Laboratories is unable to complete is subcontracted to other laboratories. An up-to-date certification for each subcontracted laboratory is kept on file at Phoenix Environmental Laboratories to ensure that all subcontracted work meets certification. The client is notified if any work is to be subcontracted and the client is supplied with any necessary information regarding the subcontract.

All contracts are reviewed and agreed upon by both the client and the laboratory prior to the commencement of work. All records regarding a contract are maintained, including correspondence regarding deviations from the contract or significant changes to client or laboratory requirements.

5.0 Personnel Qualifications

5.1 Introduction

Phoenix Environmental Laboratories has over 50 employees within the Laboratory with the scientific and technical expertise needed to serve the analytical needs of our clients. These employees have been chosen based upon their education, training and experience to ensure that Phoenix Environmental Laboratories Incorporated can perform their assigned tasks and successfully follow their chosen career paths.

Phoenix Environmental Laboratories, Incorporated provides its employees with opportunities for continuing education and training so that our employees may grow with the company. The benefits of supplying continuing education, training, and on the job experience are not only for the individual employee. The company benefits also, since it profits by the stability of the work force and internal promotion of its employees. Finally, the benefits to the clients are that they may have confidence in the precise and accurate performance of contracted analyses.

5.2 Qualifications

Phoenix Environmental Laboratories, Incorporated has minimum education and experience qualifications for all job categories within the laboratory. In-house training programs and policies augment these basic education and experience requirements by supplying additional information about technical subjects, safety, corporate policy, quality assurance, ethics, and supervisory and managerial techniques.

For each position, critical educational requirements, specialized training requirements, and experience have been identified. Documentation of personnel qualifications training, and experience is accomplished through the use of an Employee Training and Experience Record system. The Employee Training and Experience Files are maintained and reviewed by the Quality Assurance Office. The files contain Training forms, Job Description forms, Capability forms, and any Training and Experience Update forms that may be necessary. Additional items which may be included are copies of company resumes, copies of training certificates, or professional certifications, and any other documentation of training or educational course work.

Personnel resumes are attached as Appendix A: Resumes of Laboratory Personnel. A Laboratory Organizational Chart is attached as Appendix C.

5.3 Training

New employees are trained on a one-on-one basis with their supervisor or assigned individual. Training is initiated by discussion of applicable SOP and method documents for a particular analysis. The procedures are then demonstrated by the trainer, to be repeated by the new employee, on a set of trial samples. Results of the trainee's analysis, and an appraisal of techniques used are reviewed by the trainer. Successful results and suitable techniques are to be the basis for the qualification of an analyst in a particular procedure. Failure in either of these areas must result in additional one-on-one training. Until the trainer is convinced of the ability of the new employee, and the new employee has completed an acceptable demonstration of capabilities, the new employee may not perform analysis on client supplied samples.

After initial training, an employee's performance is monitored by their supervisor for compliance with quality, production and safety goals.

Documentation of employee training procedures is accomplished through the Employees Training and Experience files as described in Section 5.2. These training records are maintained by the Quality Assurance Office. Additionally, training is routinely performed upon the introduction of new instruments into the laboratory. Generally, these courses are provided by the instrument manufacturer who issues training certificates upon successful completion of the course. Copies of such certificates are to be placed in the employees' qualifications file.

Training is also presented in the form of seminars given to explain new methods, techniques, and procedures. These courses generally are given by senior level personnel for the benefit of those with less experience. These courses are also documented and included in the employees' qualification files.

Each employee is trained under the Federal Right-to-Know statute. We believe that employees well trained in safety issues, working in a safe environment produce a better quality product.

5.4 Data Integrity/Ethics Policy

Phoenix Environmental Laboratories, Inc. is committed to maintaining high ethical standards. This is accomplished by promoting a highly trained and motivated staff. All personnel are urged to discuss any problem or uncertainty that may have an effect on data quality. All personnel can report data integrity issues to management, confidentially and outside of the chain of command, without concern of exposure. As part of the training process, each employee is educated in the ethical and legal aspects of the analysis performed and should be confident about their responsibility for ensuring data integrity and ethical conduct in the workplace. Employees complete Ethics & Data Integrity Training annually.

Compromising standards for any purpose is unacceptable at Phoenix Labs. Any employee found to misrepresent analytical data would be disciplined up to termination. If merited, outside authorities would be notified, which could lead to civil or criminal prosecution.

Data integrity is defined as a state that exists when information is predictably related to its source and has been processed in an authorized manner.

Any data manipulation to misrepresent quality control will be considered fraud by Phoenix Environmental and will result in immediate employee dismissal.

The following practices are not tolerated by Phoenix and will result in termination:

- Time travel (reporting the analysis date incorrectly to meet the sample holding time),
- Manual integration of chromatography (not following accepted criteria) for the sole purpose of meeting QC criteria,
- Modifying a reported method without permission of the client to cut costs, save time, etc.,
- Using white out or obliterating data (The only approved method for an analyst to correct data is single line cross out with initials and date),
- Falsifying data.

5.5 CONFLICT OF INTEREST POLICY

Phoenix Environmental Laboratories, Inc. ensures that its personnel are free from any commercial, financial, and other undue pressures which may adversely affect the quality of their work; by emphasizing that potential conflicts of interest can occur.

The company has a stringent policy not to profit from any potential conflict. All personnel are urged to notify management of any known or suspected conflict. All potential conflicts are completely divulged to clients and regulatory authorities.

6.0 Facilities, Equipment and Services

6.1 Introduction

Phoenix Environmental Laboratories, Incorporated is located in Manchester Connecticut, east of Hartford CT. The facility encompasses approximately 10,000 square feet, which includes the laboratories, data processing, copy areas, and administrative offices. All entrances to the facility are locked and alarmed after hours. Supplemental security is provided by a contracted security service. Members of the staff escort visitors while in the facility.

Laboratory Safety is a important aspect of Phoenix Environmental Laboratory. The Phoenix Safety Manual is located in the general area along side the chemical MSDS volumes. The Safety program at Phoenix includes:

The role of the safety committee
The chemical hygiene Plan
The Right to Know SOP
The Hazardous Chemical Handling SOP
The Emergency Evacuation Plan
Safety Equipment Procedures

The entire facility is provided with a sprinkler system for fire protection. Additionally, there are fire extinguishers throughout the building and emergency showers, fire blankets, and eyewash stations in the laboratories.

The laboratory has a full complement of instrumentation and support equipment such as fume hoods, refrigerators, freezers, ovens, a deionized and reverse osmosis water systems, etc.

All instruments are maintained by trained employees, and by manufacturer service personnel, in some cases working under service contract for critical equipment. Instrument logbooks are maintained for each individual instrument in each of the laboratories.

A complete listing of instrumentation and equipment may be found in the Laboratory Capabilities Statement (Appendix B).

6.2 Laboratory Facilities

The analytical laboratories adjoin the administrative offices in order to provide close interaction between management and the analytical staff. Figure 1 presents a floor plan of the facility, Laboratory environmental aspects that could affect the quality of data generated are discussed below.

- **Environmental Control**

  The facility is divided into numerous laboratory and office areas each with its own requirements for airflow, exhaust, and equipment cooling. The entire facility is served by two large central HVAC units equipped with carbon filters to minimize contamination. These units are maintained by a local HVAC contractor by service agreement. Filters on the units are replaced on a quarterly basis to reduce dust and pollen infiltration into the facility. Temperature is maintained between 68" and 72" F to prevent temperature induced artifacts in the data obtained from the instrumentation. Laboratory hoods are required to have a face velocity of at least 100 linear feet per minute flow at all points across the hood face. Facility Maintenance is responsible for performing semi-annual compliance checks for all laboratory hoods. General housekeeping is provided by full-time in-house personnel. Wet mopping of all laboratory tile floors is performed regularly to provide for additional dust control. All labs and office areas are adequately lighted with fluorescent-type lighting. Emergency battery powered lighting is installed in all areas in the event of total power failure.

- **Electrical Power**

  Power is supplied to the facility via underground cable by Northeast Utilities. Service capacity is 1000 amperes at 208 volts. Three-stage surge and spike suppression equipment is employed on instrumentation sensitive to this type of power problem.

- **Laboratory Utilities**

  The laboratory benches are supplied with electrical power, compressed air, vacuum, hot and cold water, and deionized reagent water utilities, where needed. Compressed air and vacuum systems are maintained by the Facilities Maintenance. An electric water heater supplies hot water.

  There is located within the laboratory a deionized water system. The system utilizes a filter unit, anion, cation and mixed bed ion-exchange resin tanks for deionization, along with activated carbon tanks for organic contamination removal. There is also a reverse osmosis system. These systems are maintained by their contractors. The laboratory water is checked monthly for bacteria, volatiles, metals, and other inorganics. The conductivity and pH of the laboratory water is checked daily with a calibrated conductivity/pH meter.

- **Laboratory Facility Safety Engineering**

  Laboratory Safety is taken as a serious responsibility. To that end the laboratory has special solvent storage and waste storage areas.

  - Solvent supplies are stored in a large flammable solvent storage locker. Bulk solvents are stored here while small quantities of solvents for immediate use are stored in flammable solvent lockers beneath the laboratory hoods. Corrosive liquids are stored separately in corrosive liquid storage lockers.

- Waste solvents are placed in waste solvent containers for transfer to 55-gallon DOT 17H closed head drums for the accumulation and storage of laboratory wastes prior to shipment. Waste samples are generally handled as labpacks and are sent to a licensed facility for incineration.

- The entire facility is provided with a sprinkler system for fire protection. The building is equipped with dry chemical, carbon dioxide, and Halon fire extinguishers strategically placed throughout the laboratory and office areas. All laboratories are equipped with eye wash stations and drench-type safety showers. Safety glasses are issued to each employee for use in the laboratory.

- **Laboratory Areas**

  - **Shipping and Receiving/Sample Control**

    The Shipping and Receiving/Sample Control area is located immediately adjacent to the Extractions and Preparations Laboratory. The Shipping and Receiving portion of the space encompasses approximately 140 square feet of floor space. The Sample Control area encompasses approximately 250 square feet in addition to the space taken by a large walk-in refrigerator used for the storage of environmental samples. Samples arriving are inspected and entered into the laboratory sample control system at the computer entry work station. Adequate bench space is provided for the unpacking and inspection of samples upon receipt at the laboratory.

- **Walk-in Refrigeration System**

  A walk-in refrigeration system for the storage of environmental samples is located adjacent to the Sample Control area. The walk-in encompasses approximately 2500 cubic feet of storage space and the temperature is controlled to 4°C ± 2.0°C with continuous temperature sampling and monitoring devices. The temperatures are taken every 30 seconds and automatically stored into the laboratory LIMS system. The unit is maintained by the Sample Control Supervisor to maintain strict controlled access and chain-of-custody at all times.

- **Volatile Freezer**

  A temperature controlled freezer is located in the Organics Laboratory for storing EnCore and DI water preserved low level vials for Volatile analysis.

- **Extractions and Preparations Laboratory**

  The Extractions and Preparations Laboratory has nearly 2500 square feet of floor space and is equipped with several large laboratory fume hoods, steam baths for Zymark apparatus, approximately 120 linear feet of bench space, and adequate storage cabinet space necessary to process thousands of samples per month. Additional equipment in the lab includes TCLP extraction equipment including zero headspace extractors (ZHEs), Continuous liquid-liquid extractors, Soxhlet extractors, block digestors, a vacuum system, a laboratory shaker, a six-horn sonicator, a chilled water source for use with reflux equipment, Accelerated Solvent Extractors (ASEs), and analytical balances.

- **Metals Analysis Laboratory**

  The Metals Analysis Laboratory is over 1000 square feet in size. There is approximately 60 linear feet of open laboratory bench space for use in inorganic analysis. The room is equipped with special air extractors for the atomic absorption spectrophotometers and the ICPs located in the room.

The AA Spectrophotometers are Perkin-Elmer AA Analyst 600 Spectrophotometers with autosamplers and data systems which are used for Graphite Furnace Atomic Absorption (GFAA). A PSA Mercury Millennium System with a cold vapor detector is used for mercury analysis. Two Spectro Axial Simultaneous ICP with Autosamplers, are also used for metals analyses.

- **Inorganic Analysis Laboratory**

  The Inorganic Analysis Laboratory is over 2000 square feet in size. There is approximately 200 linear feet of open laboratory bench space for use in inorganic analysis. Equipment includes a GE and an Elementar TOC analyzers with autosamplers, Lachat QuikChem autoanalyzers for automated spectrometry, Dionex 120 Ion Chromatographs, and a Man-Tec automated titration system. Additional equipment includes ovens, analytical balances, classical chemistry apparatuses, UV spectrophotometers, flash point apparatuses, and ion-selective electrode equipment.

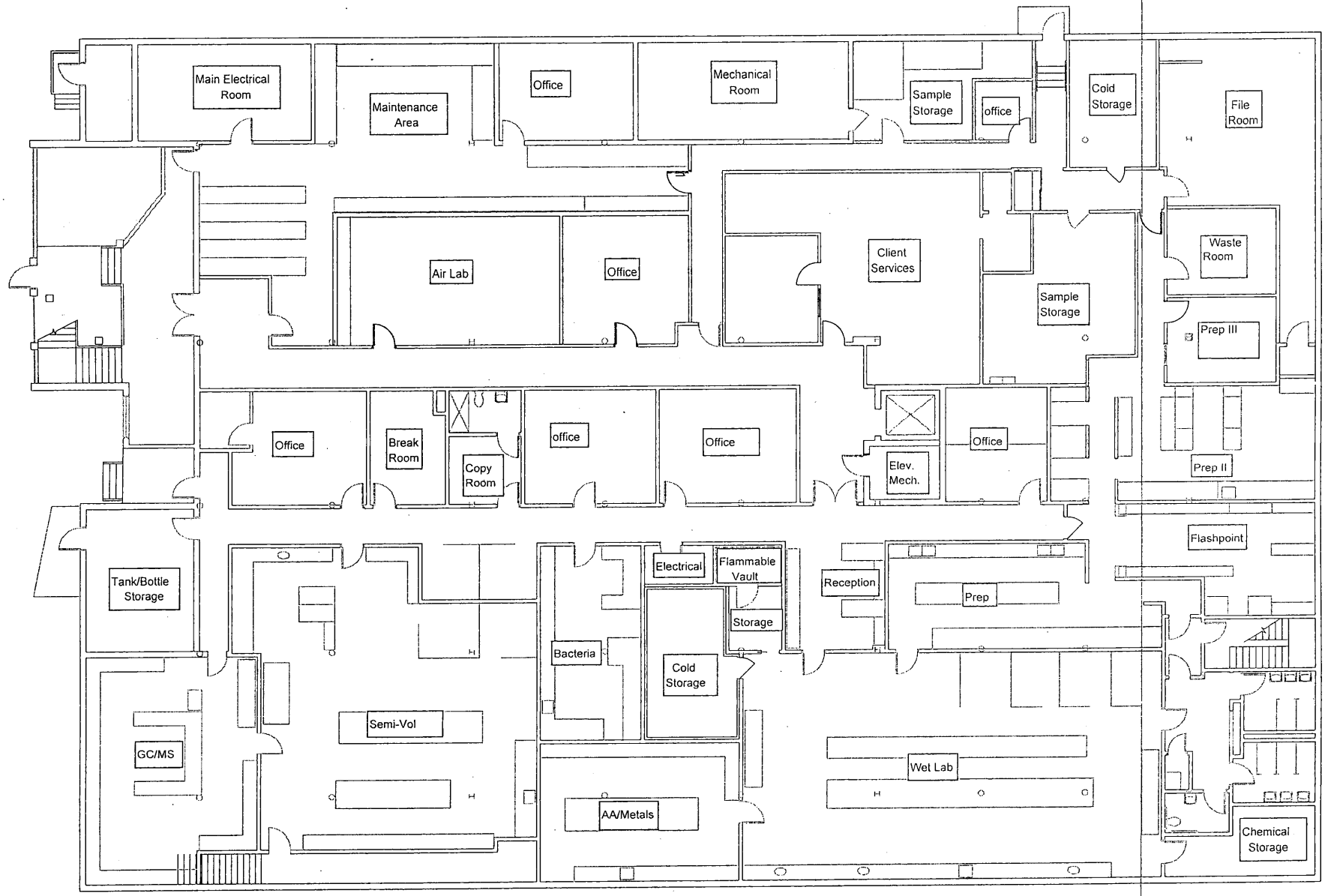
- **Bacteriological Analysis Laboratory**

  The Bacteriological Analysis Laboratory is over 250 square feet in size. There is approximately 30 linear feet of open laboratory bench space for use in analysis. Equipment includes four large BOD incubators, an autoclave, a long-wave ultraviolet visualization device, water baths, dry incubators and numerous pieces of miscellaneous equipment for the preparation and storage of sterile media and cultures.

- **Organic Analysis Laboratories**

  The Organic Analysis Laboratories have over 2200 square feet of floor space dedicated to organic laboratory instrumentation plus a repair area of over 100 square feet used to make instrument repairs and store spare parts. All volatile analyses are segregated into one of the laboratories to prevent the possibility of solvent cross-contamination. This area also has positive airflow to prevent the influx of vapors. The general features of the organic laboratories include several small hoods for use when spiking standard materials into sample extracts and for the preparation of standards; refrigerators and freezers for the storage of samples and samples extracts, and for the storage of standard materials and solutions; ovens; a Hewlett Packard HPLC Chromatograph; Perkin Elmer and Agilent Gas Chromatographs (GCs) and accessory detectors, autosamplers, headspace samplers and data systems; Hewlett-Packard (Agilent) Gas Chromatograph/Mass Spectrometer (GC/MS) instruments.

Figure 1
Facility Floor Plan



Phoenix Environmental Floor Plan

6.3 Instrument Maintenance

In an effort to reduce unexpected instrument failure, ensure reliable and accurate data generation, and control the costs associated with, non-routine maintenance and down time the laboratory has implemented a preventative maintenance system. Routine preventative maintenance is performed as suggested by the manufacturer. When it is found that maintenance is required more often or that additional maintenance is required these procedures are added to the Standard Operating Procedure.

Each instrument has a maintenance logbook that describes the routine inspection, cleaning, maintenance, testing, calibration, and/or standardization. Written records are maintained to document all inspection, preventative and non- routine maintenance, test, calibration and/or standardization procedures. The records include date, description of activity and actual findings, the name of the person performing the operation and a statement as to whether the maintenance operations were routine or unscheduled. Non-routine repairs performed as a result of equipment malfunction are documented in the instrument logbook to show the nature of the problem, when the problem was discovered and remedial actions taken. Repairs made by the manufacturers instrument repair technicians are also documented and the service reports filed in the instrument logbook.

The Quality Assurance Office monitors equipment maintenance and calibration through inspections of instruments and logbooks. Deviations are communicated to the Section Director via memoranda or report. Service contracts have been obtained for most instrumentation identified as vital by management i.e., GC, GC/MS, AA (furnace) and ICP instrumentation. For other equipment, factory service can be arranged, on a time and materials basis, usually within 24 hours.

Preventative/Routine Maintenance Schedule for Organics:

Gas Chromatography

- ECD detector baked out at 450 degrees C for 12 hours quarterly or when mV reading exceeds 15.
- NPD detector bead changed quarterly or as needed.
- FID detector assembly cleaned with solvent quarterly or as needed.
- Injector septum replaced weekly or per every 250 injections.

- Injector liners inspected weekly and replaced as needed.

Gas Chromatography/Mass Spectometry- Volatiles
- Clean source quarterly or if method performance criteria fails.
- Change Tekmar 3000 concentrator trap monthly or as needed.
- Change Tekmar 3000 concentrator MCS loop every 2 months for drinking water, otherwise change quarterly or as needed.
- Replace soil purge needle on autosampler every 2 months.
- Bake Tekmar 3000 concentrator trap daily for 30 minutes.

Semivolatiles
- Clean source monthly or if method performance criteria fails.
- Change liner and clip column daily.

Preventative/Routine Maintenance for Inorganics:

Atomic Absorption Spectrophotometer
- Change graphite tubes weekly or as needed.
- Clean graphite tubes daily with methanol.
- Trim capillary tubing daily or as needed.
- Replace contact rings monthly or as needed.
- Replace lamps as needed.

ICP
- Check tubing daily and replace every 2 days.
- Clean torch daily.
- Check and clean nebulizer daily.
- Clean chilling water quarterly.
- Replace air filters quarterly or as needed.
- Preventative Maintenance yearly by manufacturer.

General Preventative/Routine Maintenance:
- Balances- Calibrate daily with class "s" weights. Reference weights are certified annually.
- Refrigerators/Freezers- Temperatures taken daily with calibrated thermometers. Thermometers are calibrated annually. Reference thermometers are certified every three years.

6.4 Laboratory Materials Procurement and Tracking

The LIMS system stores ordered reagents, standards, and supplies for tracking purposes. The standard certificate of Analysis (COA) are scanned and linked to each standard used. Dilutions of standards are also traced in the LIMS system from ordered "stock". The expiration dates of the stock as well as the prepared standard are tracked. The amount and identity of each stock as well as the final volume and concentration of the prepared standard is recorded electronically.

Each chemical purchased for laboratory use is ordered by specifying the grade required for the intended use. Persons who place the orders are not permitted to make any substitutions without authorization from the Section Director. This restriction is intended to avoid inadvertent purchase of materials of substandard quality. The grades typically used include the following:

Technical	used for cleaning or non-quantitative purposes.
Purified	used for some qualitative analytical work where purity is not critical and specific contamination is noted to be absent.
ACS Reagent	used for analytical work.
Spectrograde	used in IR, AA, and UV applications.
Pesticide Grade	used for pesticide determinations and other GC applications
Primary Standard	used for preparation of standards, calibration, quality control, and standardization.

Standards for organic compounds are typically obtained as concentrated solutions from a commercial source. Metals standards are obtained from commercial sources as 1,000 or 10,000 ppm certified solutions. Standard materials for inorganic parameters are typically primary standard grade, when available, or analytical grade. Independent quality control standards are obtained from commercial sources. Quality Control standards must not be from the same lot as materials used for calibration. Typically, different commercial sources are used.

All reagents, acids, solvents, standards and other chemicals are logged into the LIMS system upon receipt, making them available for tracking.

All stock, prepared standards, reagents, and prepared reagents have the vendor expiration date, the date opened, and the laboratory expiration (which is the earliest of either the vendor expiration or the expiration from date opened) defined in the LIMS setup for each department. All stock, prepared standards, reagents, and prepared reagents flag "red" when they are within 2 weeks of their expiration date. The lab analyst of record will then mark them "inactive" before the expiration date so they can be disposed of.

Solvents are stored in a large flammable storage locker in accordance with laboratory safety requirements. Individual bottles of solvents are kept in the flammable storage cabinets under the laboratory hoods. Acids are kept in a safety cabinet for corrosives and in corrosives storage cabinets under fume hoods. Dry chemicals are stored on designated shelves at ambient temperature. Organic compound standards are stored in several freezers or areas within refrigerators, which are dedicated to standards only. Standards for inorganic compound analysis are stored within a dedicated standard refrigerator and those for metals analysis are stored at room temperature in cabinets.

To control quality of purchased chemicals, the oldest supply is used before a new bottle is opened ("first in, first out"). Analysts are responsible for checking appearance of the chemical prior to use to assure that the physical state of the material is correct. Purity and stability of reagents are monitored by performing blank determinations and Quality Control samples along with analytical batches. Additionally, each manufacturer's lot of solvent is checked for potential contaminants by pre-screening the solvent through the appropriate method.

7.0 Data Generation

7.1 Standard Operating Procedures

Standard Operating Procedures (SOPs) are utilized by Phoenix Environmental Laboratories, Incorporated to define exact routines to be followed in each section. There are SOP documents covering all aspects of the laboratory operation, from sample receipt and analytical methodology through data review and archiving. The entire SOP Manual is available for review during client visits.

Each SOP document is individually reviewed and approved. A Document Control System has been designed for SOP documentation and a historical file is maintained. SOPs are identified by a SOP numbering and revision identification system administered by Quality Assurance. Once approved by signature, an effective date is assigned to the document. Distribution of new SOP documents and retirement of old documents is the responsibility of the Quality Assurance Office. Obsolete documents are maintained in a historical file where they are marked obsolete and the date of replacement noted. Standard Operating Procedure documents are reviewed at least annually to determine if updating is required.

SOP documents may be initiated by any staff member. The proposed document is submitted to the Quality Assurance Office, which, after review, circulates the draft document to lab management for comments. The draft document and management comments are returned to the originator for resolution. The revised document is then circulated by the Quality Assurance Office for approval signatures. Each SOP must be signed by the originator, as well as the Section Supervisor, QA Officer or Director.

All SOP documents are scanned and available to all employees in electronic format in the LIMS system. Original SOP Manuals are controlled by the QA department.

The Quality Assurance Office has a critical role in the establishment and maintenance of the SOP documentation program. The Quality Assurance Office prepares or assists others in the preparation of many SOP documents. The Quality Assurance Office is responsible for the circulation and review of draft SOPs, for maintenance of the SOP document control system, including the historical file, and electronic versions in the LIMS.

All laboratory employees are responsible for reading, understanding and following SOPs particular to their job function. To document this, employees are required to sign a SOP Review Sheet, which states that they have read and understand SOPs particular to their job function. These forms are kept in the original SOP manuals, maintained by the Quality Assurance Office.

Appendix D of this document contains the Table of Contents of the SOP manual.

7.2 Sample Chain-of-Custody

All incoming samples are delivered to the Sample Control office for inspection, log-in, and storage. Immediately upon receipt, the sample set is unpacked and checked versus the chain-of-custody sheet. It is the responsibility of the Sample Coordinator to sign for laboratory custody upon receipt.

The Sample Control inspection of the samples include the following checks:

- Custody seal status
- Sample container integrity
- Holding temperature at time of receipt
- Type of container (plastic or glass)
- Addition of preservation to sample if chemical preservation is required
- Volume of sample
- Sample identity and collection information

The Sample Acceptance Policy details the procedures for inspection of samples upon receipt and the EPA requirements concerning sample preservation and holding times. The client is notified if the samples do not meet the guidelines for sample identification, holding time or preservation. Procedures utilized in the logging of samples are detailed in a separate SOP document.

The result of the incoming sample inspection is noted on the Chain of Custody Form. The temperature of samples and coolant information is also noted on the chain of custody. The pH of preserved containers is recorded in the electronic Sample Preservation record.

Samples are assigned a unique, sequential number during the logging process. Individual sample labels are generated for each sample that reflects the Phoenix sample number. Reporting requirements and criteria should also be recorded on the Chain of Custody by the client and they are logged in accordingly.

The samples are stored in secure sample storage areas by Sample Control. Distribution of samples within the laboratory is monitored by the LIMS. Each analyst logs start time and end time with every use of the sample or sample extract.

Commercial samples are kept for at least 30 days from the time the samples are received. After 30 days the samples are disposed of unless otherwise specified by the client. Extracts of samples submitted for organic analysis will be retained for a period of 30 to 90 days after data submission.

7.3 Sample Management

Phoenix Environmental Laboratories, Inc. uses five techniques as part of its complete sample management program:

- Computerized sample login including printing of Sample Receipts for verification of analyses requested.
- Database printouts of assignments and work backlogs.
- Centralized LIMS input or data transfer of all analytical results and comments regarding any problems encountered during analysis.
- Validation and generation of the final analytical report for transmission to the client in either hard copy or electronic form.
- Archiving of all reports and raw analytical results on a hard disk for storage and potential future retrieval if required.

In section 7.2 of the Quality Manual, the Chain of Custody (COC) Form was discussed as the location where results of the incoming sample inspection are recorded. After this step, a copy of the chain of custody and any field paperwork or client paperwork, which arrived with samples, is sent to the Client Services group. Client Services compares the information submitted with that in its own files to assure that the sample set agrees with work arranged via previous communication. Client Services then records the test codes required for each sample if not previously established. Special instructions to the lab regarding report due date, sample preparation, QC requirements, criteria and reporting requirements or special handling required are also recorded by Client Services.

Sample Control enters the client information, sample identification and test codes into the database. Each set of samples, which are received from a client at the same time, is assigned to a *Login Group.*

After log-in on the Phoenix Laboratory Information Management System (LIMS), *Sample Receipt Forms* are generated from the database. These receipts are checked against the original chain of custody for accuracy.

Departmental, work assignment sheets (backlog reports) are generated by the Section Managers. The LIMS system has been programmed to create a separate backlog for each department or analysis. The backlog contains essential information such as Sample Identification, test required, collection date to comply with EPA holding times, and date results are due to the client.

Each supervisor is responsible for assigning analytical batches for processing by analysis. The analyst then creates a batch, or a listing of samples to be analyzed. The Phoenix LIMS generates batches by holding time and due date, and includes the quality control samples and any special sample instructions. As each test is completed, the majority by data transfer, the LIMS database is updated to close out the test.

The data is then validated and cross-checked for accuracy and conformance with parameter limits, history, and inter-parameter correlations. The final analytical report is then generated for review by the project manager, along with the quality assurance department or Laboratory Directors before transmittal to the client. Preliminary results (before final data validation and review) may be sent to the client as a Sample Progress Report. This preliminary report clearly indicates that the data is of a preliminary nature and subject to review and revision.

After the final data report is submitted to the client in electronic or hard copy form, the final report and raw analytical data is archived on a hard disk for storage and potential future review. See Section 8.3 of this Manual for storage information.

7.4 Additional Procedural and Calibration Requirements to Achieve Quality Assurance Objectives

7.4.1 Organics

Sample Preparation

A minimum of three surrogate standards is added to each organic sample requiring GC/MS analysis for volatile and acid and base neutral extractables. For pesticide analysis, two surrogates are added and for herbicide analysis, one surrogate is added. These surrogate compounds are quantitatively analyzed in the GC/MS or GC phase. Historical records, in the form of laboratory control charts, are maintained on the percent recovery of surrogate compounds for each sample. These records form the statistical basis upon which preparation techniques are monitored. Surrogate recoveries must meet acceptance criteria before the analytical data will be released. All sample preparation methods with analysis dates are reported to the client on the final report. In some instances, the sample matrix may produce interferences that adversely affect recoveries. These interferences must be confirmed by a repreparation and reanalysis of the samples. Affected data are qualified in the report.

A method blank per matrix is prepared at a frequency of at least one for every twenty samples processed for each analysis requested or daily, whichever is greater. The purpose of the method blank is to ensure that contaminants are not introduced by the glassware, reagents, personnel, and sample preparation or sample analysis environment.

Standards

Calibration standards are traceable to the National Institute of Standards and Technology (NIST), formerly the National Bureau of Standards (NBS). Commercial sources of standards and reagents are checked for purity against a second source standard.

All standards prepared for use throughout the organic laboratory are assigned a unique identification number. The standard number is entered into the LIMS system with all information regarding the preparation of that standard, i.e., date, technician, name and lot number of each compound and amount used, final volume, expiration date and solvent used. All stock standard containers are labeled with the standard's identification number and name, lot number, code, manufacturer, date prepared and expiration date.

The instrument response obtained for each compound in a newly prepared standard is compared to the response obtained from the previous standard. The two standards must agree within $\pm$ 15% for all but a few compounds recognized as being chromatographably atypical or the new standard may not be used until the discrepancy has been resolved.

Gas Chromatography/Mass Spectrometer (GC/MS)

The Gas Chromatograph/Mass Spectrometer analyses are an integral part of the analytical services provided by Phoenix Environmental Laboratories Incorporated. The analyses involve very sophisticated instrumentation, which is operated by a highly trained staff. To assure that the results are of the highest quality, a rigorous program of calibration and quality assurance has been established.

Prior to the utilization of the instrumentation, the instrument performance is adjusted to assure that all manufacturer and method's performance criteria are met. The instrument's performance is monitored, recorded and when appropriate charted in control charts. The instrument is continually monitored and is adjusted on an as-needed basis (specified in the Standard Operating Procedures).

Tuning

At a frequency which complies with the analytical method, the mass spectrometer is adjusted to meet the method defined tune criteria, using FC-43. Bromofluorobenzene (BFB) or Decafluorotriphenylphosphine (DFTPP) is then used to confirm that the instrument meets these criteria. The BFB ion abundance criteria are outlined within the particular methods and must be satisfied for all volatile organic analyses. The DFTPP ion abundance criteria are outlined within the applicable methods and must be satisfied for all semi-volatile organic analyses. After the tuning criteria are confirmed, the instrument is calibrated for the analyses of interest.

Calibration

The analytical procedure followed for analyses of both volatile and semivolatile organic compounds involves an initial calibration of the instrument. The SOP of each analytical method details the criteria of the calibration curve. This calibration is performed using multiple concentrations of standards. The validity of the calibration is then confirmed using an NIST traceable standard mix containing known concentrations of each analyte. On a daily basis, the instrument calibration is confirmed to be unchanged by analysis of a single standard. The SOP of each analytical method details the criteria of the calibration curve.

Blanks

After calibration, a method blank is analyzed to demonstrate that the system is free of any of the analytes of interest. The method blank consists of organic free water for volatile analyses and an extraction blank for semi-volatile analyses. After demonstration that the system is free of contamination, sample analyses are begun. Maximum allowable levels of contamination are up to the method detection limit for most organic compounds and up to 10X the Contract Required Detection Limit (CRDL) for common laboratory contaminants as defined in the EPA Statement of Work.

Gas Chromatography (GC)

Pesticide, Herbicide and Polychlorinated Biphenyl (PCB) analyses are performed using a gas chromatograph equipped with the appropriate detectors. These analyses often are performed on complex matrices that require an experienced staff for the interpretation of the results. The analysts also must determine the clean-up requirements needed for each individual sample.

Prior to all analyses, the elution time and elution order for each analyte of interest is determined. They are determined by analyses of several standards over a seventy-two (72) hour period. These analyses also define the retention time window. This window is calculated by multiplying the standard deviation of the retention times by a factor of three (3).

Calibration

The instrument is calibrated by analysis of a standard mixture that contains the analytes of interest. The number of standards and their concentration are method specific, but all assure an accurate determination of the concentration of an analyte in the sample. The instrument's sensitivity is adjusted so that all standards are integratable and are also within the instruments linear response range. On a daily basis, and after every twenty samples, the instrument calibration is confirmed to be unchanged by analysis of a single standard. The SOP of each analytical method details the criteria of the calibration curve and the continuing calibration check samples.

Blanks

After calibration, a method blank is analyzed to demonstrate that the system is free of any of the analytes of interest. The method blank consists of an extraction blank for pesticide, herbicide and PCB analyses. After demonstration that the system is free of contamination, sample analyses are begun.

7.4.2 Metals

The analyses performed on the ICP, GFAA and AAS instrumentation are an extremely important part of the analytical services provided by Phoenix Environmental Laboratories, Incorporated. The analyses involve very sophisticated instrumentation, which is operated by a highly trained staff. To assure that the results from this phase of the operation are of the highest quality, a rigorous program of calibration and quality assurance has been established.

Prior to the utilization of the instrumentation, the instrument performance is adjusted to assure that all manufacturer's and accrediting body's performance criteria are met. The instrument's performance is monitored, recorded and when appropriate charted in control charts (specified in the Standard Operating Procedures).

Standards

Calibration standards are traceable to the National Institute of Standards and Technology. Commercial sources of standards and reagents are checked for purity against a second source standard. All standards prepared for use throughout the laboratory are assigned a unique identification number. The standard number is entered in the LIMS system with all information regarding the preparation of that standard, i.e., date, technician, name and lot number of each compound and amount used, final volume, and concentration of acid in the diluent used. All stock standard containers are labeled with the standard's identification number and name, date prepared and expiration date.

Calibration of GFAA, AAS and ICP Systems

Instruments are calibrated each time an analytical run of less than twelve hours is set up. Calibration standards are prepared by diluting the stock metal solutions at the time of analysis. Source identification and analysis date are recorded on the analysts run log cover sheet, which is attached to the analytical data and stored electronically.

The calibration standards are be prepared using the same type of acid or combination of acids as in the sample extracts. Calibration standards are prepared fresh daily for cold vapor and furnace methods. Calibration standards are prepared at least weekly for ICP methods. The calibration curve consists of a blank and at least three calibration standards in the appropriate range

Quality Control Requirements

The quality control program within the metals department consists of analysis and evaluation of various samples. Each QC sample analyzed reflects the conditions of analysis of all associated analytical samples. The duration of analysis, rinses and other related operations that may affect the QC measured result may not be applied to the QC to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CV analysis and the blank immediately following it as well as the difference in time between the CV and the analytical sample immediately preceding it may not exceed the lowest difference in time between any two consecutive analytical samples associated with the CV. The requirements of each are detailed in the standard operating procedure (SOP).

Calibration Verification Standard

Immediately after calibration and every ten samples, a standard at the midpoint range of the calibration is analyzed and evaluated for each analyte. When measurements exceed the control limits criteria, the analysis for that analyte is terminated. Samples are accepted only when bracketed by acceptable CV standards.

Calibration Blank Standard

After each CV standard, a standard blank is analyzed and evaluated. The purpose of the calibration blank is to determine the effect of instrument drift at the level near the reporting limit.

Laboratory Control Standard (LCS)

After calibration, a LCS standard is analyzed and evaluated for each analyte. The LCS is a certified solution provided by a source independent from the calibration standards. Sample analytes are accepted only when the LCS meets the acceptance criteria.

Fortified Blank/Blank Spike/Preparation LCS

Aqueous and solid Laboratory Control Samples (LCS) are analyzed for each analyte using the same sample preparations and analytical methods as the samples being analyzed. The aqueous LCS solution is obtained by spiking a preparation blank with a spiking solution prepared by the metals department from certified materials. One LCS is prepared and analyzed for every batch samples digested. The control limits are defined by internal control charts or by method SOP. If any analyte exceeds criteria, the analysis will be terminated, the problem corrected and the samples associated with that LCS re-digested and re-analyzed.

Preparation Blank

At least one matrix matched preparation blank to be processed through each sample preparation and analysis procedure must be prepared and analyzed with every sample batch. This blank is reported for each sample batch, if required, and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner,

A If the absolute value of the concentration of the blank is less than or equal to the method requirements (see individual SOP), no contamination of the sample results is suspected.

B If any analyte concentration in the blank is above the method requirements, the lowest concentration of that analyte in the associated samples must be 10x the blank concentration. Otherwise, all samples associated with that blank must be redigested and reanalyzed for that analyte. The sample concentration is not to be corrected for the blank value.

Interference Check Sample

An Interference Check Sample (ICSAB) is analyzed daily to verify the accuracy of the inter-element corrections. The control limits for this sample are 80-120% of true value.

Spike Sample Analysis

The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. The spike is added before the digestion steps. At least one spike sample analysis is performed on each group of samples of a similar matrix type (i.e., water, soil) or for each sample batch.

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations are performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks should not be used for spiked sample analysis. The same spiking solution is used for the matrix spike as the blank spike. If two analytical methods are used to obtain the reported values for the same element within a Sample Batch (i.e., ICP, GFAA), spike samples must be run by each method used.

The spike recovery is reported in the Quality Control Sample Section of the LIMS. This sample can be included in the client report if required. In-house limits are produced from control charts.

For ASP-like analyses, if the spike recovery is not at or within the limits of 75-125%, the data of all samples received associated with that spike sample and determined by the same analytical method shall be noted in the report. An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

Duplicate Sample Analysis

One duplicate sample is analyzed from each group of samples of a similar matrix type (i.e., water, soil) or for each sample batch.

Samples identified as field blanks should not be used for duplicate sample analysis. If two analytical methods are used to obtain the reported value for the same element for a Sample Batch (i.e., ICP, GFAA), duplicate samples must be run by each method used.

The relative percent differences (RPD) for each component are calculated as follows:

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

Where, RPD = Relative Percent Difference
S = First Sample Value (original)
D = Second Sample Value (duplicate)

The RPD is reported in the Quality Control Sample Section of the LIMS. This sample can be included in the client report if required.

Instrument Detection Limit Determination (for ASP-like analyses)

The instrument detection limits in *ug*/L shall be determined for each instrument used at a frequency of at least annually, and must meet the method requirements.

The Instrument Detection Limits (in *ug*/L) shall be determined by multiplying by 3 the average of the standard deviations obtained on three non-consecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x-5x the instrument manufacturer's suggested IDL, with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDL's must be determined and reported for each wavelength used in the analysis of the samples.

The most recently determined IDL for an instrument are used as the IDL for that instrument. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined and the results submitted for use as the established IDL for that instrument. Instrument detection limits are retained and are available for inspection.

Demonstration of Capability/Performance

Method Detection limits are determined for each instrument for each analyte at least annually following the procedure described in 40 CFR 136 Appendix B.

Linearity of calibration is determined by evaluation of the calibration curve. The correlation coefficient must be 0.9975 or greater. The highest standard must agree within 5% of the true value.

Quality control samples from a source different than the calibration standards are used to verify the calibration standards.

Accuracy and Precision Studies are performed at least yearly where required. Four standards at or near the mid-point of the working range are analyzed and evaluated.

7.4.3 Classical Chemistry

The analyses performed by the classical chemistry department are an extremely important part of the analytical services provided by Phoenix Environmental Laboratories, Incorporated. The analyses, which are performed by a highly trained staff, are the most varied in the laboratory. To assure that the results from this phase of the operation are of the highest quality, a rigorous program of calibration and quality assurance has been established

Standards

Calibration standards are traceable to the National Institute of Standards and Technology. Commercial sources of standards and reagents are checked for purity against a second source standard. All standards prepared are assigned a unique identification number. The standard number is entered into the LIMS system or a bound Standards Notebook with all information regarding the preparation of that standard, i.e., date, technician, name of each compound and amount used, final volume, and expiration date. All stock standard containers are labeled with the standard's identification number, lot number and name, date prepared and expiration date.

Demonstration of Capability/Performance

Method Detection limits are determined for each instrument for each analyte at least annually following the procedure described in 40 CFR 136 Appendix B.

Accuracy and Precision Studies are performed at least yearly where required. Four standards at or near the mid-point of the working range are analyzed and evaluated.

Laboratory Control Standard (LCS)

A LCS is analyzed and evaluated for each batch of samples. The LCS is obtained from certified source independent from the calibration standards. The acceptance criteria are determined by in house control charts. The LCS is reported in the LIMS and is available for the client report if required.

Preparation Blank

A preparation blank, consisting of deionized distilled water processed through each sample preparation and analysis procedure is prepared and analyzed with every sample batch. This blank is reported for each sample batch, if required, and used in all analyses to ascertain whether sample concentrations reflect contamination.

Spike Sample Analysis

The spike sample analysis is designed to provide information about the effect of the sample matrix on the distillation/digestion and measurement methodology. The spike is added before the digestion or distillation steps. At least one spike sample analysis is performed on each group of samples of a similar matrix type (i.e., water, soil) or for each sample batch.

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations must be performed using the results of the sample designated as the "original sample". The average of the duplicate results cannot be used for the purpose of determining percent recovery.

The spike recovery is reported in the Quality Control Sample Section of the LIMS. This sample can be included in the client report.

Duplicate Sample Analysis

One duplicate sample is analyzed from each group of samples of a similar matrix type (i.e., water, soil) or for each sample batch.

Samples identified as field blanks should not be used for duplicate sample analysis.

The relative percent differences (RPD) for each component are calculated as follows:

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

Where, RPD = Relative Percent Difference
S = First Sample Value (original)
D = Second Sample Value (duplicate)

The RPD is reported in the Quality Control Sample Section of the LIMS. This sample can be included in the client report if required.

7.4.4 Bacteria Department

The bacteria department analyzes samples for the presence of coliform (total, fecal and e.coli), Fecal Streptococcus, and Enterococcus. In addition, a Heterotrophic plate count provides an enumeration of all forms of bacteria. These analyses are an important part of the analytical services provided by Phoenix Environmental Laboratories Incorporated. These analyses are performed by a highly trained staff utilizing a rigorous quality assurance program.

Preparation of Culture Media

The culture media used at Phoenix are either prepared from dehydrated material or purchased ready to use. Preparation of media is recorded in the electronic Media Prep Log, and media are given a batch number for each time it is prepared. Prepared media is recorded in the Bacteria Chemicals Receipt Logbook and in the Media Preparation Logbook.

Negative and Positive Control

Coliform Analysis
Coliform bacteria are Gram negative, non-spore-forming rod-shaped bacteria that ferment glucose at 35°C. Each batch and lot of media is then tested to verify amenability to Coliform growth and inability to grow other bacteria. The Gram positive bacterium (P.aeroginosa) is used as the negative control, as it will not grow on coliform media. Two species of coliform bacteria are used to verify amenability (positive control) to the media: K.pneumonea, and E.coli.

Fecal Coliform Analysis
Fecal coliforms are bacteria that fulfil the definition of a Coliform, yet are able to sustain growth at elevated temperatures (thermo-tolerant coliforms). E.coli are fecal coliforms, and are used as the positive control test organism for the culture media. Klebsiella variicola, not considered a fecal coliform because it is absent in the lower digestive tract of mammals, but a thermo-tolerant strain is also used as a positive control. Enterobacter aerogenes is used as the negative control for the culture media.

E. Coli Analysis
E.coli is a fecal coliform that is determined biochemically, rather than by increasing the temperature. K.pneumonea is used as the negative control, and E.coli is used as the positive control when testing the culture media.

Heterotrophic Plate Count Analysis
Heterotrophic Plate Counts (Standard Plate Counts) are the enumeration of all forms of bacteria. Unlike the culture medium for Coliforms (which has a Gram Positive inhibitor), the Standard Plate Count culture medium will allow growth of many kinds of organisms. S.aureus is used as a positive control to verify the lack of inhibition present in the media.

Enterococcus Analysis
Enterococci are a sub-group of fecal streptococci. The most common bacterium in this group is Enterococcus Faecalis, which is used as a positive control. E.coli and Streptococcus bovis are used as the negative controls.

Blanks

Aliquots of sterile buffered water are run at the beginning and end of each batch of membrane filtration. They are incubated as samples, and are checked for growth. The blanks demonstrate the sterility of the glassware used throughout the filtration process. The initial blank demonstrates that the glassware was clean when the batch was begun, and the final blank demonstrates that the glassware was clean when the batch was completed. Batches of greater than 10 samples require blanks that are performed mid-way. When the blanks come back with no growth, it can be assumed that the cleaning between sample filtration was sufficient to remove residue from the previous samples. If any of the blanks come back with growth, this assumption does not hold, and all of the results must be thrown out.
Blanks for methods that do not involve membrane filtration (like multiple tube fermentation, and sample plating) require only one blank, done at the end of the batch of samples. For these methods, it is necessary to demonstrate the sterility of the work area at the time of the testing. If the final blank is shown to have no growth, then it can be assumed that the work area was sterile at the end of the batch and therefore was sterile throughout the run. If the final blank comes back with growth, this assumption does not hold, and the results must be thrown out.

General Equipment

Incubators and waterbaths are monitored to ensure they maintain constant temperatures within the acceptable guidelines. The sterilization apparatus is tested routinely, with a heat resistant strain of spore, to ensure proper sterilization.

7.5 Determination of Detection and Quantitation Limits

Two types of detection limits are routinely determined at Phoenix Environmental Laboratories, Incorporated; the Method Detection Limit study and the Limit of Detection and Quantitation verification study. A Method Detection Limit (MDL) is the minimum concentration that can be measured with 99 percent confidence that the analyte is greater than zero. The MDL's are determined from analysis of spiked blank waters and soils. The Limit of Detection (LOD) is the laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect. The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target analyte that can be reliably quantitated.

Method Detection Limits are measured for all tests employed at Phoenix Environmental Laboratories, Incorporated. The procedure is defined in 40 CFR Part 136, Appendix B (Federal Register, revision 3, October 2020). The procedure is outlined below:

a) An estimate of the detection limit is made.

b) A minimum of seven replicates of blank water or soil are spiked at a level 2 to 5 times the estimated detection limit or three times the standard deviation of a set of method blanks.

c) The spiked samples are processed through every step of the analytical method, and for ongoing MDL studies, two are analyzed each quarter.

d) The standard deviation for the seven samples is multiplied by 3.143 (students t value at 99% confidence at N-l degrees of freedom) to obtain the MDL.

e) A minimum of seven method blanks must be calculated annually using the same procedure.

The Practical Quantitation Limit (PQL) is the lowest calibration standard calculated using sample preparation conditions and the percent solids. The MDL study verifies the capability of the laboratory to detect the compounds at the practical quantitation limit.

7.6 Determination of inter-element correction factors

Inter-element corrections are applied by the manufacturer's software and are established when the instrument method is setup. On a daily basis, the background points are assessed for correctness.

Phoenix Environmental Laboratories, Incorporated

Quality Manual

Appendix A

Resumes of Key Personnel

THE PEOPLE OF PHOENIX ENVIRONMENTAL LABORATORIES, INC.
Technical Staff Education and Experience

Phyllis Shiller
Laboratory Director

Responsibilities:	Technical Director of Laboratory Operations and Services. Manages laboratory personnel and staffing. Responsible for laboratory scheduling and maintenance of high sample throughput. Provides client interface and management of special projects, technical issues and regulatory matters. Works with QA/QC Manager to ensure all aspects of corporate quality control program are strictly adhered to.
Education:	University of Rhode Island, B.S. Chemistry, 1986
Experience:	Thirty-five years of environmental laboratory experience, including positions as QA/QC Director, Inorganic, ICP/GFAA Specialist, Inorganic Manager of a large (CLP) laboratory, Operations Manager, and Laboratory Director.

Bobbi Aloisa
Vice President

Responsibilities:	Management of Client Services Operation. Provides client interface with laboratory. Responsible for scheduling report deadlines with the client. Responsible for the generation of reports including progress reports, final reports, and electronic deliverables. Provides second level of review for all reports. Provides immediate review of incoming projects for completeness. Manages program that furthers the laboratory's ability to achieve consistent high levels of performance and quality.
Education:	Manchester Community Technical College, A.S. Science, 1994
Experience:	Twenty-eight years of environmental laboratory experience.

Greg Lawrence
Assistant Laboratory Director

Education:	University of Hartford, Hartford, CT, Masters Business Administration, 1988 Keene State College, Keene, NH, B.S. Chemistry, 1982
Experience:	Forty years of environmental laboratory experience, including the position of Laboratory Director since 1985. Background in Organic Instrumentation, AA Spectrometry and Quality Control.

Kathleen Cressia
QA/QC Officer
Microbiology Laboratory Director

Education:	Western Connecticut State University, Danbury, CT, B.A. Earth Science/Biology, 1985
Experience:	Thirty-five years of environmental laboratory experience, including positions as Laboratory Director, Laboratory Operations Manager, QA/QC Manager, Director of Microbiology, Inorganic Manager, and Wet Chemistry Section Leader for a CLP Laboratory.

Linnea Skoglund
Quality Specialist

Education:	Westfield State University, Westfield, MA, B.S. Biology, Chemistry minor, 2019
Experience:	Three years of environmental laboratory experience.

Peter LaBarre
Database Administrator

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Computer Science, 1990
Experience:	Fifteen years of Laboratory Information Systems support.

Maryam Taylor
Project Manager

Education:	Nizam College, India, B.S. Chemistry, 1978
Experience:	Twenty-seven years of experience in the environmental laboratory field including GC/MS analyst, Organics Department Manager and Project Manager.

Alejandro Paredes
Project Manager

Education:	Universidad Rafael Landivar, Guatemala, B.S. Marketing, 2004
Experience:	Thirteen years in the environmental laboratory field, including GC/MS analyst and Data Specialist.

Helen Geoghegan
Project Manager

Education:	University of New Haven, New Haven, CT, M. S. Environmental Science, 1995 University College Dublin, Dublin, Ireland, B.S. Chemistry, 1986
Experience:	Thirty-two years of environmental laboratory experience, including the position of Co-Laboratory Director. Background in Organic and Inorganic Analysis, Quality Control, Data Validation, Technical Review.

Keith Aloisa
Organics Department Team Leader

Education:	Quinnipiac University, Hamden, CT, B.S. Chemistry, 1993
Experience:	Twenty-seven years of experience in the environmental laboratory field including Organic manager and QA Specialist.

Raman Makol
Organics Department Team Leader

Education:	Guru Nanak Dev University, India, M.S. Chemistry, 1986 Guru Nanak Dev University, India, B.S., Chemistry, 1984
Experience:	Thirty-one years of analytical and environmental laboratory experience as an analyst and R&D Specialist.

Harry Mullin
GC/MS Lead Analyst

Education:	Providence College, Providence, RI, B.S. Biology, 1986
Experience:	Thirty-five years of experience in the environmental laboratory field including Organic Laboratory Manager.

Hongjie Li
GC/MS Analyst

Education:	University of Petroleum, Beijing, China, M.S. Applied Chemistry, 1989 University of Alberta, Edmonton Alberta, Canada, M.S. Environmental Engineering, 2000
Experience:	Eighteen years of experience in the environmental laboratory and R&D field.

Michael Hahn
GC/MS Analyst

Education:	University of Connecticut- Biological Sciences Embry-Riddle Aeronautical University- Avionics Engineering
Experience:	Thirty-one years of environmental laboratory experience.

Wes Bryon
GC Analyst

Education:	Holyoke Community College, Holyoke, MA, A.S. Environmental Science, 2000
Experience:	Twenty-one years of environmental laboratory experience.

Jeffery Bucko
GC Analyst

Education:	Eastern Connecticut State University, B.A. History, 1991
Experience:	Twenty-seven years of experience in the analytical laboratory field.

Adam Werner
GC/MS Analyst, GC Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Molecular & Cellular Biology, 2011
Experience:	Thirteen years of experience in the analytical laboratory field.

Saadia Chudary
GC Analyst

Education:	Central Connecticut State University, New Britain, CT, B.S. Biomolecular Science, 2013, M.S. Biomolecular Science, 2015
Experience:	Eight years of environmental laboratory experience.

Christina Nieves
GC/MS Analyst

Education:	University of New Haven, West Haven, CT, B.S. Forensic Science, 2012
Experience:	Eight years of environmental laboratory experience.

James Karabetsos
GC/MS Analyst, Sample Preparation Analyst

Education:	University of New Haven, West Haven, CT, B.S. Biology / Forensic Science, 2013
Experience:	Seven years of environmental laboratory experience.

Emily Kolominsky
ICP Analyst

Education:	Pharmaceutical College, Zhitomir, Ukraine, A.S. Pharmacology, 1978
Experience:	Forty years of environmental laboratory experience.

Cynthia Pearce
ICP Analyst

Education:	University of Florida, Gainesville, FL, B.S. Chemistry 1977
Experience:	Thirty-three years of environmental laboratory experience.

Mike Hornak
Metals Analyst

Education:	Quinnipiac University, Hamden, CT, B.S. Chemistry, 1989
Experience:	Thirty-two years of environmental laboratory experience.

Tina Hall
Metals Analyst, Sample Preparation Analyst

Education:	Hood College, Fredrick, MD, B.A. Biology 1995
Experience:	Twenty-four years of environmental laboratory experience.

Ian Enders
Metals Analyst

Education:	Central Connecticut State University, New Britain, CT B.S. Earth Science, 2021
Experience:	One year of environmental laboratory experience.

Christina Szachon
Metals Analyst

Education:	Western Connecticut State University, Danbury, CT B.S. Chemistry, 2021
Experience:	One year of environmental laboratory experience.

Rashmi Makol
Microbiology Team Leader

Education:	Kurukeshtra University, India, B.S. Chemistry
Experience:	Twenty-two years of environmental chemistry and microbiology lab experience.

Lauren Johnson
Microbiology Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Biology, 2018
Experience:	Three years of environmental laboratory experience.

Eric Geyer
Inorganic Team Leader

Education:	University of Connecticut, Storrs, CT, B.S. Natural Resources, 1997
Experience:	Twenty-four years of environmental laboratory experience.

Kandi Della Bella
Inorganic Analyst, Microbiology Analyst

Education:	Saint Joseph College, West Hartford, CT, B.S. Natural Science, 1996 M.S. Biology, 2007
Experience:	Fourteen years of environmental laboratory experience.

Greg Danielewski
Inorganic Analyst

Education:	Capital Community Tech College, Hartford, CT, A.S. Chemical Engineering Technology, 1993
Experience:	Twenty-eight years of environmental laboratory experience.

Matt Fijolek
Inorganic Analyst

Education:	University of New England, Maine, B.S. Marine Biology
Experience:	Sixteen years of environmental laboratory experience.

Jean Rawlings
Inorganic Analyst

Education:	Bucknell University, Lewisburg, PA, B.S. Biology 1995
Experience:	Eighteen years of environmental laboratory experience.

Brian Sheriden
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Biology/English, 2001
Experience:	Sixteen years of environmental laboratory experience.

Michael Tran
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Computer Science and Engineering, 2012
Experience:	Five years of environmental laboratory experience.

Mary LaVallee
Inorganic Analyst

Education:	Southern Connectcut State University, New Haven, CT, B.S. Chemistry, 2022 Naugatuck Community College, Waterbury, CT, A.S. Science, 2007
Experience:	Seven years of environmental laboratory experience.

Elizabeth Rideout
Inorganic Analyst

Education:	Stevenson University, Ownings Mills, MD, Central Connecticut State University, New Brittain, CT, Chemistry Major
Experience:	One year of environmental laboratory experience.

Meredith Weigert
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Biology, 2019
Experience:	One year of environmental laboratory experience.

John Mark Woodworth
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Animal Science, 2020
Experience:	One year of environmental laboratory experience.

Catherine Lundigan
Inorganic Analyst

Education:	Bay Path University, Longmeadow, MA, B.S. Biology, 2022.
Experience:	Less than one year of environmental laboratory experience.

Daniel Kinney
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Environmental Science, 2019
Experience:	One year of environmental laboratory experience.

Angelica Martinez
Inorganic Analyst

Education:	Bay Path University, Longmeadow, MA, B.S. Forensic Science, 2020
Experience:	One year of environmental laboratory experience.

Praveena Krishnaprasad
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Biology, 2021
Experience:	Less than one year of environmental laboratory experience.

Gabrielle Sweeney
Inorganic Analyst

Education:	McGill University, Montreal, Canada, B.S. Biology
Experience:	One year of environmental laboratory experience.

Dylan Tillman
Inorganic Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Biology, Expected 2022
Experience:	Less than one year of environmental laboratory experience.

Dina Montagna
Sample Preparation Day Supervisor

Education:	Springfield College, Springfield, MA, B.S. Biology/Chemistry, 1999
Experience:	Twenty-two years of environmental laboratory experience.

Tara Banning
Sample Preparation Evening Supervisor

Education:	University of Connecticut, B.S. Biology, 2007
Experience:	Thirteen years of environmental laboratory experience.

Ashraf Sheikh
GC/MS Sample Preparation Analyst

Education:	South Gujarat University – Surat, India, B.S. Chemistry, 1990
Experience:	Twenty-two years of environmental laboratory experience.

Mary Tran
GC/MS Sample Preparation Analyst

Education:	Central Connecticut State University, New Britain, CT, B.S. Biology, 2015
Experience:	Seven years of environmental laboratory experience.

Jose Paz Soldan
GC/MS Sample Preparation Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Molecular and Cellular Biology, 2021
Experience:	Less than one year of environmental laboratory experience.

Melissa Kemp
GC/MS Sample Preparation Analyst

Education:	University of Saint Joseph, West Hartford, CT, B.S. Chemistry, 2018
Experience:	Less than one year of environmental laboratory experience.

Adrian Jaworski
Air Sample Preparation Analyst, GC/MS Assistant

Education:	Manchester Community College, B.S. Chemistry Expected.
Experience:	Less than one year of environmental laboratory experience.

Matthew Richard
GC/MS Assistant, Sample Preparation Analyst

Education:	Central Connecticut State University, New Britain, CT, B.S. Biology and Psychology, 2014
Experience:	Six years of environmental laboratory experience.

Robert Looney
GC/MS Assistant, Sample Preparation Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, Environmental Earth Science major
Experience:	Three years of environmental laboratory experience.

Sarah Kelting
GC/MS Assistant, Air Lab Assistant

Education:	University of New England, Biddeford, ME, B.S. Environmental Science
Experience:	Three years of environmental laboratory experience.

Anvarhusen Sheikh
Sample Preparation Analyst

Education:	Polytechnic Institute, Valsad Gujarat India, A.S. Chemical Engineering, 1983
Experience:	Twenty-one years of environmental laboratory experience.

Lisa Luchini
Sample Preparation Analyst

Education:	Central Connecticut State University, M.S. Biomolecular Science, 2010; B.S. Biomolecular Science, 2005
Experience:	Seven years of environmental laboratory experience.

Amber Roldan
Sample Preparation Analyst

Education:	Smith College, Northampton, MA, B.A. Neuroscience, 2020
Experience:	Less than one year of environmental laboratory experience.

Dustin Harrison
Sample Preparation Analyst

Experience: Seventeen years of environmental laboratory experience.

Autum Burke
Sample Preparation Analyst

Education: Central Connecticut State University, New Brittain, CT,
M.S. Environmental Science, Ongoing,
B.S. Biology, 2019

Experience: One year of environmental laboratory experience.

Gregory Mercier
Sample Preparation Analyst

Education: Eastern Connecticut State University, Willimantic, CT,
B.S. Environmental Earth Science, 2013

Experience: Seven years of environmental laboratory experience.

Rowena Wagner
Sample Preparation Analyst

Education: Trace Computer College, Laguna College, West Negros
University, Philippines – Computer Programming Certificate

Experience: Four years of environmental laboratory experience.

Edgar Cortez
Sample Preparation Analyst

Experience: Fifteen years of environmental laboratory experience.

Kira Wayman
Sample Preparation Analyst

Education:	University of Southern Mississippi, Hattiesburg, MS
Experience:	Three years of environmental laboratory experience.

Christine Luckhoo
Sample Preparation Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Biology, 2021
Experience:	Less than one year of environmental laboratory experience.

Dylan O'Hagan
Sample Preparation Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Health Science, 2021
Experience:	Less than one year of environmental laboratory experience.

Tamara Rodriguez
Sample Preparation Analyst

Education:	University of Saint Joseph, West Hartford, CT, B.S. Biology, 2020
Experience:	One year of environmental laboratory experience.

Kailey LeBlanc
Sample Preparation Analyst

Education:	Rhode Island College, Providence, RI, B.S. Health Science
Experience:	Less than one year of environmental laboratory experience.

Michelle Lopes
Sample Preparation Analyst

Education:	Colby-Sawyer College, New London, NH, B.S. Environmental Science, 2019
Experience:	Two years of environmental laboratory experience.

Megan Ortiguerra-Rojas
Sample Preparation Analyst

Education:	University of North Texas, Denton, TX, B.S. Ecology
Experience:	Less than one year of environmental laboratory experience.

Javaun Murphy
Sample Preparation Analyst

Education:	Western New England University, Springfield, MA, B.S. Chemistry, 2022
Experience:	Less than one year of environmental laboratory experience.

Aleisha Price
Sample Preparation Analyst

Education:	University of Connecticut, Storrs, CT, B.S Chemistry Expected 2023
Experience:	Less than one year of environmental laboratory experience.

7.7 Table of Methods

Wet Chemistry	
Acidity	SM2310B
Alkalinity	SM2320B
Ammonia/TKN	EPA 350.1/351.1
BOD/cBOD	SM5210B
Bromide	EPA300.0 SW9056
Chloride	SM4500CL E EPA300.0 SW9056
Chlorine	SM4500CL G
Chlorine Demand	SM2350B
COD	SM5220 D
Color	SM2120 B
Conductivity	SM2510 B
Cyanide	EPA335.4 SM4500CN SW9010/9012
DO electrode	SM4500 O G
Flashpoint	SW1010
Fluoride	EPA300.0
Hardness by Calculation	EPA200.7
Hexavalent Chromium soil	SW3060A
Hexavalent Chromium water	SM3500Cr B
Surfactants (MBAS)	SM5540 C
Nitrate	SM353.2 EPA300.0 SW9056
Nitrite	EPA353.2 EPA300.0 SW9056
Odor	SM2150 B
Oil & Grease	EPA1664 / SW9071B

Table of Methods (cont.)

Paint Filter Liquid Test	SW9095
pH and Corrosivity	SM4500H B SW9040/SW9045
Phenols	EPA420.4/SW9066
Phosphorus	SM4500P E
Reactivity	SW7.3
Salinity	SM2520B
SPLP Extraction	SW1312
Solids, Dissolved	SM2540 C
Solids, Fixed & Volatile	SM2540E
Solids, Suspended	SM2540 D
Solids, Total	SM2540 B
Sulfate	SM4500D EPA300.0/SW9056
Sulfide, Total	SW9030A
Sulfite	EPA377.1
TCLP Extraction	SW1311
TKN block digestion	EPA.351.2
TOC soil (sm)	SW9060 / L.Kahn
TOC water (wm)	SM5310B
Turbidity (NTU)	SM2130 B
Bacteria	
E. coli MF	SM9222G
Enterococcus	Enterolert
Fecal coliform MF	SM9222D
Fecal Streptococcus MF	SM9230C
Heterotrophic Plate Count	SM9215B
Total coliform DW	SM9223B
Total coliform MF	SM9222B
Total coliform/E.Coli MPN	SM9223B
Fecal coliform MPN	Colilert18 MPN
Metals	
Mercury by Cold Vapor	EPA245.1 SW7470 SW7471

Metals (continued)	
Metals by GFAA	EPA 200.9, SM 3113 SW 7000 series
Metals by ICP	EPA 200.7/200.8 EPA 200.5 SW 6010/6020
Organic Instrumentation	
EDB, DBCP, 123TCP	EPA 504.1
Carbamates	EPA 531.2
Glyphosate	EPA 547
Diquat, Paraquat	EPA 549.2
Extractable Total Petroleum HC	CTETPH
PCB	EPA 608.3 SW 8082
PCB congeners	SW 8082
Pesticide (NPD)	EPA 525.3 SW 8141
Pesticide (ECD)	EPA 525.3 EPA 608.3 SW 8081
Haloacetic Acids	EPA 552.2
Herbicide	EPA 515.3 SW 8151
VOA by GC/MS	EPA 524.1 EPA 624.1 SW 8260
SVOA by GC/MS	EPA 525.3 EPA 625.1 SW 8270
1,4-Dioxane	EPA 522 SW 8270SIM
PCB in air	EPA TO-10
Volatiles in air	EPA TO-14 EPA TO-15 NJ LL TO-15

EPA: "Methods for chemical Analysis of Water and Wastes", EPA, Environmental Monitoring Systems Laboratory –Cincinnati (EMSL-CI), EPA-600/4-79-020, 1983
40CFR Part 136. Revised July 1, 1998.
"Method for the determination of Organic Compounds in Drinking Water", EPA, Office of Research and development –

Washington, EPA/600/4-88/039.
SM: "Standard Methods for the Examination of Water and Wastewater", American Public Health Association.
SW: "Test Methods for Evaluating Solid Waste", EPA SW-846 Third Edition 1986

8.0 Data Processing

8.1 Collection

Accuracy and completeness of data records are essential in maintaining the quality of laboratory results. Ink is used for all entries. All entries are signed and dated. Corrections are made with a single line through the error, a description of the reason for the change, initials, and date.

Data records are maintained for all transfers and processing of each sample from the time the sample is received until the results are reported and the sample is disposed of. The records kept for receipt, log-in, and sample custody have been discussed in Sections 7.3 and 7.4. Preparation of standard solutions is documented in the LIMS. Each stock material and solution is assigned a number, and referenced in the preparation log electronically. Prepared organic solution numbers are recorded on the analysis data sheets. In metals analysis, most solutions are prepared fresh daily and the source and identification information is recorded on the data sheets. The electronic standard solution preparation log contains entries regarding the source material, which includes:

- Compound name
- Purity
- Manufacturer and lot number
- Date received
- Concentration, if in solution form
- Solvent, when appropriate
- Date consumed or disposed of
- Expiration date
- Solution identification number

The solution preparation is documented by the following information:

- Compound identification
- Source material (by number)
- Assigned solution number
- Date prepared
- Quantity weighed out or measured by volume
- Final volume after preparation
- Solvent used
- Final concentration
- Expiration date
- Date disposed of

Data for inorganic (nonmetal) analyses are recorded in bound notebooks or LIMS batching logs assigned to each test. The required information for each analysis includes, but is not limited to: the analytical procedure; any procedure changes required; sample number; raw analytical data; standard solutions used; preparation of reagents when appropriate; signature and date. If an instrument printout is obtained for the analyses, the printouts are signed, dated and retained. The printout is inserted in the notebook if size permits. Otherwise the printout is filed in a separate file with a cross-reference recorded in the lab notebook and on the printout.

For metals analysis, a digestion log is maintained in the LIMS batching program. The digestion is documented by record of internal sample number, Client ID, analysis required or method quantity and identity of spiking solution used, initial sample volume final sample volume, initials of technician and date.

Printouts of results are obtained for graphite furnace, and cold vapor analysis. A Run log cover page is prepared to reference the analysis date, instrument identification, Sample ID, concentration corrected final results (for Cold Vapor), identity of QC or spiked samples, percent recovery obtained, and any comments. This run log is attached the instrument's data system printout. Each data set is filed in the metals department. All ICP analytical information and results are stored in the LIMS database.

Data for organic extractions are recorded in the LIMS batching program. All details regarding the extraction are recorded. The data includes the following entries: extraction method; sample matrix, extraction date; surrogate spiking solution number and concentration; matrix spiking solution numbers and concentration; Sample identification number; sample amount; quantity of surrogate and matrix spike added; final extract volume; extract storage location and signature of chemist.

Analytical data from the GC and GC/MS instruments is generated by the computer data system. Data outputs include identification of the sample, identifications of compounds retention times, and comparisons to standards. Outputs are in tabular form (retention times, areas, mass listings, etc.) and in graphic form (chromatograms, TICs, etc.). Outputs are in a standard format specified for each analysis type. Data produced are compared to information concerning the sample history, sample preservation, QC Data, etc., to judge the validity of the results.

8.2 Data Review and Validation

Phoenix Environmental Laboratories, Incorporated performs data review and validation studies on all data packages generated. Data validation is the process whereby data are accepted or rejected based upon defined criteria. Information concerning the sample history, sample preparation, Quality Control data and other factors are used in the judgement of the validity of the results. A Quality Control Audit Report is generated daily and reviewed by the Laboratory Director, Quality Control Officer and Supervisors. This computerized report compares data against current Quality Control limits, historical data information, and client specified permit exceedences among other parameters. Quality Control information is judged against set criteria to accept or reject data. Criteria used to accept or reject data are dependent upon the methodology, the client's requirements, and the eventual use of the data. All quality control parameters including method blanks, surrogate spikes, matrix spikes and duplicates, sample duplicates, laboratory control samples (QCs), field blanks, trip blanks and storage blanks must meet acceptance criteria. Where applicable, sample flags or qualifier codes shall be used to qualify data. Either the supervisor or a second analyst of equal or higher experience and responsibility reviews data. This review ensures that the following requirements have been appropriately met:

Organic Section

The analyst and Supervisor review data to ensure the laboratory provides the following where appropriate:

- Calculates the recoveries of surrogate spikes and verifies that criteria are not exceeded:
- Verifies that there are no contaminants in associated blanks outside acceptable limits;
- Compares samples and duplicates for precision in data results;

- Reviews surrogate and spike recovery data to make sure they are within quality acceptance limits;
- Verifies calibration performance for acceptability;
- Reviews and verifies instrument tuning; and
- Reviews internal standard areas of response for acceptability.
- For GC analysis, the compounds identified fell within the daily retention time window. (The daily retention time window is defined as the absolute retention time of a mid-level standard + 3 standard deviations. The standard deviation is obtained from an initial check of 3 injections of standards within a 72-hour period.)

Upon meeting all technical criteria the sample data file is then reviewed by the Organic Team Leader to:

- Verify that holding time criteria have been met;
- Ensure surrogate recovery section has been completed and acceptance limits are not exceeded;
- Ensure that all analyte compounds have been properly recorded;
- Ensure accuracy of calculations on compound quantities; and
- Ensure confirmation by GC/MS has been performed and spectra are included.

The reviewer examines the entire sample data file to ensure that all data transcription and documentation included meet customer requirements. The Organic Team Leader performs a final technical review to verify that the completed package conforms to all Quality Control criteria.

Upon completion of review, the sample data files are forwarded to the Project Manager for final review and compilation of the entire data package.

All Other Sections

- Verify that holding time criteria have been met;
- Calibration met or exceeded a correlation coefficient of 0.9975.
- Standards in the calibration curve cover the expected concentration ranges of the samples including the detection limit. All sample results fell within the range of the standard curve.
- Initial and continuing calibration verification checks met the acceptance criteria defined in the method SOP.
- Method blanks were processed with each analytical batch and were acceptable.
- Results of duplicate samples and matrix spike duplicates were within the laboratory or contract-established precision control limits.
- Matrix spike recovery was within acceptable control limits (as defined by internal control charts).
- Laboratory control samples were analyzed according to frequency specified in the SOP or contract and the results obtained were within control limits.
- Calculations have been accurately performed.
- Data for the analyses provide a complete audit trail. Data notebooks and data sheets correctly reference the analytical method, the standard solutions used, internal numbers, original data values, sample results in correct units, calculation formula for all conversions, signature of the analyst, and date. Instrument printouts must identify the person responsible for the data generation and the date of the run.

The supervisor or other data reviewer signs the data sheet to document approval. If the complete review was performed by someone other than the supervisor, a spot check is performed by the supervisor. The supervisor checks a minimum of 10% of the data. No data may be reported without supervisor approval evidenced by signature on the data page. The Laboratory Director performs a final technical review to verify that the completed package conforms to all Quality Control criteria.

The reviewed data is entered or data transferred into the LIMS by either the analyst or the supervisor. For ASP-like deliverables, a tabulation of results is prepared by the supervisor or analyst and placed in the central project file. The tabulation is transcribed into the report format by assigned report writers. The report and complete project file go to the Section Manager for final check.

The Laboratory Director's review covers the following points:

- Transcriptions are checked for accuracy and use of appropriate units.
- QC data are reviewed to assure that internal specification and contract requirements have been met.
- Nonconformance reports, if any, are reviewed for completion of corrective action and impact upon results. Information contained in the nonconformance report may need to be included in the project narrative.
- Results make sense compared to historical information about the site and results for other parameters tested at the same time.

Upon completion of review, the reports are forwarded to the Project Manager for final review and compilation of the entire data package. A copy of the signed report package is retained in the project file for archiving.

8.3 Report Information and Storage

Laboratory reports shall include:

- A cover page, which lists the states in which current certification are held, along with the laboratory identification number for that state.
- The results of specific analysis of samples with corresponding surrogate recoveries, where applicable, date and time of analysis, and analytical methods used.
- The results of batch or site specific quality control associated with specific samples and analysis, which includes blanks, laboratory control samples, matrix duplicates and matrix spikes.
- The Chain of Custody and any correspondence regarding the samples received on Chain of Custody.
- Parameters where certification is not available or not held in a certain state and/or by NELAC will be notated on the report.
- Samples that represent potable water are reported with their corresponding state or Federal Maximum Contaminant Levels (MCL). Clients are notified of MCL exceedance within 24 hours of the lab obtaining valid data. Sub-contract laboratories are notified of this requirement in writing, when utilized.
- Samples that are subcontracted are clearly marked as such, and the subcontract labs certification number is noted on the report.

Data notebooks, instrument printouts, sample chain-of-custody logs, files, and contracts are retained for a period of 12 years. If contract requirements deviate from this procedure, the contract-specified holding time is followed.

Equipment usage and calibration logs that are not study-specific are kept for a minimum of 12 years. Original SOPs, current and outdated, are permanently archived.

Most of the laboratory operations are part of the LIMS system; the prep, distillation, and analytical runs are stored electronically. These analyses are transferred from these electronic files into the result tables of the LIMS system. Once all the results are entered, the final data report is generated, reviewed and released to the client through the laboratory website. All versions of the final report and any electronic deliverables are stored on the client server drive (Y). All of these electronic drives containing all of the files are backed-up nightly and stored on ioSafe disaster proof external hard drives. Once per week an encrypted copy is taken offsite and stored in a remote safe.

For the few analyses and operations that are not yet stored electronically, hard copy logbooks and hard copy printouts of raw analytical data or supporting documents are archived by instrument, analysis or department and stored in a secure offsite facility. The facility can only be accessed by PEL employees that have signed an access log, which is kept in the control of the Operations Manager. A description of what is being retrieved from the storage area is recorded. The employee is then responsible for returning the data and signing that is has been returned.

Should the laboratory change ownership or go out of business, records will still be retained for the period of time specified above.

8.4 Transcription

Transcription is a potential source of error. The majority of data reporting is electronic, which involves transferring reviewed data directly from the instrumentation into the LIMS system. Report generation is also done electronically, keeping transcriptions to a minimum.

8.5 Data Reduction

Data reduction includes all processes that change either the form of expression or quantity of data values. The size or dimensionality of the data set is reduced.

To validate all reduction operations, all calculations or manipulations of data are recorded in the data. A description of the formula used must be provided.

Phoenix Environmental Laboratories, Incorporated uses computers, computer data systems, and microprocessor controlled instrumentation to reduce raw data to final form, such as:

- HP Environquant GC & GC/MS Data processing system (includes EPA/NIST Mass Spectral Database)
- Perkin Elmer Turbochrom 4 Data system operating on personal computers
- Perkin Elmer AA Analyst 600 & WinLab Data system
- PSA Millennium Mercury Avalon Data System
- Spectro ICP Micro Evolution and Smart Analyzer Data Systems
- HP Chem Station GC/MS Data System
- Perkin Elmer Syngistix ICP MS Software
- IC Peak Net Data System

Calculation of results is performed by these systems based on standard curve responses and is printed with each sample response and/or summarized in tabular form at the end of each analysis set.

When data calculations using linear regression are performed with calculators, the correlation coefficient, slope, and y-intercept values are recorded in the data.

The procedure for correct use of significant figures and rounding of numbers is defined in a SOP. The rounding rules cited in the USEPA Handbook of Analytical Quality Control in Water and Waste Water Laboratories are followed for all manual rounding of numbers.

9.0 Data Quality Assessment

9.1 Introduction - Definition of Terms

Accuracy

Accuracy is defined as the degree of agreement of a measurement, X with an accepted true value, T. Two types of accuracy check samples are used, Laboratory Control Samples (Blank Spike) and the Matrix Spike. The formula used to calculate accuracy for the Laboratory Control Sample is:

Accuracy = (A/B) X 100

Where A = Concentration measured
and B = Concentration spiked

which is the same formula as is used for percent recovery. For calculating accuracy in Matrix Spike analysis, a correction for background concentration found in the unspiked sample must be made. The formula is:

Accuracy = ((A- B)/C) X 100

Where A = Spiked Concentration Measured
B = Unspiked Concentration Measured
and C = Concentration Spiked

Precision

Precision is a measure of the mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Analysis precision is assessed through comparison of duplicate samples or duplicate matrix spike samples.

The term expressing precision is Relative Percent Difference (RPD) and is calculated as follows:

$$RPD = ((A_1 - A_2)/((A_1 + A_2)/ 2))X\ 100$$

Where $A_1 = Rep_1$
and $A_2 = Rep_2$

where Rep_1 and Rep_2 are replicate analyses of the same sample. and,

$$RPD = (\backslash\ MS- MSD\ ) /((MS + MSD)/ 2))X\ 100$$

Where MS = the Matrix Spike sample result
and MSD = the Matrix Spike Duplicate Result

where the Matrix Spike and Matrix Spike Duplicate analyses are performed upon the same sample.

Representativeness

Representativeness expresses the degree to which data accurately and precisely represent an environmental or process condition.

Field sampling operations have a major impact on data representativeness. Factors including site selection, sampling tools, equipment cleaning procedures, sample preservation, and many others must be considered. Similarly, laboratory operations could impact representativeness if there were day-to-day fluctuations. Accuracy and precision results of the daily quality control samples provide a measure of representativeness associated with laboratory operations.

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected under correct normal conditions. To maximize completeness of laboratory analysis, it is essential to have a sufficient quantity of each sample to provide for original and repeat analyses should the original analysis fail to meet acceptance criteria. Our goal for completeness is 100%.

Comparability

Comparability expresses the confidence with which one data set can be compared with another. This indicator of quality is enhanced at Phoenix Environmental Laboratories, Incorporated by the following controls:

- Standardized EPA approved methodology for sample preservation, holding and analysis.
- Consistent reporting units for each parameter in similar matrices.
- NIST traceable standards when available.
- Frequent analysis of QC samples.
- Participation in interlaboratory performance evaluation studies.

9.2 Methods for Attaining Quality Control Requirements

Quality Control Samples

Data quality is evaluated by the performance of Quality Control (QC) sample analysis, including:

- Method Blanks
- Surrogate Spikes
- Matrix Spikes and Duplicates
- Sample Duplicate Analysis
- Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates
- Calibration Check Samples
- Field Blank Samples
- Trip Blank Samples
- Storage Blank Samples

The particular types and frequency of QC samples processed with production samples are determined by the requirements of the client. Most common needs are those presented in the various EPA Methods, EPA SW-846, New York Analytical Services Protocol (ASP), state requirements, project requirements, customer requirements, and those requirements specified in our SOPs.

Information obtained from the above listed Quality Control samples is used to assess the quality of the data generated and is useful in identifying problems in the sampling process, in the shipment of samples, in the storage of samples, in the analysis of samples and even help in identifying problems in the analysis of the samples caused by the samples themselves. Specifically:

Method Blanks

A method blank is defined as a volume of deionized laboratory water, or in some cases a purified solid matrix carried through the entire analytical process. Data obtained from these samples indicate possible contamination in the samples picked up during the analytical process.

Surrogate Spikes

Samples are spiked with a surrogate to monitor the preparation and analysis processes of the samples. If the surrogate material(s) are not recovered in sufficient quantity from the sample the preparation and/or analysis of the sample is suspect. In the processes that surrogates are used, they are spiked into all samples including blanks. Data from the analysis of surrogates is used to construct control charts. Tables containing the in house control limits are updated by the Quality Assurance department regularly and are located at the bench for the analyst's use.

Matrix Spikes and Matrix Spike Duplicates

Matrix Spike and Matrix Spike Duplicate analysis are performed to evaluate the effect of the sample matrix upon the methodology and the precision of the method with the particular matrix. If Matrix spike compounds are not adequately recovered or vary in recovery between duplicates some measure of matrix interference is suspected. Data from the analysis of matrix spikes is used to construct control charts. Tables containing the in house control limits are updated by the Quality Assurance department regularly and are located at the bench for the analyst's use.

Sample Duplicate Analysis

Sample duplicate analysis is used to assess sample preparation and analytical method precision.

Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

Laboratory Quality Control Samples are used to assess the laboratories ability to perform an analytical method and to what level of precision. Data from the analysis of the LCS/LCSD is used to construct control charts. Tables containing the in house control limits are updated by the Quality Assurance department regularly and are located at the bench for the analyst's use

Calibration Check Samples

A Calibration Check Sample is used as a method of determining the accuracy of an instrument's calibration. If the source of the material is the same as that used for the calibration, a second check sample is also analyzed which is from a second source and of known quality and concentration. Each procedure details the acceptance limits.

Field Blank Samples

Analysis of field blank samples can give some measure of information into the possibility of contamination of samples occurring in the field during the sampling process.

Trip Blank Samples

Trip blank sample analysis is used to determine if sample contamination may have occurred during transit of the samples.

Storage Blank (Refrigerator Blank) Samples

Storage blank (Refrigerator Blank) sample analysis is used to determine if sample contamination may have occurred during the storage of the samples once they reach our laboratory facility.

Blind Quality Control Samples

The Quality Assurance Office periodically formulates blind samples for submission to the laboratory for analysis. The samples are produced by the QA Office from standard materials or from EPA ampules. Sample sets usually contain blanks, and replicates of known concentration. Analysis of the data produced from these sample are used to assess quality of data produced by the laboratory, particularly laboratory precision and accuracy.

Proficiency Samples

The Quality Assurance Office oversees the laboratories participation in routine Proficiency Testing Studies throughout the year. All NELAC certified PT analytes are analyzed for each matrix and technology certified, twice a year. The laboratory also participates in the annual EPA DMR QA study, Bacteria proficiency studies, and additional studies for new or non-NELAC analytes. Proficiency samples are analyzed as regular field samples, and are integrated into the laboratory using chain of custodies and SDG numbers.

Quality Control Charts

The QC requirements for accuracy and precision are mandated by the method and of course the clients' needs and the regulatory authority under which the work is being performed. Control Charts allow the laboratory to establish in house limits based on historical data as recommended in the Federal Register. The quality assurance department continually updates control charts based on current data points. The mean value, the warning limits and the control limits are determined for each chart.

Warning and control limits are based upon the following formula:

Upper Control Limit (UCL) = X + 3s
Upper Warning Limit (UWL) = X + 2s
Lower Warning Limit (LWL) = X - 2s
Lower Control Limit (LCL) = X - 3s

Where:
X = Mean Percent Recovery
s = Standard Deviation

Client uncertainty data is calculated using the warning limits from our control charts.

All QC sample results are tabulated immediately following analysis and compared to the in-house limits, the contract-mandated, the method-mandated, or client project-mandated control limits for precision and accuracy. Out-of-control results are cause for immediate generation of a Nonconformance report as described in Section 9.5 and possible re-extraction and/or re-analysis.

An analysis may be considered out of control whenever, as a minimum, any one of the following conditions is demonstrated by a control chart used to monitor that analysis.

- Any one point is outside of the control limits.
- Any three consecutive points are outside the warning limits.
- Any eight consecutive points are on the same side of the plotted mean.
- Any six consecutive points are such that each point is larger (or smaller) than its immediate predecessor.
- Any obvious cyclic pattern is seen in the data points.

Policy

The management and staff of Phoenix Environmental Laboratories, Incorporated makes every effort to generate data of the highest quality possible and continues to apply state-of-the-art analytical methodologies to ensure that our data continues to be of the best quality available anywhere.

Phoenix Environmental Laboratories, Incorporated makes every attempt to produce and deliver analytical data which has been demonstrated to meet contract-, method-, or client-required quality control acceptance criteria. Should anomalies occur in the processing and/or analysis of samples, which affect that objective, they are documented in the data and/or described in the report narrative.

9.3 Data Quality Objectives and Analytical Data Quality Levels

In the planning of projects for the investigation of environmental contaminants, Data Quality Objectives (DQOs) are established. Data Quality Objectives are qualitative and quantitative statements which specify the quality of data required to support decisions during remedial response activities. DQOs are applicable to all data collection activities including those performed for preliminary assessments/site investigations, remedial investigations, feasibility studies, remedial design, and remedial actions.
The level of quality and detail will naturally vary depending upon the intended use of the data. Therefore, a number of data quality reporting levels are available.

Phoenix Standard Report

A standard report includes the Sample Chain of Custody, the analytical results for the required analytes, along with reporting units, date analyzed and analyst's initials. It also includes a Quality Control section where batch QC is reported for Blank analysis, Laboratory Control Samples, Sample Duplicates and Matrix Spikes. If certain criterion is requested, a Sample Criteria Exceedance Report is also generated.

Phoenix Standard Report with General, CT-RCP, and MA-MCP Narration

This is a standard report, as above, with a Laboratory Quality Assurance Quality Control Reasonable Confidence Protocol (RCP) Narration and Checklist for Connecticut samples or Quality Control Requirements and Performance Standards in Support of Response Actions under the Massachusetts Contingency Plan (MCP) Checklist and Narration for Massachusetts samples.

Enhanced Phoenix Report – Full Data Packages

The Full Data Packages include a Phoenix Standard Report with a full data summary, which includes the following:

ASP B and Army Corps Data Packages

Organics:

- Surrogate recovery summary
- QC recovery summary
- Analytical sequence summary
- Instrument tuning logs
- Internal standard and retention time summary
- Project sample, blank sample, and QC sample raw data
- Calibration data
- Injection logs

Inorganic:

- Project sample results
- Calibration results
- Blank results
- Interference checks
- QC results
- Laboratory duplicate results
- ICP serial dilution results
- Instrument run logs
- All applicable raw data

New Jersey Reduced Deliverables Data Package

Provides a Full Data Package as above, but does not include calibration raw data for organics or instrument run logs and raw data for inorganics.

10.0 Corrective Action

10. 1 Introduction

The Quality Assurance Office is responsible for conducting periodic inspections (audits) of the quality systems, data generation, and support systems of the laboratory. The purpose of the internal audit is to assist management in identifying and correcting deficiencies and to reinforce acceptable practices. This ensures that services meet the requirements of the Laboratory Quality Manual as well as the requirements of the client.

These inspections help to ensure that the policies of the laboratory for production of high quality data are being followed, including laboratory standard operating procedures, instrument procedures, sample preparation procedures and data review policies. If discrepancies are found, corrective action is taken. Two types of audits are in place: Systems and Performance Audits. Additionally, there are routine data audits, independent audits, and audits for subcontracted services.

10.2 System Audits

A Systems Audit is an inspection and review of an entire data-generation and support system. Quality-related activities are reviewed, assessed, and compared against the Quality Assurance Program requirements for compliance. The audit includes the evaluation of personnel, facilities, Standard Operating Procedures (SOPs), and records. Systems Audits generally follow performance audits (usually by state or EPA auditors, required for certification and contract awards), and may be instituted as part of corrective action monitoring programs.

Systems Audits may also focus on a single area or aspect of laboratory operations. These inspections may consist of an in-process inspection of a particular analytical procedure, review of raw data for compliance to SOPs, or an inspection of the laboratory facility. On a quarterly basis, in-depth monitoring of data integrity is also performed on a final report. Any of these audits may be performed at any time at the discretion of the Quality Assurance Manager. Management may also direct the initiation of an audit for cause.

Systems Audits are documented in the form of an Audit Report. The Audit Report describes any findings of the audit, recommendations to correct the finding and identifies the person or persons responsible for correction implementation. A two-column format is used for the Audit Report where the left column is used to document responses by the responsible parties. A copy of the Audit Report is maintained in a chronological file while the original document is circulated to the Laboratory Supervisor, Laboratory Manager and the Laboratory Director. Once circulation is completed and all items are responded to, the Audit Report is filed by Quality Assurance. Follow-up audits will be performed to verify correction implementation. Audit Reports are considered confidential documents and shall not be shown to or discussed with those outside the company without the express consent of the Director of Laboratories and the Quality Assurance Manager.

If deficiencies are observed during a performance audit, the Quality Assurance Manager evaluates the audit report and initiates a follow-up Systems Audit, with emphasis on actions necessary to correct the deficiencies. A Corrective Action Report is completed, detailing all remedial actions to be taken, and issued to the Director of Laboratories and the Laboratory Manager for approval. If corrective action cannot be taken immediately, the anticipated date of action is provided. Once approved, the report is forwarded to the performance auditing agency or client.

Many of the objectives of a routine Systems Audit are similar to those a client or independent auditor would hope to accomplish during an On-Site Laboratory Evaluation and Data Audit. These goals include ensuring that:

- Necessary quality control (including corrective action measurement) is being applied,
- Adequate facilities and equipment are available to perform the client's required scope-of-work,
- Personnel are qualified to perform the assigned tasks,
- Complete documentation is available, including sample Chain-of-Custody,
- Proper analytical methodology is being applied,
- Acceptable data handling techniques are being used,
- Corrective actions identified in any previous on-site visits have been implemented, and
- The Laboratory Management continues to demonstrate a commitment to quality.

These objectives may be documented by completing a Laboratory Evaluation Checklist. In response to performance audits, any corrective actions taken are noted with reference to the auditor's deficiency report and the Standard Operating Procedure. Should a quantitative or qualitative error be noted in a Data Audit, a blind Performance Evaluation (PE) sample may be entered into the system to test affected parameters. Additionally, Laboratory Proficiency Tests may be scheduled if method performance is in question. Specifics of these two programs are outlined in the following sections.

10.3 Performance Audits

A performance audit is a planned independent check of the operation of a measurement system to obtain a quantitative measure of the quality of the data generated. In practice, this involves analysis of standard reference samples or materials that are certified as to their chemical composition or physical characteristics.

The Quality Assurance Office prepares and submits performance testing (PT) samples to the laboratory periodically. The fact that the samples are PT samples is not revealed to analysts or supervisors. These blind samples provide a check on all operations performed in the lab, including bottle preparation, sample holding, extraction, analysis, data validation, and reporting. The blind PE samples are prepared from EPA reference materials. Findings reported by the laboratory are compiled into a summary report by the assigned QA Specialist and issued to the Director of Quality Assurance and Laboratory Directors. Unacceptable results require investigation by the Laboratory Director, documentation of corrective action by the Laboratory Director, and follow-up review by the Quality Assurance Office.

10.4 Audits of Subcontractors

Analysis performed by subcontractors must conform to Phoenix Environmental Laboratories' Quality Control requirements. Subcontractors must meet the requirements of the Phoenix Environmental Laboratories' Quality Assurance Program or have in place an equivalent program of their own. Potential subcontractors will be reviewed by the Phoenix Environmental Laboratories for suitability.

The Quality Assurance Office will evaluate the Quality Assurance Program of the subcontractor through review of the laboratory's written Quality Manual, the Quality Assurance Project Plan (where applicable), Quality Control SOPs, typical SOPs, and latest applicable USEPA Performance Evaluation or NELAC Performance Testing Study results. If the results are not available, Phoenix Environmental Laboratories may submit blind PE samples to the subcontractor. An on-site audit of the facility will be performed as deemed necessary by the Laboratory Director or Director of Quality Assurance.

10.5 Nonconformance Event Corrective Action and Documentation

Documentation of analytical problems and corrective action taken is an essential part of the data record. Identification, implementation, and monitoring for the actions that could have prevented the analytical problem provide a method for improving the quality of laboratory performance. A Nonconformance report sheet (Figure 1) has been designed to record problems, corrective actions, impact on analytical results, and suggested preventive actions for the future.

The Nonconformance Report must show complete background information about the event, including date and shift; analysis and phase; the client name; the sample identification number; and a description of the event that occurred. The report further includes the corrective action taken; indication of the status of the system; an assessment of impact on analytical results; and suggestions for preventive action.

The Nonconformance Report should be initiated by the person experiencing or noticing the discrepancy and completed by his or her supervisor. For example, the initiator may provide the description of the event and corrective action taken; the supervisor adds the impact and preventive action.

Copies of the completed reports should be distributed to the Project Manager, the Laboratory Section Director, and the Director of Quality Assurance. If the event has caused any impact on the analytical results, the Project Manager will meet with the Quality Assurance or Laboratory Director and then communicate with the client, either personally or through the Client Services group. If the impact on analytical results affects drinking water potability or MCL exceedances (such as bacteria, or nitrate/nitrite), the client will be notified immediately (within 24 hours). Client notification of other issues will be made in a reasonable time frame, and usually within three to five working days.

The Laboratory Director should check that corrective action has been appropriate, confirm analytical impact, and ensure the implementation and monitoring of preventive action.

The Director of Quality Assurance should review the Nonconformance Reports for follow-up action. On a regular basis, the Director of Quality Assurance will meet with Project Managers and Laboratory management to evaluate corrective action and preventive action effectiveness. All effective preventive action will be documented for all appropriate laboratory sections. Supervisors of each area will be responsible for any SOP revision needed to reflect these preventive actions.

Initial preventive action plans, which prove to be ineffective, will cause a team to be formed to identify the root cause of the problem and the effective preventive action. This team will be led by the supervisor of the area where the initial nonconformance occurred and at least one member of the Quality Assurance Unit and management. Progress of this team and monitoring of the effectiveness of preventive action will be documented by the team leader and by the Director of Quality Assurance.

Figure 1
Nonconformance Report

Document #:

Phoenix Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O.Box 370, Manchester, CT 06040
Tel. (860) 645-1102 Fax (860) 645-0823

Date Closed:

Corrective / Preventive Action Log

BASIS:	DESCRIPTION:	METHOD:
Audit Complaint ☐ PT Failure ☐ Deficiency ☐ QC Failure ☐ SOP Departure ☐ Prevention		

DATA:
Type:
Samples:

RECORDED BY: DATE:

ROOT CAUSE: / PURPOSE:

INVESTIGATED BY: DATE:

POTENTIAL CORRECTIVE / PREVENTIVE ACTIONS:

RECOMMENDED BY: DATE:

ACTIONS PERFORMED:

Disposition of Data:
☐ Reanalyzed
☐ Rejected
☐ Qualified
☐ Recalled

PERFORMED BY: DATE:

FOLLOW-UP ACTIVITIES:

ASSESSED BY: DATE:

Closed by
Date Closed:

11.0 Client Complaint Policy

In order to best meet the needs of our clients, Phoenix Environmental Laboratories has implemented a procedure for the prompt handling of client complaints. The project manager summarizes the nature of the complaint in their logbook located in the Client Services Department.

If the complaint includes a request for re-analysis or re-evaluation of the data, the complaint and a printout of the error report is provided to the QA/QC department and to the section supervisor. This is recorded in the logbook. If a non-conformance event is uncovered as a result of the re-analysis or re-evaluation, a non-conformance or error report is generated.

Whether a non-conformance or error report is generated or not, the Client Services Department responds promptly (usually within 24hours) to the Client.

12.0 Client Confidentiality

Confidentiality is an important aspect of the service that Phoenix Environmental Laboratories provides our clients.

All material containing client's analytical results, project specific information, and invoice information is considered strictly confidential. Reports containing any of this information are provided only to the client or his/her designee as provided on the chain of custody.

Additional requests for information are provided only after verbal authorization by the client.

13.0 Implementation Requirement and Schedule

The Quality Assurance Manual shall become fully effective on the first day of October 1995. Any questions regarding implementation should be addressed to the Director of Quality Assurance or the Laboratory Director.

14.0 References

Regulations

40 CFR 136.3e	Required containers, preservation techniques, and holding times
40 CFR 136	Guidelines establishing test procedures for the analysis of pollutants under the Clean Water Act
40 CFR 136 Appendix A	Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater
40 CFR 136 Appendix B	Definition and Procedures for the Determination of the Method Detection Limit – Revision 3.0
40 CFR 136 Appendix C	Inductively Coupled Plasma - Atomic Emission Spectrophotometer Method for Trace Element Analysis of Water and Wastes Method 200.7
40 CFR 141	National Primary Drinking Water Regulations
40 CFR 143	National Secondary Drinking Water Regulations
40CFR160	Federal Insecticide, Fungicide and Rodenticide Act (FIFRA), Good Laboratory Practice Standards, Final Rule

Manuals

EPA 600/4-79-020	Method for Chemical Analysis of Water and Wastes (1983)
EPA 600/4-79-012	Quality Assurance Handbook for Analytical Quality Control In Water and Wastewater Laboratories (1979)
EPA 600/R-94-111	Methods for the Determination of Metals in Environmental Samples - Supplement I (May 1994)

EPA 600/R-93/100	Methods for the Determination of Inorganic Substances in Environmental Samples (August 1993)
EPA 600/4-88/039	Methods for the Determination of Organic Compounds in Drinking Water (Rev July 1991)
EPA 600/4-90/020	Methods for the Determination of Organic Compounds in Drinking Water Supplement I, (July 1990)
EPA 600/R-92/129	Methods for the Determination of Organic Compounds in Drinking Water Supplement II (August 1992)
EPA 600/R-95/131	Methods for the Determination of Organic Compounds in Drinking Water Supplement III (August 1995)
EPA 540/G-87/003	Data Quality Objectives for Remedial Response Activities, Development Process
EPA 540/G-87/004	Data Quality Objectives for Remedial Response Activities, Example Scenario: RI/FS Activities at a Site with Contaminated Soils and Groundwater.
EPA 815-B-97-001	Manual for the Certification of Laboratories Analyzing Drinking Water 4th edition (March 1997)
EPA 821-R-16-009	Approved Clean Water Act Test Methods: Organic Compounds (August 2017)
SW-846	Test Methods for Evaluating Solid Wastes, Revision 8, Update 5, July 2014
Standard Methods	Standard Methods for the Examination of Water and Wastes, 22nd Edition, American Public Health Association.

QAMS 004/80	Guidelines and Specifications for Preparing Quality Manuals, USEPA Office of Monitoring System and Quality Assurance, September 20, 1980
QAMS 005/80	Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, USEPA Office of Monitoring System and Quality Assurance, December 29, 1980
NEESA 20.2-047B	Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program, June 1988
USATHAMA PAM 11-41	U.S. Army Toxic and Hazardous Materials Agency, Quality Assurance Program, January 1990.
	Drinking Water Regulations and Health Advisories by Office of Drinking Water USEPA, April, 1990

Phoenix Environmental Laboratories, Incorporated

Quality Manual

Appendix A

Resumes of Key Personnel

THE PEOPLE OF PHOENIX ENVIRONMENTAL LABORATORIES, INC.
Technical Staff Education and Experience

Phyllis Shiller
Laboratory Director

Responsibilities:	Technical Director of Laboratory Operations and Services. Manages laboratory personnel and staffing. Responsible for laboratory scheduling and maintenance of high sample throughput. Provides client interface and management of special projects, technical issues and regulatory matters. Works with QA/QC Manager to ensure all aspects of corporate quality control program are strictly adhered to.
Education:	University of Rhode Island, B.S. Chemistry, 1986
Experience:	Thirty-six years of environmental laboratory experience, including positions as QA/QC Director, Inorganic, ICP/GFAA Specialist, Inorganic Manager of a large (CLP) laboratory, Operations Manager, and Laboratory Director.

Bobbi Aloisa
Vice President

Responsibilities:	Management of Client Services Operation. Provides client interface with laboratory. Responsible for scheduling report deadlines with the client. Responsible for the generation of reports including progress reports, final reports, and electronic deliverables. Provides second level of review for all reports. Provides immediate review of incoming projects for completeness. Manages program that furthers the laboratory's ability to achieve consistent high levels of performance and quality.
Education:	Manchester Community Technical College, A.S. Science, 1994
Experience:	Twenty-nine years of environmental laboratory experience.

Greg Lawrence
Assistant Laboratory Director

Education:	University of Hartford, Hartford, CT, Masters Business Administration, 1988 Keene State College, Keene, NH, B.S. Chemistry, 1982
Experience:	Forty-one years of environmental laboratory experience, including the position of Laboratory Director since 1985. Background in Organic Instrumentation, AA Spectrometry and Quality Control.

Kathleen Cressia
QA/QC Officer
Microbiology Laboratory Director

Education:	Western Connecticut State University, Danbury, CT, B.A. Earth Science/Biology, 1985
Experience:	Thirty-six years of environmental laboratory experience, including positions as Laboratory Director, Laboratory Operations Manager, QA/QC Manager, Director of Microbiology, Inorganic Manager, and Wet Chemistry Section Leader for a CLP Laboratory.

Linnea Skoglund
Quality Specialist

Education:	Westfield State University, Westfield, MA, B.S. Biology, Chemistry minor, 2019
Experience:	Three years of environmental laboratory experience.

Peter LaBarre
Database Administrator

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Computer Science, 1990
Experience:	Sixteen years of Laboratory Information Systems support.

Maryam Taylor
Project Manager

Education:	Nizam College, India, B.S. Chemistry, 1978
Experience:	Twenty-eight years of experience in the environmental laboratory field including GC/MS analyst, Organics Department Manager and Project Manager.

Alejandro Paredes
Project Manager

Education:	Universidad Rafael Landivar, Guatemala, B.S. Marketing, 2004
Experience:	Fourteen years in the environmental laboratory field, including GC/MS analyst and Data Specialist.

Helen Geoghegan
Project Manager

Education:	University of New Haven, New Haven, CT, M. S. Environmental Science, 1995 University College Dublin, Dublin, Ireland, B.S. Chemistry, 1986
Experience:	Thirty-three years of environmental laboratory experience, including the position of Co-Laboratory Director. Background in Organic and Inorganic Analysis, Quality Control, Data Validation, Technical Review.

Keith Aloisa
Organics Department Team Leader

Education:	Quinnipiac University, Hamden, CT, B.S. Chemistry, 1993
Experience:	Twenty-eight years of experience in the environmental laboratory field including Organic manager and QA Specialist.

Raman Makol
Organics Department Team Leader

Education:	Guru Nanak Dev University, India, M.S. Chemistry, 1986 Guru Nanak Dev University, India, B.S., Chemistry, 1984
Experience:	Thirty-two years of analytical and environmental laboratory experience as an analyst and R&D Specialist.

Harry Mullin
GC/MS Lead Analyst

Education:	Providence College, Providence, RI, B.S. Biology, 1986
Experience:	Thirty-six years of experience in the environmental laboratory field including Organic Laboratory Manager.

Hongjie Li
GC/MS Analyst

Education:	University of Petroleum, Beijing, China, M.S. Applied Chemistry, 1989 University of Alberta, Edmonton Alberta, Canada, M.S. Environmental Engineering, 2000
Experience:	Nineteen years of experience in the environmental laboratory and R&D field.

Michael Hahn
GC/MS Analyst

Education:	University of Connecticut- Biological Sciences Embry-Riddle Aeronautical University- Avionics Engineering
Experience:	Thirty-two years of environmental laboratory experience.

Wes Bryon
GC Analyst

Education:	Holyoke Community College, Holyoke, MA, A.S. Environmental Science, 2000
Experience:	Twenty-two years of environmental laboratory experience.

Jeffery Bucko
GC Analyst

Education:	Eastern Connecticut State University, B.A. History, 1991
Experience:	Twenty-eight years of experience in the analytical laboratory field.

Adam Werner
GC/MS Analyst, GC Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Molecular & Cellular Biology, 2011
Experience:	Fourteen years of experience in the analytical laboratory field.

Saadia Chudary
GC Analyst

Education:	Central Connecticut State University, New Britain, CT, B.S. Biomolecular Science, 2013, M.S. Biomolecular Science, 2015
Experience:	Nine years of environmental laboratory experience.

Christina Nieves
GC/MS Analyst

Education:	University of New Haven, West Haven, CT, B.S. Forensic Science, 2012
Experience:	Nine years of environmental laboratory experience.

James Karabetsos
GC/MS Analyst, Sample Preparation Analyst

Education:	University of New Haven, West Haven, CT, B.S. Biology / Forensic Science, 2013
Experience:	Eight years of environmental laboratory experience.

Cynthia Pearce
ICP Analyst

Education:	University of Florida, Gainesville, FL, B.S. Chemistry 1977
Experience:	Thirty-four years of environmental laboratory experience.

Mike Hornak
Metals Analyst

Education:	Quinnipiac University, Hamden, CT, B.S. Chemistry, 1989
Experience:	Thirty-three years of environmental laboratory experience.

Tina Hall
Metals Analyst, Sample Preparation Analyst

Education:	Hood College, Fredrick, MD, B.A. Biology 1995
Experience:	Twenty-five years of environmental laboratory experience.

Ian Enders
Metals Analyst

Education:	Central Connecticut State University, New Britain, CT B.S. Earth Science, 2021
Experience:	Two years of environmental laboratory experience.

Rashmi Makol
Microbiology Team Leader

Education:	Kurukeshtra University, India, B.S. Chemistry
Experience:	Twenty-three years of environmental chemistry and microbiology lab experience.

Eric Geyer
Inorganic Team Leader

Education:	University of Connecticut, Storrs, CT, B.S. Natural Resources, 1997
Experience:	Twenty-five years of environmental laboratory experience.

Kandi Della Bella
Inorganic Analyst, Microbiology Analyst

Education:	Saint Joseph College, West Hartford, CT, B.S. Natural Science, 1996 M.S. Biology, 2007
Experience:	Fifteen years of environmental laboratory experience.

Greg Danielewski
Inorganic Analyst

Education:	Capital Community Tech College, Hartford, CT, A.S. Chemical Engineering Technology, 1993
Experience:	Twenty-nine years of environmental laboratory experience.

Matt Fijolek
Inorganic Analyst

Education:	University of New England, Maine, B.S. Marine Biology
Experience:	Seventeen years of environmental laboratory experience.

Jean Rawlings
Inorganic Analyst

Education:	Bucknell University, Lewisburg, PA, B.S. Biology 1995
Experience:	Nineteen years of environmental laboratory experience.

Brian Sheriden
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Biology/English, 2001
Experience:	Seventeen years of environmental laboratory experience.

Michael Tran
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Computer Science and Engineering, 2012
Experience:	Six years of environmental laboratory experience.

Mary LaVallee
Inorganic Analyst

Education:	Southern Connectcut State University, New Haven, CT, B.S. Chemistry, 2022 Naugatuck Community College, Waterbury, CT, A.S. Science, 2007
Experience:	Eight years of environmental laboratory experience.

Elizabeth Rideout
Inorganic Analyst

Education:	Stevenson University, Ownings Mills, MD, Central Connecticut State University, New Brittain, CT, Chemistry Major
Experience:	One year of environmental laboratory experience.

Meredith Weigert
Inorganic Analyst, 2nd shift Team Leader

Education:	University of Connecticut, Storrs, CT, B.S. Biology, 2019
Experience:	Two years of environmental laboratory experience.

John Mark Woodworth
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Animal Science, 2020
Experience:	One year of environmental laboratory experience.

Catherine Lundigan
Inorganic Analyst

Education:	Bay Path University, Longmeadow, MA, B.S. Biology, 2022.
Experience:	Less than one year of environmental laboratory experience.

Daniel Kinney
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Environmental Science, 2019
Experience:	One year of environmental laboratory experience.

Angelica Martinez
Inorganic Analyst

Education:	Bay Path University, Longmeadow, MA, B.S. Forensic Science, 2020
Experience:	Two years of environmental laboratory experience.

Praveena Krishnaprasad
Inorganic Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Biology, 2021
Experience:	One year of environmental laboratory experience.

Dylan Tillman
Inorganic Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Biology, 2022
Experience:	One year of environmental laboratory experience.

Dina Montagna
Sample Preparation Day Supervisor

Education:	Springfield College, Springfield, MA, B.S. Biology/Chemistry, 1999
Experience:	Twenty-three years of environmental laboratory experience.

Tara Banning
Sample Preparation Evening Supervisor

Education:	University of Connecticut, B.S. Biology, 2007
Experience:	Fourteen years of environmental laboratory experience.

Ashraf Sheikh
GC/MS Sample Preparation Analyst

Education:	South Gujarat University – Surat, India, B.S. Chemistry, 1990
Experience:	Twenty-three years of environmental laboratory experience.

Mary Tran
GC/MS Sample Preparation Analyst

Education:	Central Connecticut State University, New Britain, CT, B.S. Biology, 2015
Experience:	Eight years of environmental laboratory experience.

Jose Paz Soldan
GC/MS Sample Preparation Analyst

Education:	University of Connecticut, Storrs, CT, B.S. Molecular and Cellular Biology, 2021
Experience:	One year of environmental laboratory experience.

Melissa Kemp
GC/MS Sample Preparation Analyst

Education:	University of Saint Joseph, West Hartford, CT, B.S. Chemistry, 2018
Experience:	One year of environmental laboratory experience.

Adrian Jaworski
Air Sample Preparation Analyst, GC/MS Assistant

Education:	Manchester Community College, B.S. Chemistry Expected.
Experience:	One year of environmental laboratory experience.

Matthew Richard
GC/MS Assistant, Sample Preparation Analyst

Education:	Central Connecticut State University, New Britain, CT, B.S. Biology and Psychology, 2014
Experience:	Seven years of environmental laboratory experience.

Robert Looney
GC/MS Assistant, Sample Preparation Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, Environmental Earth Science major
Experience:	Four years of environmental laboratory experience.

Sarah Kelting
GC/MS Assistant, Air Lab Assistant

Education:	University of New England, Biddeford, ME, B.S. Environmental Science
Experience:	Three years of environmental laboratory experience.

Anvarhusen Sheikh
Sample Preparation Analyst

Education:	Polytechnic Institute, Valsad Gujarat India, A.S. Chemical Engineering, 1983
Experience:	Twenty-two years of environmental laboratory experience.

Lisa Luchini
Sample Preparation Analyst

Education:	Central Connecticut State University, M.S. Biomolecular Science, 2010; B.S. Biomolecular Science, 2005
Experience:	Eight years of environmental laboratory experience.

Dustin Harrison
Sample Preparation Analyst

Experience:	Eighteen years of environmental laboratory experience.

Autum Burke
Sample Preparation Analyst

Education:	Central Connecticut State University, New Brittain, CT, M.S. Environmental Science, Ongoing, B.S. Biology, 2019
Experience:	Two years of environmental laboratory experience.

Gregory Mercier
Sample Preparation Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Environmental Earth Science, 2013
Experience:	Eight years of environmental laboratory experience.

Rowena Wagner
Sample Preparation Analyst

Education:	Trace Computer College, Laguna College, West Negros University, Philippines – Computer Programming Certificate
Experience:	Five years of environmental laboratory experience.

Edgar Cortez
Sample Preparation Analyst

Experience:	Sixteen years of environmental laboratory experience.

Kira Wayman
Sample Preparation Analyst

Education:	University of Southern Mississippi, Hattiesburg, MS
Experience:	Four years of environmental laboratory experience.

Christine Luckhoo
Sample Preparation Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Biology, 2021
Experience:	One year of environmental laboratory experience.

Dylan O'Hagan
Sample Preparation Analyst

Education:	Eastern Connecticut State University, Willimantic, CT, B.S. Health Science, 2021
Experience:	One year of environmental laboratory experience.

Tamara Rodriguez
Sample Preparation Analyst

Education:	University of Saint Joseph, West Hartford, CT, B.S. Biology, 2020
Experience:	One year of environmental laboratory experience.

Kailey LeBlanc
Sample Preparation Analyst

Education:	Rhode Island College, Providence, RI, B.S. Health Science
Experience:	One year of environmental laboratory experience.

Megan Ortiguerra-Rojas
Sample Preparation Analyst

Education:	University of North Texas, Denton, TX, B.S. Ecology
Experience:	One year of environmental laboratory experience.

Javaun Murphy
Sample Preparation Analyst

Education:	Western New England University, Springfield, MA, B.S. Chemistry, 2022
Experience:	Less than one year of environmental laboratory experience.

Aleisha Price
Sample Preparation Analyst

Education:	University of Connecticut, Storrs, CT, B.S Chemistry Expected 2023
Experience:	Less than one year of environmental laboratory experience.

Phoenix Environmental Laboratories, Incorporated

Quality Manual

Appendix B

Equipment List, Laboratory Overview & Certifications

PHOENIX ENVIRONMENTAL LABORATORIES, INC.
Major Equipment List

Organics GC

16 - Perkin Elmer Autosystem with dual Electron Capture Detectors

1 - Markelov HS 9000 Headspace Analyzer with Perkin Elmer Autosystem with F10

1 - Perkin Elmer Autosystem with Nitrogen Phosphorus Detector

11 - Perkin Elmer Autosystem with Flame Ionization Detectors

1 - Agilent 7890A Autosystem with PID and FID detectors, Centurion autosampler and Tekmar 3000 Purge and Trap concentrator

12 - PE Nelson 970 Data Interfaces

6 - PE Nelson 600 Series Link Interfaces

8 - PE Nelson Turbochrom 4.1 Data System

2 - Agilent 6890N Autosystem GC/MS with 7683 autosampler

1 - Agilent 7890B Autosystem with dual ECD detectors, with 7693 autosampler

Organics GC/MS

2- Agilent 5973 MSD with 6890 GC, Arcon 8100 Autosampler, two Tekmar 3000 Purge and Trap concentrators, PT2 switching valve box, HP Chemstation and Enviroquant software

9- Agilent 5973 MSD with 6890 GC, 7683 injector, HP Chemstation and Enviroquant software Semivolatiles

2- Agilent 5975 MSD with GC, 7683B injector, HP Chemstation and Enviroquant software Semivolatiles

1- Agilent 5973 MSD with 6890 GC, Arcon 8100 Autosampler, two EST Encon Purge and Trap concentrators, PT2 switching valve box

2- Agilent 5975 MSD with 7890 GC, Centurion Autosampler, Encon Evolution purge and trap concentrator

6- Agilent 5973 MSD with 6890GC, Encon Evolution Purge and trap concentrator, Centurion Autosampler, Chemstation Enviroquant software

1- Agilent 5973 MSD with 6890GG, Centurion Autosampler, Encon Evolution purge and trap concentrator

1- Agilent 5977A MSD, 7890B GC, Centurion Autosampler, Encon Evolution purge and trap concentrator

1- Agilent 7980A GC with OI 4430 PID/FID Centurion Autosampler, Tekmar 3000 purge and trap concentrator

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Organics HPLC

2- Agilent 1100 series with Diode Array Detectors G131SB. G1316A Colum Thermostat, G1312A Binary Pump, G1367A Autosampler

2- Agilent 1100 series with FLD (1321A) Autosampler (G1313A), column thermostat (G1316a) Quarter Pump, (G1311a), Pickering PCX5100, Pickering vector post column derivatization unit

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Air Laboratory

1- Agilent 5975 with 7890 GC and HP Chemstation

1- Agilent 5977A with 7890 GC and HP Chemstation

1- Entech 7100AR Cryogenic concentrator- cold trap dehydration

1- Entech 7200 Cryogenic concentrator

1- Entech 7650 20 Minican Autosampler with 18 auxiliary positions.

1- Entech 7500A minican Autosampler with 9 auxiliary positions.

2- Entech 3100A canister cleaner accompanied with Thermoscience oven

1- Entech 3100D canister cleaner accompanied with Thermoscience oven

1- Entech 4600A Dynamic Dilutor

1- Entech 4700 Precision Dilutor

Metals

1- Spectro Blue 37 Channel Simultaneous Axial Plasma ICP Spectrometer with Autosampler and Smart Analyzer software

2- Spectro Arcos ICP-EOP with ESI SC Autosampler and Smart Analyzer Software

1- Perkin Elmer NexION 350X ICP Mass Spectrometer

2- Perkin Elmer AAnalyst 600 Atomic Absorption Spectrophotometer (AA) with graphite furnace, Zeeman background & AS 800 Autosampler

1- PSA Mercury Millennium System with Autosampler and mercury cold vapor detector.

2- OHAUS Model SPX223 balance

Prep Department

3- UTC Vacuum Solid Phase Extractor Manifolds

100- Liquid/Liquid Extraction Systems

7- Buchi Synacore Concentration Systems with V-855 Vacuum Controllers

4- Zymark TurboVap II Automated Sample Concentration Workstations

4- Zymark TurboVap LV Automated Sample Concentration Workstations

1- Vacuum and Pressure Filtration System, 11 positions

2- Branson DHA1000 Ultrasonic Cleaners

2- VWR 250D Ultrasonic Cleaners

25- Millipore Zero Headspace Extraction Chambers

3- Millipore TCLP Rotary Extractors ZHE, 12 positions

1- Multi Position TCLP Rotation Extraction Systems

12- Dionex ASE200 Accelerated Solvent Extractors

40- Radley Manual Soxhlet Extractors- 5 position

2- Questron Vulcan 84 AutoBlock Digester

5- Environmental Express HotBlocks Digesters, 54 Position

1- Milestone Ethos UP Microwave Digester

1- IEC Centra-8 Centrifuge

6- Tekmar TM600-2 Dual Horn Sonic Disruptors

6- Mettler PB802S/PB1502S/PB3002 Balances

1- EM Series EK-1 Balance

1- Mettler Analytical AE240 Balance

1- PlasLabs 863-CG Dessicator

1- Blue M DC336F Oven

1- VWR 1300U Oven

2- GlasCol 3D Separatory Funnel Shaker, 8 position

1- GlasCol 3D Separatory Funnel Shaker, 4 position

2- Thermo Scientific 40 position block digester

3- MARS 6 Microwaves

WET LAB

1- Lachat Quikchem 8000 Dual Channel Wet Chem Autoanalyzer with 360 Position Autosampler.

2- Lachat Quikchem 8500 Four Channel Wet Chem Autoanalyzer with 360 Position Autosampler.

2- Tekmar LOTIX TOC Analyzer with 30 position Autosampler

1- Tekmar Apollo TOC in soil Analyzer

1- HACH DR5000 Spectrophotometer

2- Beckman Coulter DU 720 Spectrophotometer

2- Hach Sension 7, Conductance Meter

1- Yamato DX600 Drying Oven

4- Precision Scientific Pensky-Martens Flash Point Testers

1- Orion 710A Meter

1- Thermo Scientific Heratherm Drying Oven

1- Mettler PJ360 Top Loader Balance

1- Mettler AE240 Analytical Electronic Balance

1- Man-Tech AM197 Automated Liquid Handler (pH, Alkalinity, Conductivity, Turbidity)

2- Mettler XS-104 Analytical Electronic Balance

1- Mettler PB5001-S Top Loader Electronic Balance

3- LabCrest Midi Distillation Systems, 10 position

3- AIM 500 Automated Block Digesters, 28 position

2- Dionex DX120 Ion Chromatograph with Autosampler

2- Beckman Coulter CU720 Spectrophotometer

1- Yamato DX400 Drying oven

1- Thermolyne 48000 Furnace

1- Thermolyne 1300 Furnace

2- Hach COD reactor, 25 position

2- Horizon SpeedVap II 9000 Solvent Evaporation System

1- VWR 750HT Ultrasonic Cleaner

1- GlasCol 3D Separatory Funnel Shaker, 8 position

1- CAI SmartBlock 226 COD Digester, 100 position

1- Hydro System Reverse Osmosis 500 gallon water system

2- Hach TL2300 Turbidimeter

1- VWR B30PCI pH meter

2- Barnstead Nanopure Diamond DI Water System

1- Tekmar Dohrman Apollo 9000 TOC Analyzer w/ Model 183 Boat Sampler for Solids

1- Mantech Gilson 215 Liquid Handler

1- Mantech T10 Turbidimeter

Microbiology

4- VWR 2020 BOD Incubators, High Volume

2- VWR 2030 BOD Incubators, High Volume

1- Sheldon Manufacturing SR120P Incubator, High Volume

2- YSI 52 Oxygen Meter (BOD)

1- ManTech AM300 series dual probe, 90 position BOD analyzer

3- Precision Coliform Incubator Water baths

1- VWR Bacteriological Incubators

1- Market Forge Sterilmatic Autoclave

1- Vacuum Filtration System, 3 position

1- Fisher Scientific Isotemp Bacteriological Incubator

1- Thermo Scientific HeraTherm Bacteriological Incubator

1- Reihert-Juns Quebec Darkfield Colony Counter

1- Spectroline EA-160 UV light (366 nm)

1- American UV Company UV box (254 nm)

1- IDEXX Quanti-Tray Sealer

1- IDEXX Quanti-Tray Sealer PLUS

2- Insignia Fridge

PHOENIX ENVIRONMENTAL LABORATORIES, INC.

GENERAL INFORMATION & CONDITIONS

HOURS OF OPERATION/PRIOR NOTIFICATION

Hours of Operation: Sample receiving hours are 7:00 a.m. to 7:00 p.m. Monday through Friday; and 9:00 a.m. to 1:00 p.m. on Saturdays. Laboratory operation hours are 6:00 a.m. to 11:00 p.m. Monday through Friday and a limited Saturday schedule. Prior notification is required for delivery of emergency samples.

SAMPLE PICKUP

Phoenix Environmental Laboratories, Inc. offers courier service throughout our service area of Connecticut, New York, Massachusetts, Rhode Island, Vermont, Maine and New Hampshire. Pickups should be scheduled 24 hours in advance. Please contact Phoenix Client Services for sample pickup or emergency response.

TURNAROUND TIMES

Phoenix Environmental Laboratories, Inc. shall make its best effort at meeting all client specified turnaround times. Phoenix shall not however be liable for late delivery of services except as provided by written agreement prior to sample receipt.

SURCHARGE FOR EXPEDITED WORK

Normal turnaround is 5 working days. Results required in less than five working days are assessed a surcharge for accelerated turnaround. Please contact the Sales Department for available turnaround times and applicable charges.

EXPEDITED WORK/RUSH PROJECTS

A computer generated progress report or verbal results will be made available within the agreed time period with the written report available within (1) day following the progress report. Client requirements for "same day" written reports must be approved prior to sample delivery.

DUE DATE

Due date is defined as the date of analysis completion with verbal or computer generated sample progress reports results available "same day" for expedited rush work. Completed written reports are available by 5 p.m. the following day and are mailed first class, U.S. Postal Service.

SAMPLE RECEIPT

Samples must be received at Phoenix before 3:00 p.m. to be considered as received on that day. Samples received after 3:00 p.m. shall be considered as having been received on the next working day for purposes of calculating turnaround time. Phoenix Environmental Laboratories, Inc. reserves the right to reject samples deemed unsuitable.

SAMPLE HOLDING TIME/PRESERVATION

Customers must deliver all samples to Phoenix within holding time or where short holding times are not required, a maximum of two days from sample collection. It is the client's responsibility to assure that all samples are preserved and delivered in accordance with published protocol.

DOCUMENTATION

All samples submitted to Phoenix Environmental Laboratories, Inc. must be accompanied with a completed Chain-of-Custody form.

SAMPLE DISPOSAL/STORAGE

Phoenix will responsibly dispose of most unused samples, while reserving the right to return unused samples to the client. Please consult our sample custodian at time of delivery for additional information. Sample storage will not extend past 30 days from final report date except by previous arrangement.

SUBCONTRACTED SAMPLES

A limited number of analysis such as radionuclides are subcontracted to licensed laboratories, which Phoenix maintains a contractual agreement. Subcontracted samples maybe subject to extended turnaround times.

RECORD RETENTION

Phoenix shall retain all pertinent records for a period of ten (10) years from sample receipt, except in the case of Lead and Copper testing, where records are retained for twelve (12) years. There may be a minimal charge for the retrieval of these records from archives, should a client request this service.

CERTIFICATIONS

Phoenix Environmental Laboratories, Inc. participates, on an annual basis in many different certification and proficiency programs. Some states extend reciprocal certification to Phoenix Environmental Laboratories, Inc.

Phoenix Environmental Laboratories Inc. holds certifications in the following states:

Connecticut (Lab. Registration #PH-0618)

Maine (Lab. Registration #CT-007)

Massachusetts (Lab. Registration #MA-CT007)

New Hampshire (Lab. Registration #2136 and #2058)

New York / NELAC (Lab. Registration #11301)

New Jersey (Lab. Registration #CT003)

Rhode Island (Lab. Registration #63)

Vermont (Lab. Registration #VT11301)

Pennsylvania (Lab. Registration #68-03530)

Phoenix Environmental Laboratories, Incorporated

Quality Manual

Appendix C

Organizational Chart

Phoenix Environmental Laboratory Staff

July 2022

Peter Labarre
IT

Kathy Cressia
Linnea Skoglund
QA/QC

Phyllis Shiller
Laboratory Director

Greg Lawrence
Assistant Laboratory Director

Client Services Department

Bobbi Aloisa
Vice President/Manager

Loreen Fay
Project Mngr / Asst to VP

Sarah Bell
Project Manager

Makrina Nolan
Project Manager

Helen Geoghegan
Project Manager

Lisa Arnold
Project Manager

LIMS
- Shannon Whilhelm
- Lori Bailey
- Christine Paradise

Sample Custodians
- Monica Pellerin
- Stefanie Lesight
- Krystal Delgado
- Emma Johnson
- Madison Lathe

Organic Department

Raman Makol
GC/MS Team Leader & HPLC

Keith Aloisa
GC/MS Team Leader & Air

Harry Mullin
GC/MS Lead Analyst

GC/MS Analysts:
- Jane Li
- Michael Hahn
- Wes Bryon
- Adam Werner

GC Analysts:
- Adam Werner
- Jeff Bucko
- Saadia Chudary
- Christina Nieves

VOA Assistants:
- Ashraf Sheikh
- James Karabetsos
- Jose Paz Soldan
- Melissa Kemp

GC & SV Assistants
- Mary Tran
- Christina Nieves
- Matthew Richards
- Adrian Jaworski
- Robert Looney

Metals Department

Emily Kolominski
ICP Analyst

Cynthia Pearce
ICP-MS Analyst

Tina Hall
Analyst

Mike Hornak
Analyst

Ian Enders
Hg Analyst

Christina Szachon
Analyst

Sample Preparation Department

Dina Montagna
Day Team Leader

1st Shift Analysts:
- Lisa Luchini
- Autum Burke
- Kira Wayman
- Amber Roldan
- Bridget Curry
- Christine Luckhoo
- Kailey LaBlanc

Tara Banning
Night Team Leader/Lims

2nd Shift Analysts:
- Anvar Sheikh
- Greg Mercier
- Dustin Harrison
- Michelle Lopes
- Tamara Rodriquez
- Edgar Cortez
- Chamyra Washington
- Dylan O'Hagen
- Javaun Murphy

3rd Shift Analysts:
- Rowena Wagner
- Aleisa Price
- Megan Ortriquerra Rojas
- Brad Foster

Microbiology Department

Kathleen Cressia
Micro Lab Director

Rashmi Makol
Micro Team Leader

Lauren Johnson
Analyst

Gabby Sweeney
Analyst

Dylan Tillman
BOD Analyst

Classical Chemistry

Eric Geyer
Team Leader

1st Shift Analysts:
- Greg Danielewski
- Kandi Della Bella
- Jean Rawiling
- Matt Fijolek
- Blake Antil
- Queenie Hsiao
- Angelica Martinez
- Daniel Kinney

Meredith Weigert
Night Team Leader

2nd Shift Analysts:
- Brian Sheridan
- Michael Tran
- Elizabeth Rideout
- John Mark Woodworth
- Praveena Kirshnaprasad
- Catherine Lundigan
- Mary LaValle

Phoenix Environmental Laboratories, Incorporated

Quality Manual

Appendix D

Standard Operating Procedure Table of Contents

Phoenix Environmental Laboratories
SOP Table of Contents

Version: 81
Date: July 2022

SOP No.	SOP Title/Comment	Dist. #	Previous Version	Current Version	Date Finalized
Sampling					
101.0	Drinking Water Sampling Procedure		3 (12/3/09)	4	10/28/2015
102.5035	Soil Prep for VOA 8260		2.2 (9/23/19)	2.3	12/12/2019
103.0	Sample Acceptance Policy	1	1 (10/21/08)	2	2/16/2015
104.Temp	Temperature		0 (4/19/05)	1	6/6/2007
105.5030	Water Prep for VOA 8260		1 (7/25/13)	1.1	10/20/2016
106.Air	Air Sampling			O	10/3/2019
107.IR	Temperature at Sample Receipt			O	11/15/2019
108	Chlorine Residual			O	3/2/2021
121.0	Sample Container Preservation	1	8 (10/9/12)	8.1	3/18/2015
Sample Preparation					
203.SONC	Sonication Extractions		10.2 (5/1/19)	10.3	2/15/2021
204.552.2	Haloacetic Acids		8.1 (1/20/17)	8.2	6/27/2022
205.TMD.DISS	Metals Digestion-Dissolved		5.4 (6/12/19)	5.5	7/6/2022
206.paint filter	Paint filter free liquids test		2 (6/21/12)	3	11/15/2019
208.EPH	Extractable Petroleum Hydrocarbons		6.7 (7/27/21)	6.8	6/14/2022
213.sep508	Separatory extraction PCB 508		3.3 (1/12/18)	3.4	5/11/2022
214.515.3	Herbicide Ext of Drinking Water		5 (12/2/13)	5.1	3/21/2017
217.Sep Herb	Herbicide ext by methylation		4.3 (2/5/20)	4.4	7/26/2022
219.TMD.dw	Metals Digestion Drinking Water		6 (12/27/16)	6.1	7/27/2017
220.Form	Formaldehyde		3.1 (11/6/17)	3.2	7/11/2022
224.TMD.wm	Metals Digestion Wastewater Matrix		10 (1/4/17)	10.1	7/23/2019
226.wastedilutions	Waste dilutions for oil matrix		4.2 (4/22/15)	4.3	2/15/2021
231.TMD.sm	Metals Digestion in Soils/Wastes		6.1 (4/22/15)	6.2	7/28/2017
234.%sol	% Solids		1 (3/22/00)	2	4/11/2006
235.ASE-SM	Soil Extraction by PFE		9.4 (3/4/21)	9.5	7/14/2021
236.HGSM	Mercury digestion (soil matrix)		3.4 (5/13/21)	3.5	4/11/2022
237.HGWM	Mercury digestion (water matrix)		5.3 (5/13/21)	5.4	5/3/2022
238.TMD.3051A	Microwave digestion metals SM/Oils		2.3 (1/27/16)	2.4	2/26/2016
239.sepext	Separatory extractions (water matrix)		6.4 (9/23/19)	6.5	2/12/2021
240.liq/liq	Continuous liquid-liquid extraction		12.2 (9/23/19)	12.3	2/12/2021
242.TCLP	Toxicity Characteristic Leaching		1.2 (4/3/15)	1.3	7/17/2019
243.SPLP	Synthetic Precipitation Leaching		0 (11/15/01)	1	1/21/2010
245.SepSIM	Separatory extraction WM SIM		1 (1/11/2006)	1.1	1/10/2019
246.sox Wipes	Soxhlet extraction of wipes		3 (11/12/13)	3.1	1/25/2017
247.Soncherb	Sonication Ext for Herbicides		6 (1/10/20)	6.1	7/7/2022
250.ASE care	ASE cell cleaning procedure		3 (6/9/14)	4	12/31/2014
251.Soxhlet	Soxhlet Extraction procedure		4 (11/12/13)	4.1	11/17/2016
253.Baking Chem	Baking Chemicals			O	7/7/2008
255.ASE-SM SV-SIM	Semivolatiles in Soil by SIM		O (3/19/09)	1	2/23/2017
258.ZHE Clean	ZHE Cleaning Procedure	1		O	1/3/2011
259.PUFsoxhlet	Soxhlet Extraction for PCB Air-PUF		1 (8/19/11)	2	11/12/2013
260.HgDW	Mercury digestion (drinking water)		1 (3/22/17)	1.1	5/3/2022
261.525.3	Preparatory SPE 525.3		1.1 (7/14/16)	1.2	11/3/2021
262.Carbo	Carbo cleanup for Pests		1 (2/25/2020)	1.1	6/27/2022
263.ASTMext	ASTM extraction			O	8/1/2019
264.Dioxane	SPE for 1,4-Dioxane			O	1/2/2020
265.3546	Microwave digestion organic soils		2.1 (3/4/21)	2.2	7/14/2021
266.sox shutdown	Soxhlet shutdown for weekends			O	9/30/2020
267.concentrations	Sample concentrations		O (5/2/22)	1	8/3/2022
Wet Chemistry					

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301.IC.DX120	Ion Chromatography DX120		5.4 (1/27/21)	5.5	4/15/2022
302.Lachat	Lachat Autoanalyzer		3 (1/26/05)	4	3/12/2007
303.2310B	Acidity		1.1 (12/18/14)	1.2	3/7/2018
304.4500NH3 G	Ammonia/TKN		8.4 (7/26/17)	8.5	7/26/2019
305.2320B	Alkalinity		6 (6/11/07)	6.1	3/12/2018
306.5210B	BOD/cBOD		8.7 (7/26/19)	8.8	12/17/2021
307.4500CL G	Chlorine		3.1 (11/6/19)	3.2	5/27/2022
308.2510 B	Conductivity		5 (6/21/12)	5.1	3/12/2018
309.335.4/4500CN	Cyanide-Total, Amenable & Free		10.2 (12/17/21)	10.3	4/22/2022
310.2120 B	Color		4 (2/5/15)	4.1	3/22/2018
311.5220 D	COD		4 (7/19/12)	4.1	3/12/2018
312.4500 O G	DO electrode		2.1 (1/29/18)	2.2	6/5/2019
313.1010	Flashpoint		5 (4/28/21)	5.1	4/29/2022
314.3500 Cr B	Hexavalent Chromium WM		4.3 (3/14/18)	4.4	8/19/2022
315.3060A	Hexavalent Chromium SM		7.2 (4/20/22)	7.3	8/25/2022
316.5540 C	MBAS		2.3 (8/22/19)	2.4	5/20/2022
317.2150 B	Odor		5 (11/13/12)	6	9/20/2013
318.1664	Oil & Grease		8.2 (7/8/22)	8.3	8/19/2022
319.SM4500H+B	pH and Corrosivity		5.1 (1/17/17)	5.2	3/15/2018
320.420/9066	Phenols		5.1 (2/3/15)	5.2	6/27/2022
321.4500P E	Phosphorus		5.2 (3/15/18)	5.3	7/12/2022
322.React	Reactivity		2 (9/10/09)	2.1	9/30/2016
323.2540 C	Solids, Dissolved (TDS)		3.1 (3/22/18)	3.2	1/10/2022
324.2540 D	Solids, Suspended (TSS)		5.2 (8/22/19)	5.3	1/10/2022
325.2540 B	Solids, Total (TS)		3.1 (3/22/18)	3.2	1/10/2022
326.9030	Sulfide, Total (distil followed by Titr.)		2.1 (1/12/17)	2.2	1/10/2020
330.2130 B	Turbidity (NTU)		3 (12/27/06)	3.1	3/16/2018
331.2540E	Solids, Fixed & Volatile (FS/VS)		2.0 (3/22/18)	2.2	3/17/2022
332.2350B	Chlorine Demand		1 (4/5/99)	2	3/22/2000
336.353.2	Nitrate by Lachat		3.3 (6/9/17)	3.4	7/26/2022
339.377.1	Sulfite		O (5/5/00)	1	9/7/2016
340.2520 B	Salinity		1 (7/1/03)	2	3/22/2006
341.SO4grav	Sulfate, gravimetric		1 (4/5/99)	2	4/2/2001
343.4500-S2 D	Sulfide, Total (colormetric)		6 (4/1/15)	6.1	8/15/2022
344.TOCSM	TOC soil (sm)		2.3 (12/3/2019)	2.4	6/12/2020
345.FI2	Fluoride by electrode		3.3 (4/29/22)	3.4	8/17/2022
346.4500Cl-E	Chloride Automated Ferricyanide		3 (2/28/07)	3.1	3/16/2018
347.VFA	Volatile Fatty Acids		1 (1/13/03)	2	10/4/2011
352.CO2	Free Carbon Dioxide			O	11/30/2005
353.AVS/SEM	Acid Volatile Sulfide/SEM metals			O	11/6/2004
354.cyanate	Cyanate by NH3 probe			1	2/16/2007
355.OP Lachat	Orthophosphate Lachat		1 (2/23/07)	1.1	3/16/2018
356.5910B	UV-254		3 (5/12/12)	3.1	9/23/2019
357.2540F	Settleable Solids		1 (3/21/18)	1.1	2/12/2022
358.MetalsDW	Phoenix Metals DW procedure		O (8/14/07)	1	4/26/2013
359.4500CN WAD	Weak & Dissociable Cyanide			O	10/31/2007
360.PCT	PC Titrator (pH, Alk, Cond, Turb)		2 (6/21/12)	2.1	3/7/2018
361.SpecGrav	Specific Gravity			O	5/11/2011
362.9071B	Oil & Grease in Soil / Solids		1 (4/7/15)	1.1	5/9/2018
363.5310B	TOC Lotix by 5310B		1.2 (4/22/22)	1.3	8/19/2022

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Bacteria					
401.E.Coli MF-DW	E.coli MF in DW		7.2 (5/23/19)	8	2/1/2022
403.9222D	Fecal coliform MF		5.8 (1/10/22)	5.9	9/2/2022
404.FecalMPN	Fecal Coliform MPN			1.1	5/23/2019
406.9215B	Heterotrophic Plate Count		8 (6/28/12)	8.1	7/26/2017
407.9223B	Total coliform DW by Colilert		7.1 (2/14/17)	7.2	5/23/2019
408.9222B	Total coliform MF		8.4 (7/31/19)	8.5	2/1/2022
410.TColiQ/EColiQ	Total coliform Colilert MPN		1.1 (5/23/19)	1.2	1/12/2022
411.Enterolert	Enterococcus MPN		1.2 (5/23/19)	1.3	3/3/2022
412.FecalQ	Fecal coliform Colilert MPN		O (2/2/17)	1	9/27/2017
413.SRB	Sulfate Reducing Bacteria			1	9/27/2017
414.m-ColiBlue24	Total Coliform/E.Coli MF		O (11/4/20)	1	1/4/2022
450	Disposal of sample cultures		4 (5/6/09)	4.1	5/23/2019
451	Cleaning of UV equipment		0 (6/21/01)	1	3/20/2009
452	Autoclave sterility check		5.2 (2/13/18)	5.3	11/26/2019
454	Air Monitoring		0 (6/25/01)	1	5/8/2009
455	Sample volume adjustment Procedure			O	9/26/2022
457	UV Box Check		1 (5/14/07)	2	5/6/2009
458	InHouse DI Water Monitoring		O (12/28/09)	1	2/18/2016
Metals					
501	Metals by GFAA		6.5 (1/10/20)	6.6	6/30/2022
503	Mercury by CV		8.4 (4/13/22)	8.5	6/28/2022
506	Metals by ICP		1.4 (1/10/20)	1.5	3/31/2020
507	Hardness by Calculation		3 (2/18/2016)	3.1	9/9/2021
508	Metals by ICP-MS		1.5 (4/8/22)	1.6	7/6/2022
Organic Instrumentation					
601.8270/625.1	SVOA by GC/MS		12.4 (6/12/18)	12.5	7/26/2019
602.624.1	VOA by GC/MS		9 (11/7/2017)	9.1	4/10/2018
603.552.2	Haloacetic Acids		8 (2/17/15)	8.1	9/17/2019
605.CTETPH	CT ETPH by GC/FID		5.1 (1/19/16)	5.2	3/4/2021
611.504/8011	EDB, DBCP		9 (1/4/19)	9.1	9/14/2022
613.508	PCB in drinking water		5.1 (5/10/16)	5.2	1/12/2018
614.515.3	Herbicide in drinking water		9.1 (3/8/17)	9.2	9/28/2022
617.531.2	Carbamates by HPLC		5.3 (9/4/18)	5.4	6/15/2019
619.EPH	EPH by GC/MS		4 (3/10/20)	4.1	2/12/2021
620.VPH	VPH by GC/MS		4.1 (9/30/16)	4.2	11/3/2021
621.8081	PESTS by GC		6.7 (5/13/19)	6.8	8/28/2019
622.8082	PCB by GC		10.3 (4/20/17)	10.4	12/7/2017
626.8141	OP Pesticides		2 (8/6/13)	2.1	11/13/2014
627.8151	Herbicides		8.1 (10/28/16)	8.2	2/11/2021
630.680	PCB's by 680		4.2 (2/7/2017)	4.3	4/10/2018
633.Glycol	Glycols		4.2 (2/16/17)	4.3	5/16/2017
634.DRO	Diesel Range Organics		5.2 (1/19/16)	5.3	3/4/2021
635.GRO	Gasoline Range Organics		6 (9/29/16)	6.1	6/4/2019
640.NJ TPH	NJ QAM-025		1 (6/26/2015)	1.1	1/20/2016
642.FORM	Formaldehyde HPLC		3 (3/26/14)	3.1	3/20/2017
643.Alcohol	Alcohols FID headspace		4.1 (5/3/16)	4.2	6/28/2019
644.SV-SIM	SVOA by Selective Ion Monitoring		3 (11/3/14)	3.1	2/2/2016
645.TO14-15	VOCs in Air		5.1 (3/17/17)	5.2	7/12/2017

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646.1,4-dioxane	1,4-Dioxane		2.4 (6/12/19)	2.5	1/2/2020
647.NJLLTO-15	NJ Low Level TO-15 Air		4.7 (9/24/21)	4.8	11/4/2021
650.MA APH	Air-Phase Petroleum Hydrocarbons			O	11/3/2016
651.524.2	Volatiles in DW by 524.2 5mL		3.1 (3/10/17)	3.2	9/28/2022
652.NJEPH	New Jersey EPH		1.9 (6/18/21)	2	9/30/2021
653.8260C/D	VOA by GC/MS		2.3 (7/8/19)	2.4	12/12/2019
654.549.2	Diquat & Paraquat		1.2 (6/4/19)	1.3	7/26/2022
655.547	Glyphosate by 547		1 (1/8/16)	1.1	3/20/2017
656.525.3	525.3		1.3 (3/21/17)	1.4	11/3/2021
657.Fluridone	Fluridone by 525.3			O	10/24/2017
658.608.PEST	Pesticides by 608.3		O (11/3/17)	1	8/28/2019
659.608.PCB	PBC by 608.3			O	11/3/2017
General					
701	Glassware Cleaning		1 (2/19/99)	2	8/17/2011
702	Laboratory Nonconformance		1 (7/23/99)	2	12/5/2012
703	General Waste Disposal		2 (12/3/09)	2.1	6/12/2019
704	Final Report Review		1 (2/13/01)	2	12/6/2012
705	Significant Figures and Rounding			1	9/11/2000
706	Eliminating Transcription/Calc Errors			O	11/20/2002
707	Transmission of Test Results			O	1/4/2002
708	Avoid Deterioration/Damage			O	1/4/2002
709	Raw Data Review			1	12/9/2002
710	Sample Log-in		1 (2/12/01)	2	8/24/2012
711	Purchasing			O	1/24/2005
712	Decon sampling equipment			O	12/15/2006
713	SOP update procedure		O (4/4/11)	1	11/4/2014
714	Manual Integration Policy		1.1 (3/23/16)	1.2	5/11/2017
715	Stocks & Standards Tracking				DRAFT
716	Exceedance Notifications		1.1 (9/13/17)	1.2	9/23/2019
717	Data Integrity Plan			O	5/10/2017
718	MDL Determination			O	11/15/2019
Safety					
801	Employee Right to Know	1	1 (8/21/00)	2	12/8/2009
802	Emergency Evacuation Plan	1		1	6/10/2004
803	Laboratory Hood	1		1	8/22/2000
804	Safety Committee	1	1 (8/22/00)	2	7/9/2003
805	Hazardous Chemical Procedures	1		1	1/8/2003
806	Laboratory Safety Equipment	1	1 (7/7/04)	2	6/2/2006
807	Chemical Hygeine Plan	1	2 (2/11/10)	3	3/25/2013
808	Spill Control Procedures			O	4/27/2022
809	First Aid Procedures	1		1	9/13/2000

PHOENIX
Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
Telephone: 860.645.1102 • Fax: 860.645.0823

SOP Number: 605.CTETPH

ANALYSIS OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS (ETPH) USING METHYLENE CHLORIDE GAS CHROMATOGRAPH/FLAME IONIZATION

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for analysis of extractable petroleum hydrocarbons (ETPH) in soil and water sample (i.e. surface and ground water). The conditions used are designed to measure the C_9 to C_{36} range of hydrocarbons. This range represents the major components of a number of widely used petroleum products such as kerosene, jet and diesel fuels, No. 2 and No. 6 fuel oils and motor oil. This method is not used for quantitation of gasoline contamination, because the major components of gasoline are not retained in the sample extraction and concentration procedure.

1.2 The average response factors of C_9 to C_{36} alkanes and total peak area of the sample chromatogram are used to calculate the concentration of ETPH. An N-alkane mixture is run as a performance and calibration verification to ensure GC conditions are adequate to perform ETPH analysis. This also ensures that the initial calibration meets QC criteria. Capillary GC columns are recommended for the analyses.

2.0 SUMMARY OF METHOD

2.1 This method uses a gas chromatograph coupled with a flame ionization detector (FID) for the analysis of ETPH. Semi-volatile organic compounds other than petroleum hydrocarbons, which can be extracted and detected by FID, will be calculated as part of ETPH.

2.1.1 Soil and solid samples are extracted with methylene chloride and acetone using an Accelerated Solvent Extractor (ASE), or by Microwave Digestion using Mars2.

2.1.2 Water samples are extracted with methylene chloride using a continuous liquid-liquid extractor.

2.2 A Restek RXi-5MS column and temperature program, which can separate the solvent peak from C_9 alkane and is able to elute the last component,

C_{36} alkane, in a reasonable time period (about 30 minutes), is used. Detection is achieved by FID.

2.3 Oil identification can be achieved by comparing the chromatograph of the sample to that of the known product.

3.0 INTERFERENCES

3.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe must be rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a solvent blank to check for cross contamination.

3.2 Those organic compounds that are extracted, eluted through the column and detected by FID may be calculated as ETPH. When interferences are present in significant amounts, irregular chromatograms may be observed.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatographs -- Perkin Elmer Autosystem.

4.2 Gas chromatograph software -- Perkin Elmer Turbochrom 4.

4.3 Capillary Column (Restek Rxi-1ms Catalog number 13340) -- 30 m x 0.53 mm inner diameter fused silica capillary column, 0.5 um film thickness.

4.4 Detector -- flame ionization detector (FID)

4.5 Sample introduction -- a split/splitless, Direct or on-column GC injection port for the analysis of solvent extracts by direct injection. Use Phenomenex Uniliner Part #AGO-4665; Inlet sleeve 4mm split. Hand pack a small amount of glass wool.

4.6 Vials -- Autosampler, 2 mL vials with teflon septa

4.7 Pasteur Pipettes -- Glass, disposable. 5 3/4", 9"

4.8 Micro Syringes -- 10 uL, 25 uL, 50 uL, 100 uL, 250 uL, 500 uL, and 1000 uL

5.0 REAGENTS

5.1 Solvents

5.1.1 Methylene chloride – J.T. Baker, Ultra Resi-Analyzed.

5.2 Standards

5.2.1 Fuel oil standard – Fuel #2 (ultra RGO-616) at a concentration of 50,000ug/ml.

5.2.2 N-Hydrocarbon Standard- Absolute 91488 (14 analytes at 2000ug/ml)

5.2.3 Continuing calibration standard= Level 5 standard plus 50uL of surrogate stock solution, 5.2.6. True value = 700ug/ml, surrogate concentration, 50ug/mL.

5.2.4 Calibration verification standard (CVS)= 500uL Ultra #RGO-616 into 10mL methylene chloride. True value = 2500ug/mL.

5.2.5 Calibration standards -- A six level curve is made by dilutions of the n-Hydrocarbon standard in section 5.2.2.

Level	Vol. of Std (uL)	Collective Conc. (ug/mL)	Final Volume (mL)
1	15	42	10
2	25	70	10
3	50	140	10
4	100	280	10
5	250	700	10
6	350	980	10

5.2.6 Surrogate standard – Restek 577205 Custom EPH Surrogate Standard, 1-chlorooctadecane and o-Terphenyl at 10,000ug/ml

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 General guidelines for semi-volatile sampling are applicable when sampling for ETPH. Soil and solid waste samples can be split off from samples taken for other semi-volatile analyses. See Table 1.0.

7.0 PROCEDURE

7.1 Sample extraction -- See Prep Laboratory SOP
7.2 Chromatographic conditions

7.2.1 Column 1

Carrier gas (helium) flow rate:	9.0 mL/min
Oven temperature program:	
Initial temperature:	40°C, hold for 2 minutes.
Program:	15 °C/min to 330 °C.
Final temperature:	330 °C, hold for 2 minutes.
Injector temperature:	350 °C
Detector temperature:	350 °C
Attenuation:	2
Split valve:	Off at injection
Injection volume:	1.5uL
Injection speed:	Fast

7.2.2 Performance Verification

This method is based on the fact that the response factors of hydrocarbons are essentially the same, provided the GC system does not have discrimination when the sample is introduced. The method relies on using the average response factor of alkanes to convert the total peak area of a sample chromatogram to a ETPH concentration. It is critical to maintain the gas chromatograph without significant sample introduction discrimination. The N-alkane mixture also serves other functions a) to demonstrate sufficient separation between the solvent and C_9-alkane peak, b) to ensure that the last component, C_{36}, is eluted within a reasonable amount of time (30 minutes), and c) to serve as a retention time marker for oil identification. Performance is verified at the beginning of a GC analysis batch and whenever any changes are made to the system or operational parameters. Analyze the N-alkane mixture (C_9 to C_{36}) using the identical procedure to be used for the analysis of samples. Calculate the response factor for each N-alkane in accordance with the equation below:

$$Fi = Ai/Ci$$

Where: Fi = response factor of an individual alkane

Ci = the mass (ug) of the alkane injected into the column.
Ai = response in area counts for the individual alkanes.

Use the following equation to calculate the average response factor of N-alkanes:

$$Fa = (\Sigma Fi)/n$$

Where: Fa = the average response factor.
n = the number of the alkanes used in the calculation

The deviation of an individual response factor to the average response factor is calculated using the following equation:

$$\%D = [(Fi-Fa)/Fa] \times 100$$

If the response factors for the alkane standards are not within +/- 20%, then a new initial calibration must be prepared for a standard mix. Common causes of sample introduction discrimination include an injection liner that is dirty or has an activated surface, inappropriate column installation to the injection port, incorrect sample injection (especially when using the manual injection technique), dirty column, or an injection port leak. Performance failure may result when the N-alkane mixture is not completely dissolved in the solvent. It is very important to maintain the integrity of the N-alkane solution.

7.3 Initial Calibration

7.3.1 Use the N-alkane mixture at different concentrations (concentration equaling the sum of individual alkane concentrations). The concentrations should correspond to the expected range found in real samples or should cover the working range of the detector.

7.3.1 Introduce each calibration standard by matching the technique used to introduce the actual samples into the gas chromatograph. Tabulate peak area responses under the resolved peaks against the mass injected.

7.3.2 Calculate the average response factor according to section 7.2.3 for each concentration level.

7.3.3 Calculate the average and relative standard deviation of response factors over the five concentrations. If the percent relative standard deviation (%RSD) of the response factor is less than 30% over the working range, linearity through the origin can be assumed, and the average response factor can be used in calibration.

7.4 Quantitation Range

7.4.1 The retention time range for a particular fuel is defined during initial calibration. The retention time range is the period between the mean retention time of the initial rise of the first major eluting peak and the mean of the final descent of the last major eluting peak in the fuel pattern. Major peaks are at least 10% of the height of the largest peak in the fuel pattern.

7.4.2 The retention time range for an unknown sample is defined during initial calibration using the range shown by an nC_9-nC_{36} standard.

7.5 Calibration Verification

7.5.1 The response factor and retention times must be verified at the beginning of each 12-hour work shift as a minimum requirement. Verification is accomplished by analysis of an N-alkane standard that falls in the middle of the range of concentrations chosen. It is strongly recommended that additional analyses of the verification standard(s) be run throughout a 12-hour shift.

7.5.2 If the average response for the standard is within +/- 30% of the response obtained during the initial calibration (using initial response factor as 100%), then the initial calibration is considered still valid. If the response factor varies from the predicted response by more than +/- 30% in these additional determinations, corrective action must be taken to restore the system or a new calibration curve must be prepared.

7.5.3 All N-alkanes in the calibration verification analyses must fall within previously established retention time windows.

7.5.4 Solvent blanks and any method blanks should be run with calibration verification analyses to confirm laboratory contamination does not cause false positives.

7.6 Gas Chromatographic Analysis

7.6.1 The sequence begins with a solvent blank followed by performance/calibration verification standard(s), then method blank and finally the sample extract analyses. A verification standard is also necessary at the end of each analytical batch. The sequence ends when the entire set of samples has been injected or when retention time and/or percent difference QC criteria are exceeded.
Note: If the criteria are exceeded, inspect the gas chromatographic systems to determine the cause and perform whatever maintenance is necessary before recalibrating and proceeding with sample analysis. All sample analyses performed using external standard calibrations must be bracketed with acceptable data quality analyses (e.g., calibration and retention time criteria). Therefore, all samples that fall between the standard that exceed criteria and the last standard that was acceptable must be reanalyzed.

7.6.2 Samples are analyzed with the same instrument configuration as is used during calibration. The same sample extract is split into two autosampler vials. The second vial is stored for 24 hours to ensure that an uncompromised sample is available for analysis or dilution, if the analysis of the first sample is unsuccessful or if results exceed the calibration range of the instrument.

7.6.3 Sample concentrations are calculated by comparing sample response data with the initial calibration (section 7.3). Therefore, if sample response exceeds the limits of the initial calibration range, a dilution of the sample must be analyzed. Extracts should be diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, is acceptable as long as calibration limits are not exceeded.

7.6.4 Second column confirmation is generally not necessary for petroleum hydrocarbon analysis. However, if analytical interferences are indicated, analysis using another method is required.

7.6.5 The performance of the entire analytical system should be checked every 12 hours, using data gathered from analyses of

blanks, standards, and replicate samples. Significant peak tailing must be corrected.

7.6.6 Instrument Maintenance

Injection of samples from waste sites often leave a high boiling point residue in the injection port are or the injection port end of the chromatographic column. Such samples may also splatter. This residue affects chromatography in many ways (i.e., peak tailing, retention time shifts, analyte degradation, etc.). In addition, residue buildup in a splitter may limit flow through one leg and therefore change the split ratios. If this occurs during an analytical run, the quantitative data may be incorrect. Instrument maintenance is therefore very important. Proper cleanup techniques will minimize the problem. Instrument maintenance is required.

8.0 DATA ANALYSIS AND CALCULATIONS

8.1 To measure the total area of the samples, construct the baseline of the sample chromatogram, so that it starts at the beginning of the n-C_9 alkane peak and terminates at the end of the n-C_{36} peak. Evaluate the total peak area above the baseline to make sure it reasonably represents the sample response. It may be possible to achieve complete peak integration by using software-specific automatic integration parameters. If this cannot be done, manual integration may be necessary.

8.2 Baseline subtraction may generate better integration results, especially when a sample concentration is low or when column bleed contributes significantly to the baseline. Baseline subtraction may be performed directly on the gas chromatograph or with the data acquisition software that runs the GC.

8.3 The concentration of ETPH of a sample is calculated as follows:

8.3.1 Aqueous samples

Concentration (ug/L) = [(Ax/Fa)(Vt/Vi)D]/Vs

Where:

Ax = response for the analyte in the sample, in total peak area counts
Fa = average response factor of alkane standard, count/ug
Vi = volume of extract injected into GC in mLs

D = dilution factor, if not dilution is made, D=1
Vt = volume of extract in mLs
Vs = volume of sample extracted in L

8.3.2 Nonaqueous and Solid Samples

Concentration (ug/kg) = [(Ax/Fa)(Vt/Vi)D]/W

Where:
W = weight of sample extracted, kg. The wet weight or dry weight may be used, depending upon the specific applications of the data.
Note: the injection volume of sample extract must be the same as that of the calibration standard.

9.0 QUALITY CONTROL

9.1 Each analyst is responsible for following the quality assurance / control outlined in the Phoenix Quality Assurance Plan.

9.2 Performance Check Standard (PCS)

At the beginning of an analysis set, a calibration standard must be analyzed. This check standard insures proper performance of the GC by evaluation of the instrument parameters of detector sensitivity, peak symmetry, and peak resolution. It furthermore serves as a check on the continuity of the instrument's performance. In regards to sensitivity, it allows the analyst to ascertain that this parameter has not changed drastically since the analysis of the MDL study. Inability to demonstrate acceptable instrument performance indicates the need for re-evaluation of the instrument system.

If column or chromatographic performance cannot be met, one or more of the following remedial actions should be taken. Break off approximately 1 m of the injector end of the column and re-install, install a new column, adjust column flows or modify the oven temperature program.

9.3 Initial Demonstration of Capability (IDC)

9.3.1 Calibrate for each analyte of interest as specified in Section 7. Select a representative fortification concentration for each of the target analytes. Prepare four to seven replicates laboratory

fortified blanks by adding an appropriate aliquot of the primary dilution standard or quality control sample to reagent water.

9.3.2 Calculate the mean percent recovery and the standard deviation of the recoveries. For each analyte, the mean recovery value, expressed as a percentage of the true value, must fall in the range of 80-120% and the relative standard deviation should be less than 20%. For those compounds that meet these criteria, performance is considered acceptable and sample analysis may begin. For those compounds that fail these criteria, this procedure must be repeated using four to seven fresh samples until satisfactory performance has been demonstrated. Maintain these data on file to demonstrate initial capabilities.

9.3.3 Furthermore, before processing any samples, the analyst must analyze at least one laboratory reagent blank to demonstrate that all glassware and reagent interferences are under control.

9.3.4 The initial demonstration of capability is used primarily to preclude a laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it. As laboratory personnel gain experience with this method, the quality of data should improve beyond those required here.

9.3.5 The analyst is permitted to modify GC columns, GC conditions, internal standard or surrogate compounds. Each time such method modifications are made; the analyst must repeat the procedures in Section 9.3.

9.4 Method Detection Limit Study (MDL)

9.4.1 Prior to the analysis of any field samples, the method detection limits must be determined. Initially, estimate the concentration of an analyte that would yield a peak equal to five times the baseline noise and drift. Prepare seven replicate LFBs at this estimated concentration with reagent water that contains ammonium chloride at the same concentration as that specified for samples. Analyze the LFBs according to the method.

9.4.2 Calculate the mean recovery and the standard deviation for each analyte. Multiply the student's t value at 99% confidence and n-1 degrees of freedom (3.143 for seven replicates) by this standard

deviation to yield a statistical estimate of the detection limit. This calculated value is the MDL.

9.4.3 Since the statistical estimate is based on the precision of the analysis, an additional estimate of detection can be determined based upon the noise and drift of the baseline as well as precision. This estimate is the EDL.

9.5 Laboratory Preparation Blanks (PB) -- Each time a set of samples is extracted or reagents are changed, a LRB must be analyzed. If the PB produces an interferent peak within the retention time window of any analyte that would prevent the determination of that analyte or a peak of concentration greater than the MDL for that analyte, the analyst must determine the source of contamination and eliminate the interference before processing samples. Field samples of an extraction set associated with a PB that has failed the specified criteria are considered suspect. Note: Reagent water containing ammonium chloride at the same concentrations as in the samples is used to prepare the PB.

9.6 Laboratory Control Sample (LCS) – With each batch a LCS is extracted and analyzed. The recovery requirement is 60-120%

9.7 Laboratory Fortified Sample Matrix (MS / MSD)

9.7.1 The laboratory must add known concentrations of analytes to one sample per extraction set or a minimum of 10% of the samples, whichever is greater. The concentrations should be equal to or greater than the background concentrations in the sample selected for fortification. If the fortification level is less than the background concentration, recoveries are not reported. Over time, samples from all routine sample sources should be fortified.

9.7.2 Calculate the mean percent recovery, R, of the concentration for each analyte, after correcting the total mean measured concentration, A, from the fortified sample for the back-ground concentration, B, measured in the unfortified sample, i.e.: R = 100 (A - B) / C, where: C = the fortifying concentration In order for the recoveries to be considered acceptable, they must fall between 50% and 150% for all the target analytes.

9.7.3 If a recovery falls outside of this acceptance range, a matrix induced bias can be assumed for the respective analyte and the

data for that analyte must be reported to the data user as suspect due to matrix effects.

9.8 Quality Control Sample (QCS) -- At least quarterly, analyze a QCS from an external source. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem source. The LCS meets this requirement because it is a separate source from the standards. Also the proficiencies are used to monitor performance.

10.0 SAFETY

10.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be minimized. The laboratory has a safety manual and MSDS volumes located in the general laboratory. It is important to read and understand these as well as the chemical hygiene plan.

11.0 METHOD PERFOMANCE

11.1 Control charts are maintained for LCS and MS/MSD recovery.

12.0 POLLUTION PREVENTION

12.1 This method utilizes a micro-extraction procedure that requires the use of very small quantities of organic solvents. This feature reduces the hazards involved with the use of large volumes of potentially harmful organic solvents needed for conventional liquid-liquid extractions. This method also uses acidic methanol as the derivatizing reagent.

12.2 For information about pollution prevention that may be applicable to laboratory operations consult "Less is Better: Laboratory Chemical Management for Waste Reduction" available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

13.0 WASTE MANAGEMENT

13.1 Due to the nature of this method there is little need for waste management. No large volumes of solvents or hazardous chemicals are used. The matrices of concern are finished drinking water or source water. However, the Agency requires that laboratory waste management

practices be conducted consistent with all applicable rules and regulations, and that laboratories protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Also compliance is required with any sewage discharge permits and regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult "The Waste Management Manual for Laboratory Personnel" also available from the American Chemical Society at the address in Section 12.2.

14.0 REFERENCES

14.1 CT DEP, Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) Using Methylene Chloride Gas Chromatograph/Flame Ionization Detection, March 1999

Table 1: Sample Containers, Preservation, Techniques, and Holding Times

Analyte Class	Container	Preservative	Holding Time
Concentrated Waste Samples	125 mL wide-mouth glass with Teflon lined lid	None	Samples must be extracted within 14 days and extracts analyzed within 40 days following extraction.
Water Samples			
No Residual Chlorine Present	1-gal. or 2 x 0.5-gal. or 4 x 1-L, amber glass container with Teflon lined lid	Cool, 4°C	Samples must be extracted within 7 days and extracts analyzed within 40 days following extraction.
Residual Chlorine Present	1-gal. or 2 x 0.5-gal. or 4 x 1-L, amber glass container with Teflon lined lid	Add 3 mL 10% sodium thiosulfate solution per gallon. Cool, 4°C	Samples must be extracted within 7 days and extracts analyzed within 40 days following extraction.
Soil/Sediments and Sludges	250 mL wide-mouth glass container with Teflon lined lid	Cool, 4°C	Samples must be extracted within 14 days and extracts analyzed within 40 days following extraction.

Revision History Log for SOP #605

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
9/16/14	4	Sec. 9.6 and 9.7 had wrong limits per CT RCP	Jeff Bucko	Kathy Cressia	9/16/14
10/22/14	5	Added more info on Std concentrations and columns. Updated Chromatography conditions.	Jeff Bucko	Kathy Cressia	10/22/14
1/19/16	5.1	New column vendor	Jeff Bucko	Keith Aloisa	1/19/16
3/4/21	5.2	Section 2.1.1- added microwave, Section 5.2.2 & 5.2.5 – new standard and volumes used in curve, Section 5.2.6- new surrogate	Adam Werner	Kathy Cressia	3/4/21

Expiration Date

PHOENIX
Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
Telephone: 860.645.1102 • Fax: 860.645.0823

Effective Date: 7/26/19
Version Number: 12.5
Initiated by: Kathy Gessler
Approved by: Kelly Alonzo

SOP NO: 601.8270/8270E/625.1

Title: Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS)

1.0 Scope and Application

This procedure is used to determine the concentration of semi-volatile organic compounds in extracts prepared from many types of matrices including soil, sludge, water and oil. Refer to Table 1 for the standard compound list. Please note that additional compounds may be determined using this method. Consult the appropriate method for additional extractable analytes.

2.0 Summary of Method

2.1 The sample is prepared for analysis by a suitable extraction method.

2.2 The solvent extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of a minimum of two and a maximum of three characteristics of mass/charge (m/z). Quantitative analysis is performed using internal standard techniques with a single characteristic m/z.

3.0 Interferences

3.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

4.0 Sample Collection, Preservation and Storage

4.1 Store the sample extracts at -10°C, protected from light, in sealed vials (e.g., screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa.

5.0 Equipment and Supplies

5.1 Agilent Technologies Gas Chromatograph 5890, 6890, 7890A

5.2 Agilent Technologies Autosampler 7683

5.3 Agilent Technologies Mass Spectrometer 5972, 5973N, 5975C

5.4 Column – Restek Rtx-5MS (Cat # 12623); 30m; 0.25mm ID, 0.25 micron film thickness

5.5 Data system— Hewlett Packard ChemStation and Enviroquant

5.6 Supelco Thermogreen LB-2 11mm Conditioned Septa. Part # 20654

6.0 Reagents and Standards

6.1 Acetone, methanol, methylene chloride—Pesticide quality or equivalent.

6.2 Stock standard solutions – Restek SV Calibration mixes 1-5, 7.

#31007 SV Cal Mix #1 @ 2000 ug/ml
#31008 SV Cal Mix #2 @ 2000 ug/ml
#31009 SV Cal Mix #3 @ 2000 ug/ml
#31010 SV Cal Mix #4 @ 2000 ug/ml
#31011 SV Cal Mix #5 @ 2000 ug/ml
#31013 SV Cal Mix #7 @ 2000 ug/ml
Absolute Standards #98509 Custom Mix @2000ug/ml
Ultra standard #PST-005 Atrazine (neat)
Ultra standard #EPA-1071 Benzidine 5000ug/ml

6.3 Surrogate standard spiking solution— Restek acid Surrogate Mix # 31063 (10,000ug/ml of Phenol-d6, 2-Fluorophenol, 2,4,6-Tribromophenol), and B/N Surrogate Mix #31062 (5000 ug/ml of Nitrobenzene-d5, 2-Fluorobiphenyl, Terphenyl-d14).

6.4 Internal standard – 4000 ug/ml of 1, 4-dichlorobenzene-d4, naphthalene-d8, acenaphthalene-d10, phenanthrene-d10, chrysene-d12, perylene-d12. Approximately 0.4g of each standard from Cambridge Isotope Labs Inc is weighed out and dissolved in 10% carbon disulfide / 90% methylene chloride. See standard log for procedure.

6.5 DFTPP standard mix -Prepare a 50 µg/mL solution of DFTPP in Methylene chloride – Combine 500uL of Ultra GCM-150 , which also includes 4,4'-DDT, Benzidine, and Pentachlorophenol to verify injection port inertness and GC column performance. Bring to a total volume of 10mL with methylene chloride.

6.6 Spiking Solution:
Take 1mL (8270 Supplemental List - 13 Components) Absolute Standard Part #98293, 1mL (CLP - Semi-Volatile Additional Compound Standard) Absolute Standard Part #91760, 1mL (EPA Method 8270A - Analytes Mix #8 phenols) Absolute Standard Part #10018, 1mL (CLP - Semi-Volatiles - Benzidines) Absolute Standard Part #10006, 1mL (CLP - Semi-Volatile Base/Neutral Standard) Absolute Standard Part #91759, and 3mL DCM. Bring to final volume of 40mL with MeOH. This is made for the Prep lab.

6.7 Calibration Verification Standard (CVS) for 625.1: Restek MEGA Mix 31850 @ 1000 ug/mL. Take 200uL of Mega Mix and add it to 3800uL of DCM along with 40uL if ISTD.

7.0 Definitions

7.1 Calibration Standard (CAL)- A solution of procedure analytes used to calibrate the mass spectrometer response.

7.2 Calibration Verification Standard (CVS)- A solution of procedure analytes, from a separate source than the Calibration Standard, used to verify the Calibration Standard.

7.3 Laboratory Control Sample (LCS)- A sample containing known concentrations of analytes that is added prior to sample preparation and then analyzed by the laboratory to demonstrate that it can obtain acceptable identifications and measurements.

7.4 Surrogate Compound- A compound that is not expected to be found in the sample that is added to a sample aliquot before extraction and is measured with the same procedures used to measure sample components. The purpose of a surrogate compound is to monitor procedure performance with each sample.

7.5 Method Blank- An aliquot of DI water or solid reference material that is treated as a sample. It is exposed to all glassware and apparatus, and all procedure solvents, reagents and surrogate compounds. The extract is concentrated to the final volume used for samples and is analyzed the same as a sample extract. The method blank is used to determine if method analytes are present in the lab environment, glassware or reagents.

8.0 Procedure

8.1 Calibration

8.1.1 Internal standard calibration procedure—Six internal standards that are similar in analytical behavior to the compounds of interest are used.

8.1.2 Prepare calibration standards at a minimum of six concentration levels for each parameter of interest by following this recipe:

8.1.2.1 Make a 5000ppm Atrazine standard-- Add 0.05056g Atrazine (Ultra Part #PST-005) to a 10ml volumetric flask, and dilute to a final volume of 10ml.

8.1.2.2 Make a 200ppm Semivolatile Stock Standard:

200 ppm Semivolatile Stock Standard (Mix A)	
Methylene Chloride (DCM)	200 uL
Restek 31007 Mix 1	100 uL
Restek 31008 Mix 2	100 uL
Restek 31009 Mix 3	100 uL
Restek 31010 Mix 4	100 uL
Restek 31011 Mix 5	100 uL
Restek 31013 Mix 7	100 uL
Absolute 98509 Custom Mix	100 uL
5000ppm Atrazine	40 uL
Restek 31062 B/N Surrogate	40 uL
Restek 31063 Acid Surrogate	20 uL

8.1.2.3 Make serial dilutions of Mix A for the Semivolatile Calibration standards. The low standard must be at or below the limit of quantitation:

Semivolatile Mix A Calibration Standards			
Concentration (ppm)	Methylene Chloride (uL)	200ppm Mix A Stock Standard (uL)	Internal Standard (uL)
100	250	250	5
70	325	175	5
50	375	125	5
30	850	150	10
20	450	50	5
10	475	25	5
3.5	1965	35	20
1.0	1990	10	20

8.1.2.4 Make 200 ppm Benzidine (Mix B) Stock Standard:

200 ppm Benzidine Stock Standard (Mix B)	
Methylene Chloride (DCM)	960 uL
Ultra EPA-1071 5000ppm Benzidine	40 uL

8.1.2.5 Make serial dilutions of Mix B for the Benzidine Calibration standards. The low standard must be at or below the limit of quantitation:

Benzidine Mix B Calibration Standards			
Concentration (ppm)	Methylene Chloride (uL)	200ppm Mix B Stock Standard (uL)	Internal Standard (uL)
100	250	250	5
70	325	175	5
50	375	125	5
30	850	150	10
20	450	50	5
10	475	25	5
3.5	1965	35	20
1.0	1990	10	20

8.1.3 Using injections of 1 µL, analyze each calibration standard and tabulate the area response of the primary characteristic m/z against concentration for each compound and internal standard. Calculate relative response factors (RRF) for each compound.

8.1.4 The working initial calibration for a target analyte is acceptable when the %RSD of the relative response factor (RRF) values is less than 20% (for 8270) and 35% (for 625.1) or correlation is greater than 0.99 (for 8270). If the %RSD exceeds 20%, employ a quadratic fit curve not forcing through origin with an inverse of concentration curve fit. The coefficient of determination (R2) of the weighted regression must be greater than 0.92 or the Relative Standard Error (RSE) must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable and the system must be adjusted and re-calibrated. The minimum response factors are evaluated vs table 4 (SW-846 8270D) to ensure the required sensitivity is established. For 8270E, the response factors are used as a guidance only. If >10% of the compounds fail to meet the 20% RSD limits and do not meet 0.99 correlation coefficient curve

limits the system is considered too reactive. The system requires examination, appropriate maintenance and recalibration.

8.1.5 Analytical Conditions:

50°C for 0.5 minutes
Ramp 28°C/min to 265°C
Hold for 0.00 minutes
Ramp 3°C/min to 285°C
Hold for 0.00 minutes
Ramp 25°C/min to 330°C
Hold for 1.35 minutes
Constant flow through column is 1.2 mL/min., split 4:1
Injector Temperture: 265 °C
Auxiliary Temperature: 300 °C

8.2 The working calibration curve should be verified by a second source midpoint standard for 8270. The value determined from the second source check should be 70-130 % of the true value.

8.3 The continuing calibration standard (CCAL) must be verified for the target compounds on each 12 hour clock by the measurement of one or more calibration check standards. For SIM 8270 analysis, the 2.5ppm primary ICAL standard is evaluated, for full scan 8270 analysis, the 30ppm primary ICAL standard is evaluated, and for 625.1 a second source 30ppm is evaluated (special note – for 625.1, the CCAL must be from a 2nd source). For 8270, If the recovered value is less than or equal to 20% then analysis may proceed for those compounds. For 625.1, the 2nd source CVS is evaluated against Table 6, Column #2 termed "Range for Q". See Appendix A in this SOP for Table. If any compounds exceed criteria then the system is considered too reactive and the system requires examination, appropriate maintenance or recalibration. Corrective action may consist of any or all of the following: replace the injection port liner with a new one, replace the gold inlet seal with a new one, cut between 8" and 12" of the inlet end of the column, or re-analyze using a fresh calibration standard. Alternatively, a new calibration curve must be prepared.

8.4 Gas Chromatography/Mass Spectrometry

8.4.1 Add 10uL of internal standards (equivalent to 40 ng/ul) to each 1.0 ml sample extract and mix thoroughly immediately before it is injected into the instrument. This procedure minimizes losses due to absorption, chemical reaction or evaporation.

8.4.2 If the response for any m/z exceeds the working range of the GC/MS system, dilute the extract and reanalyze.

8.4.3 Perform all qualitative and quantitative measurements.

8.4.4 When the extracts are not being used for analyses, store them refrigerated at 4°C, protected from light in screw-cap vials equipped with unpierced Teflon-lined septa. Once the run has been completed, the vials are to be moved to the sample freezer as soon as possible.

8.5 Qualitative Identification

8.5.1 Obtain EICPs for the primary m/z and secondary mass/charge. The following criteria must be met to make a qualitative identification:

8.5.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

8.5.1.2 The retention time must fall within 0.06 RRT units of the retention time of the authentic compound.

8.5.1.3 The guideline the analyst uses for relative peak height responses of the three characteristic masses in the EICPs is 30% of the relative intensities of these masses in a reference mass spectrum.

8.5.2 Structural isomers that have very similar mass spectra and less than 30 seconds difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

9.0 Calculations

9.1 Final Concentration in ppb (µg/Kg):

$$\frac{\text{ug/ml from Chemstation} * \text{Final Volume (mL)} * \text{Dilution Factor} * 1000}{\text{Initial Weight (g)} * \%\ \text{solids}}$$

9.2 Final Concentration in ppb (µg/L):

$$\frac{\text{ug/ml from Chemstation} * \text{Final Volume (mL)} * \text{Dilution Factor} * 1000}{\text{Initial Volume (mL)}}$$

10.0 Quality Control

10.1 Quality Control procedures include the following:

10.1.1 Daily DFTPP Criteria – See Table 3 (SW846-8270D)

10.1.2 Initial calibration criteria see section 8.1.4.

10.1.3 The GC/MS must meet DFTPP tuning verification criteria each 12 hours. For 8270E, daily DFTPP, degradation and peak tailing is not required. It is only required at time of ICAL.

10.1.4 The analytical system must meet calibration verification criteria section 8.1.4 / 8.3 each 12 hours.

10.1.5 GC Column Performance - Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

$$\text{Tailing Factor} = \frac{BC}{AB}$$

Where the peak is defined as follows: AC is the width at 10% height; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. (See Figure 1 for an example tailing factor calculation.) Chemstation has a peak tailing / breakdown feature within the software. To evaluate peak tailing, go to q-edit and go to the desired peak. Under the Chromeval menu, select, "Evaluate Tailing". Integrate the peak and the peak tailing factor will be shown on the screen. If the number exceeds 2, further maintenance is to be performed. Peak tailing need not be evaluated for analyses that only require PAHs. For DDT degradation, go to the DDT peak in Q edit and again, under the Chromeval menu, select Evaluate Degradation. Follow the prompts by 1st evaluating the DDT and second, evaluating the breakdown DDE and DDD. The breakdown should not exceed 20%. If breakdown fails, further maintenance is to be performed. For 625.1, unless DDT and Endrin are to be determined, then evaluation of degradation in not required.

10.2. Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and

glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safe guard against laboratory contamination. Method blanks should contain no compounds over 50% of the reporting limit.

10.3 The laboratory must, on an ongoing basis, spike and analyze a minimum of 5% of all samples to monitor and evaluate laboratory data quality.

10.4 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control, as compared to the working initial calibration.

10.5 The continuing calibration (section 6.6) internal standard responses are evaluated to ensure instrument response remains constant. If the standard changes by a factor of two (-50 to +200%) from the midpoint of the most recent initial calibration the instrument will be inspected and adjustments made as appropriate. The internal standards will be monitored and evaluated in all samples, spikes, blanks and standards to check drifting, method performance and system performance.

10.6 The surrogate recoveries for samples, spikes and blanks will be evaluated and reported versus in house control chart limits, state, or project specific requirements.

10.7 Manual integration is used only when a peak is not identified correctly. This may be a peak next to a similar peak and the software had identified the wrong peak or it may be poorly resolved peaks like benzo(b) and benzo(k). Manual integration can never be used to increase or decrease the area of a response.

10.8 Daily GC/MS Performance Tests

10.8.1 DFTPP performance test—At the beginning of each day, inject 1 µL (50 ng) of DFTPP standard solution. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 3 (SW846-8270D) are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples,

blanks, or standards are analyzed. The tailing factor tests may be performed simultaneously with the DFTPP test. For 8270E, DFTPP is to be performed prior to ICAL and confirm that all the key m/z criteria in Table 3 (SW846-8270E) are achieved.

11.0 Safety

11.1 The toxicity and carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.

11.2 Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures

12.0 Pollution Prevention

12.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

12.2 Reagents and standards should be purchased and/or prepared in volumes consistent with laboratory use to minimize the volume of disposal.

13.0 Waste Management

13.1 It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

14.0 Method Performance

14.1 This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.

14.2 See Section 10.0 Quality Control in this SOP for acceptable limits.

15.0 Corrective Action for Out-of-Control or Unacceptable Data

15.1 Curve Linearity – If standard curve has greater than 10% of analytes greater than 20% RSD, the analyst will make a judgment to see if more

significant injector maintenance is needed, a new column is needed or the ion source needs to be cleaned.

15.2 Daily Calibration – If daily calibration fails criteria as defined in section 8.3, the analyst will make a judgment to see if more significant injector maintenance is needed or a new column installed. In the event that column maintenance is performed again with similar results, a new calibration standard must be run and evaluated against the criteria in section 8.1.4. If the new curve still fails, a new column and/or clean source should be installed.

15.3 Soil Surrogate – If the surrogate fails 30% to 130% for soil samples, the sample must be re-extracted and reanalyzed. If the reanalysis yields better results, the reanalysis is reported, if the sample results are similar, the better of the two runs is reported with a comment stating that surrogates are outside of method criteria however re-analysis yielded similar results. One acid and one base surrogate may fail criteria.

15.4 Water Surrogate - If the base neutral surrogate for water fails 30% to 130%, or the acid surrogate for water fails 15%-110% the sample is re-extracted if possible. If the reanalysis yields better results, the reanalysis is reported, if the sample results are similar, the better of the two runs is reported with a comment stating that surrogates are outside of method criteria however re-analysis yielded similar results. One acid and one base surrogate may fail criteria. The analyst if should let the prep department know if the addition of more acid and/or base will help the surrogate recoveries. In the event that there is no sample for reanalysis, the initial results are reported with surrogates deficiencies noted with a comment.

15.5 LCS/LCSD Criteria for Soils – 30% to 130%

15.6 LCS/LCSD Criteria for Waters – 8270- 30% to 130%, 625.1- 60% to 140%

15.7 MS/MSD Criteria for 625.1 is 60% to 140%

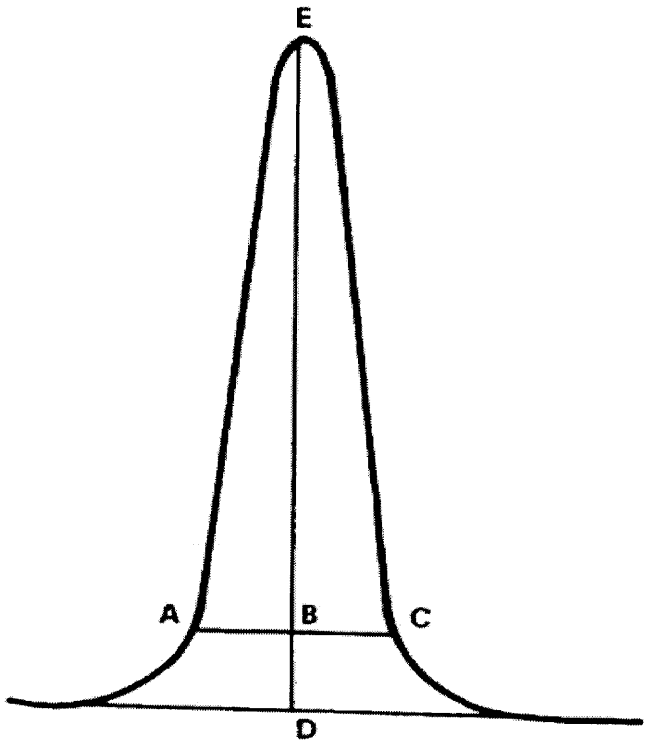
16.0 References

16.1 "Test Methods for Evaluating Solid Waste (SW-846), Fourth Edition, EPA Office of Solid Waste, Final Update IV February 2007 (Method 8270D).
16.2 USEPA Method 625.1 – Base/Neutrals and Acids by GC/MS, December 2014.
16.3 "Test Methods for Evaluating Solid Waste (SW-846), EPA Office of Solid Waste, Update VI, Revision 6, June 2018 (Method 8270E).

Table 1- Analyte List for SW-846 Method 8270 / EPA625.1

Compound	CAS#	Compound	CAS#
Acenaphthene	83329	Di-n-octylphthalate	117840
Acenaphthylene	208968	4,6-Dinitro-2-methylphenol	534521
Aniline	62533	2,4-Dinitrophenol	51285
Anthracene	120127	2,4-Dinitrotoluene	121142
Atrazine	1912249	2,6-Dinitrotoluene	606202
1,2-Diphenylhydrazine(as Azobenzene)	103333	Fluoranthene	206440
Benzaldehyde	100527	Fluorene	86737
Benzo(a)anthracene	56553	Hexachlorobenzene	118741
Benzo(b)fluoranthene	205992	Hexachlorobutadiene	87683
Benzo(g,h,i)perylene	191242	Hexachlorocyclopentadiene	77474
Benzo(k)fluoranthene	207089	Hexachloroethane	67721
Benzo(a)pyrene	50328	Indeno(1,2,3-cd)pyrene	193395
Benzoic Acid	65850	Isophorone	78591
Benzyl Alcohol	100516	2-Methylnaphthalene	91576
Bis(2-chloroethyl)ether	111444	2-Methylphenol (o-Cresol)	95487
Bis(2-chloroethoxy)methane	111911	3-Methylphenol (m-Cresol)	108394
Bis(2-chloroisopropyl)ether	108601	4-Methylphenol (p-Cresol)	106445
Bis(2-ethylhexyl)phthalate	117817	Naphthalene	91203
4-Bromophenyl-phenylether	101553	2-Nitroaniline	88744
Butylbenzylphthalate	85687	3-Nitroaniline	99092
Carbazole	86748	4-Nitroaniline	100016
4-Chloroaniline	106478	Nitrobenzene	98953
4-Chloro-3-methylphenol	59507	2-Nitrophenol	88755
2-Chloronaphthalene	91587	4-Nitrophenol	100027
2-Chlorophenol	95578	N-nitrosodiphenylamine	86306
4-Chlorophenyl-phenylether	7005723	N-Nitroso-di-n-propylamine	621647
Chrysene	218019	Pentachloronitrobenzene	82688
Dibenzofuran	132649	Pentachlorophenol	87865
Dibenzo(a,h)anthracene	53703	Phenanthrene	85018
1,2-Dichlorobenzene	95501	Phenol	108952
1,3-Dichlorobenzene	541731	Pyrene	129000
1,4-Dichlorobenzene	106467	Pyridine	110861
3,3'-Dichlorobenzidine	91941	1,2,4,5-Tetrachlorobenzene	95943
2,4-Dichlorophenol	120832	1,2,4-Trichlorobenzene	120821
Diethylphthalate	84662	2,4,5-Trichlorophenol	95954
2,4-Dimethylphenol	105679	2,4,6-Trichlorophenol	88062
Dimethylphthalate	131113	Benzidine	92875
Di-n-butylphthalate	84742	alpha-Terpineol	98555
Parathion	63653667	2,6-Dichlorophenol	87650
Caprolactam	105602	1-methylnaphthalene	60120
1,4-dioxane	123911		

Figure 1



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Appendix A- EPA 625.1 Acceptance Criteria

Table 6 – QC Acceptance Criteria – Method 625 [1]					
Analyte	**Range for Q (%) [2]**	**Limit for s (%) [3]**	**Range for $\overline{X}$ (%) [3]**	**Range for P, P_s (%) [3]**	**Limit for RPD (%)**
Acenaphthene	70-130	29	60-132	47-145	48
Acenaphthylene	60-130	45	54-126	33-145	74
Aldrin	7-152	39	7-152	D-166	81
Anthracene	58-130	40	43-120	27-133	66
Benzo(a)anthracene	42-133	32	42-133	33-143	53
Benzo(b)fluoranthene	42-140	43	42-140	24-159	71
Benzo(k)fluoranthene	25-146	38	25-146	11-162	63
Benzo(a)pyrene	32-148	43	32-148	17-163	72
Benzo(ghi)perylene	13-195	61	D-195	D-219	97
Benzyl butyl phthalate	43-140	36	D-140	D-152	60
beta-BHC	42-131	37	42-131	24-149	61
delta-BHC	D-130	77	D-120	D-120	129
bis(2-Chloroethyl)ether	52-130	65	43-126	12-158	108
bis(2-Chloroethoxy)methane	52-164	32	49-165	33-184	54
bis(2-Chloroisopropyl) ether	63-139	46	63-139	36-166	76
bis(2-Ethylhexyl) phthalate	43-137	50	29-137	8-158	82
4-Bromophenyl phenyl ether	70-130	26	65-120	53-127	43
2-Chloronaphthalene	70-130	15	65-120	60-120	24
4-Chlorophenyl phenyl ether	57-145	36	38-145	25-158	61
Chrysene	44-140	53	44-140	17-168	87
4,4'-DDD	D-135	56	D-135	D-145	93
4,4'-DDE	19-130	46	19-120	4-136	77
4,4'-DDT	D-171	81	D-171	D-203	135
Dibenz(a,h)anthracene	13-200	75	D-200	D-227	126
Di-*n*-butyl phthalate	52-130	28	8-120	1-120	47
3,3'-Dichlorobenzidine	18-213	65	8-213	D-262	108
Dieldrin	70-130	38	44-119	29-136	62
Diethyl phthalate	47-130	60	D-120	D-120	100
Dimethyl phthalate	50-130	110	D-120	D-120	183
2,4-Dinitrotoluene	53-130	25	48-127	39-139	42
2,6-Dinitrotoluene	68-137	29	68-137	50-158	48
Di-*n*-octyl phthalate	21-132	42	19-132	4-146	69
Endosulfan sulfate	D-130	42	D-120	D-120	70
Endrin aldehyde	D-189	45	D-189	D-209	75
Fluoranthene	47-130	40	43-121	26-137	66
Fluorene	70-130	23	70-120	59-121	38
Heptachlor	D-172	44	D-172	D-192	74
Heptachlor epoxide	70-130	61	71-120	26-155	101
Hexachlorobenzene	38-142	33	8-142	D-152	55
Hexachlorobutadiene	68-130	38	38-120	24-120	62
Hexachloroethane	55-130	32	55-120	40-120	52
Indeno(1,2,3-cd)pyrene	13-151	60	D-151	D-171	99
Isophorone	52-180	56	47-180	21-196	93
Naphthalene	70-130	39	36-120	21-133	65
Nitrobenzene	54-158	37	54-158	35-180	62
N-Nitrosodi-*n*-propylamine	59-170	52	14-198	D-230	87

Table 6 – QC Acceptance Criteria – Method 625 [1]					
Analyte	**Range for Q (%) [2]**	**Limit for s (%) [3]**	**Range for $\overline{X}$ (%) [3]**	**Range for P, P_s (%) [3]**	**Limit for RPD (%)**
PCB-1260	19-130	77	19-130	D-164	128
Phenanthrene	67-130	24	65-120	54-120	39
Pyrene	70-130	30	70-120	52-120	49
1,2,4-Trichlorobenzene	61-130	30	57-130	44-142	50
4-Chloro-3-methylphenol	68-130	44	41-128	22-147	73
2-Chlorophenol	55-130	37	36-120	23-134	61
2,4-Dichlorophenol	64-130	30	53-122	39-135	50
2,4-Dimethylphenol	58-130	35	42-120	32-120	58
2,4-Dinitrophenol	39-173	79	D-173	D-191	132
2-Methyl-4,6-dinitrophenol	56-130	122	53-130	D-181	203
2-Nitrophenol	61-163	33	45-167	29-182	55
4-Nitrophenol	35-130	79	13-129	D-132	131
Pentachlorophenol	42-152	52	38-152	14-176	86
Phenol	48-130	39	17-120	5-120	64
2,4,6-Trichlorophenol	69-130	35	52-129	37-144	58

[1] Acceptance criteria are based upon method performance data in Table 7 and from EPA Method 1625. Where necessary, limits for recovery have been broadened to assure applicability to concentrations below those used to develop Table 7.

[2] Test concentration = 100 µg/mL

[3] Test concentration = 100 µg/L

Q = Calibration verification (Sections 7.3.1 and 13.4)
s = Standard deviation for four recovery measurements in the DOC test (Section 8.2.4).
$\overline{X}$ = Average recovery for four recovery measurements in the DOC test (Section 8.2.4).
P, P_s = MS/MSD recovery (Section 8.3.2, Section 8.4.2).
RPD = MS/MSD relative percent difference (RPD; Section 8.3.3).
D = Detected; result must be greater than zero.

Revision History Log for SOP #601

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
8/18/13	11	Added Benzidine and alpha-Terpineol to the Analyte list	Kathy Cressia	Keith Aloisa	8/18/13
10/3/14	12	Added new mixed spiking solution, updated column & septa part #, new Std table, slight changes to conditions	Damien Drobinski	Keith Aloisa	10/3/14
12/15/14	12.1	Added Parathion to compound list	Kathy Cressia	Keith Aloisa	12/15/14
1/6/16	12.2	New column source, new Absolute custom mix std., 8.1.2.3- new custom, 8.1.2.4- new volumes cal curve, 8.1.5- slight condition changes	Damien Drobinski	Keith Aloisa	1/6/16
12/12/17	12.3	All references to 625 changed to 625.1. Sec 1.0- mentioned additional cmpds, Sec 6.2 – 6.6 – updated all for new standard sources, separate Benzidine standards and spikes, Sec. 8.1.2.3 – 8.1.2.5 – all new curve dilutions, separate Benzidine standards & curve, Sec. 8.1.4- new ICAL criteria, Sec. 15.0- added/updated 625.1 criteria, Sec 16- updated 625.1 method reference, added Appendix A	Damien Drobinski	Keith Aloisa	12/12/17

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date
6/12/18	12.4	Section 8.1.5- slight update to conditions, Table 1: added 2,6-Dichlorophenol to list	Kathy Cressia	Damien Drobinski	6/12/18
7/26/19	12.5	Section 8.1, 8.3, 10, 10.8– Added 8270E to title, changed third CCAL in the 8.3 paragraph to CVS, added section 6.7 for CVS, updated some language to reflect update, added several compounds to table, added Section 16.2 for "E" method	Kathy Cressia	Keith Aloisa	7/26/19

PHOENIX
Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
Telephone: 860.645.1102 • Fax: 860.645.0823

Date: 10/17/22
Version: 10.5
Initiated by: Kathy Celsia
Approved by:

SOP Number: 622.8082

Title: Polychlorinated Biphenyls (PCBs) by Gas Chromatography

1.0 Scope and Application

1.1 Method 8082A is used to determine the concentration of polychlorinated biphenyls (PCBs) as Aroclors or Congeners in extracts from solid, aqueous, oil and air-PUF (TO-10A) matrices. The analysis is performed by Gas Chromatography with an electron capture detector (GC-ECD).

1.2 The estimated quantitation limit for most compounds is 0.5 ppb for a 1-Liter volume of water, 1 ug for air PUF, 330ug/Kg based on a 15g-soil amount and 2.0 mg/Kg for oil. For congeners, 1ug/Kg based on a 15g soil amount and 5ug/L based on a 1L water volume.

1.3 The following procedure covers the determination of these Aroclors: Ar-1016, Ar-1221, Ar-1232, Ar-1242, Ar-1248, Ar-1254, Ar-1260, Ar-1262, and Ar-1268, and these Congeners: BZ#8, BZ#18, BZ#28, BZ#52, BZ#49, BZ#44, BZ#66, BZ#101, BZ#87, BZ#118, BZ#184, BZ#153, BZ#105, BZ#138, BZ#187, BZ#183, BZ#128, BZ#180, BZ#170, BZ#195, BZ#206 and BZ#209

2.0 Summary of Method

2.1 The PCBs are extracted from soil, water, oil and air PUF with 1:1 acetone/hexane and brought to a final volume of 5mL. For air PUF or if a LDL (low detection limit) is desired, the final volume is 1mL.

2.2 The extract is injected into the GC-ECD via direct splitless injection into a fused silica capillary column. The GC is temperature programmed to separate the Aroclor patterns over time, which are then detected by the ECD.

2.3 The samples are quantitated based on a fingerprint pattern associated with each Aroclor. In addition, the retention times for the peaks within the pattern are matched with that of a known standard.

3.0 Interferences

3.1 Interferences co-extracted from samples will vary considerably from matrix to matrix, but can be grouped into three broad categories;

3.1.1 Contaminated solvents, reagents or sample processing hardware.

3.1.2 Contaminated parts, column surfaces and detector surfaces.

3.1.3 Compounds extracted from the sample that will cause detector response.

3.2 Interferences by Phthalate esters, found mostly in plastics and introduced during sample prep, can pose a major problem in PCB analysis. Refer to Sample Preparation SOPs.

3.3 Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed with a sample clean up.

4.0 Preservation and Storage

4.1 For aqueous samples, collect 1 liter of unpreserved sample in an amber glass container with a Teflon-lined cap.

4.2 For soil samples, collect in a 4 oz soil jar.

4.3 PUF samples must be wrapped in aluminum foil and pack in a glass container.

4.4 Aqueous samples must be extracted within 7 days from time of collection, soil, oil, and air PUF samples must be extracted within 14 days from time of collection.

4.5 Samples must be kept at 4°C until time of extraction.

4.6 Sample extracts must be stored at 4°C in the dark and analyzed within 40 days of extraction.

5.0 Equipment and Supplies

5.1 Perkin Elmer Autosystem Gas Chromatography with ECD.

5.2 PCB Dual-column: Restek Rtx-440 (12924) and Restek Rxi-35sil MS (13824), 30m x 0.32mm ID x 0.25 µm film thickness.
For Congeners – Dual Column, Restek RTX-PCB (13239) and Restek Rxi-35sil MS (13824), 30m x 0.32mm ID x 0.25 µm film thickness

5.3 Gas- Ultra high purity liquid nitrogen.

5.4 Supelco Visiprep DL SPE vacuum manifold with disposable liners.

5.5 Restek Resprep SPE florisil extraction columns- 1000 mg/column, Cat. Number 24034-fl, or equivalent.

6.0 Reagents and Standards

PCBS as Arochlors

6.1 Hexane – Pesticide/PCB quality

6.2 Calibration Stock Standards

6.2.1 Aroclor 1016/1260 at 1000ug/mL in Isooctane (Ultra # PPM-8082)
6.2.2 Aroclor 1221 at 100ug/mL in Isooctane (Ultra # PP-292)
6.2.3 Aroclor 1232 at 100ug/mL in Isooctane (Ultra # PP-300)
6.2.4 Aroclor 1242 at 100ug/mL in Isooctane (Ultra # PP-310)
6.2.5 Aroclor 1248 at 100ug/mL in Isooctane (Ultra # PP-342)
6.2.6 Aroclor 1254 at 100ug/mL in Isooctane (Ultra # PP-352)
6.2.7 Aroclor 1262 at 100ug/mL in Isooctane (Ultra # PP-371)
6.2.8 Aroclor 1268 at 100µg/mL in Isooctane (Ultra # PP-382)

6.3 Pesticide Surrogate Standard Spiking Solution (TCMX and DCB) at 200ug/mL in acetone (Ultra # ISM-320). Make a 10x dilution of this solution before use.

6.4 Internal Standard- 1-Bromo-2-nitrobenzene at 5000µg/mL in acetone (Ultra PPS-351)

6.5 Calibration Verification Standard (CVS)
6.5.1 Aroclor 1016 at 1000µg/mL in methanol (Absolute # 70015)
6.5.2 Aroclor 1260 at 1000µg/mL in methanol (Absolute # 70021)

NOTE: Stock standard solutions must be replaced before the expiration date.

6.6 Calibration and Standard Curve

6.6.1 1016/1260 Working Standard solution- Prepare a 1:10 dilution of 1016/1260 stock standard Ultra PPM-8082 in hexane. Dilute this working standard as outlined below.

µL 1016/1260	µL surrogate	µL Internal Standard	Final volume mls	1016/1260 Conc. ppb	Surrogate Conc. ppb
10	10	100	10	100	20
20	20	100	10	200	40
40	30	100	10	400	60
60	40	100	10	600	80
80	50	100	10	800	100
100	60	100	10	1000	120
120	70	100	10	1200	140
Low Level					
5	5	100	10	50	10

6.6.2 Prepare 1000 ppb standards of the remaining Stock Standard Aroclors in hexane, as outlined below.

µL Aroclor	µL surrogate	µL Internal Standard	Final Volume mls	Aroclor Conc. ppb	Surrogate Conc. ppb
100	50	100	10	1000	100

6.7 Working Calibration Verification Standard

6.7.1 Prepare a calibration verification working standard from the CVS standards in section 6.5 by adding 100µL Ar-1016 (Absolute # 70015) and 100 µL Ar-1260 (Absolute # 70021) to a final volume of 1mL in methanol.

6.7.2 Prepare a calibration verification standard (CVS) of Aroclors 1016 and 1260 at 500 ppb in hexane, from the calibration verification working standard in section 6.7.1, as outlined below.

µL Calibration Verification Working Standard	µL surrogate	µL Internal Standard	Final Volume mls	Aroclor Conc. ppb	Surrogate Conc. ppb
50	50	100	10	500	100

6.8 Activated Copper, RESPREP, copper granules 26/36

<u>CONGENERS</u>

6.9 Hexane – Pesticide/PCB quality

6.10 Calibration Stock Standards

6.10.1 Ultra EPA PCB Calibration Check Solution 100ug/ml (RPC-EPA2)
6.10.2 Ultra 2,2',4,5'-Tetrachlorobiphenyl BZ#49 100ug/ml (RPC-030s)
6.10.3 Ultra 2,2',3,4,5'-Pentachlorobiphenyl BZ#87 100ug/ml (RPC-099s)
6.10.4 Ultra 2,2',3,4,4',5',6-Heptachlorobiphenyl BZ#183 100ug/ml(RPC-073s)
6.10.5 Ultra 2,2',3,4,4',6,6'-Heptachlorobiphenyl BZ#184 100ug/ml (RPC-168s)

6.11 Surrogates

6.11.1 Ultra 2,2',3,3',4,5,5',6-Octochlorobiphenyl BZ#198 100ug/ml (RPC-075s)
6.11.2 Ultra 2,4,5,6 Tetrachloro-m-xylene (TCMX) 2000ug/ml (IST-440-1)

6.12 Internal Standard-

6.12.1 1-Bromo-2-nitrobenzene at 5000µg/mL in acetone (Ultra PPS-351)
6.12.1.1 Prepare by diluting 100ul of Ultra PPS-351 to a final volume of 100ml in hexane

6.13 Calibration Verification Standard (CVS)

6.13.1 Absolute PCB Congeners 100ug/ml (99482)

NOTE: Stock standard solutions must be replaced before the expiration date.

6.14 Calibration and Standard Curve

6.14.1 PCB-Mix A – Prepare a working standard by adding 100ul EPA calibration check solution, 5ul TCMX, 100ul BZ#198, 100ul BX#49, 100ul BZ#87, 100ul BZ#183, and 100ul BX#184 to a final volume of 1ml in hexane.
6.14.2 Dilute Mix A as outlined below with a final volume of 10ml of hexane plus 100ul of internal standard.

Congener concentration	µL Mix A	Final volume mls Hexane	µL Internal Standard
2	2	10	100
5	5	10	100
10	10	10	100

25	25	10	100
50	50	10	100
75	75	10	100
100	100	10	100

6.15 Calibration Verification Standard

6.15.1 Prepare CVS by diluting 5ul Absolute PCB Congeners 100ug/ml (99482) to a final volume of 10ml of hexane plus 100ul internal standard.

7.0 Definitions

7.1 Calibration Standard (CAL) - A solution of procedure analytes used to calibrate instrument response.

7.2 Laboratory Control Sample (LCS) - A sample containing known concentrations of analytes that is added prior to sample preparation and then analyzed by the laboratory to demonstrate that it can obtain acceptable identifications and measurements.

7.3 Surrogate Compound- A compound that is not expected to be found in the sample that is added to a sample aliquot before extraction and is measured with the same procedures used to measure sample components. The purpose of a surrogate compound is to monitor procedure performance with each sample.

7.4 Method Blank- An aliquot of DI water or solid reference material that is treated as a sample. It is exposed to all glassware and apparatus, and all procedure solvents, reagents and surrogate compounds. The extract is concentrated to the final volume used for samples and is analyzed the same as a sample extract. The method blank is used to determine if method analytes are present in the lab environment, glassware or reagents.

8.0 Procedure

8.1 Gas Chromatograph Conditions

8.1.1 Injector Temperature: 250 degrees C

8.1.2 Detector Temperature: 380 degrees C

8.1.3 Carrier Gas: UHP Nitrogen at 22.0psi as determined by dead space calculation.

8.1.3.1 To determine dead space, set oven temperature to the starting temperature of the method to be used. Inject by hand, 2uL of headspace from a vial of methylene chloride. Time until a milivolts spike is seen on the detector. Ideal dead time is 1 min. +/- 10 seconds. Adjust carrier flow to reach ideal dead time.

8.1.4 Temperature Program: Benchmark, analyst may revise as needed depending on GC being used.

For PCBs:

140°C for 1 minute
Ramp 10.5 °C/min. to 310°C
Hold for 0.81 minutes
Total run time = 18 minutes.

For Congeners:

180°C for 1 minute
Ramp 7°C/min. to 220°C, no hold
Ramp 4°C/min. to 275°C, no hold
Ramp 15°C/min. to 330°C, 4 min hold
Total run time = 28.3 minutes.

8.2 Initial Calibration

8.2.1 A calibration curve must be performed prior to the analysis of any samples. For PCBs, a 50ppb low level eighth point can be added to the calibration by the analyst based on instrument performance for low level work. Identify each surrogate peak and five representative peaks for each aroclor. Calibrate the instrument. The RSD for Ar-1016 and Ar-1260 curves must be below 20%. Ar-1254 is calibrated with one calibration standard in every run. The other remaining Aroclors are calibrated using one standard level, identifying five peaks representative of that aroclor, when that aroclor is found in a sample. The assumption can be made if the RSD is below 20% for mix Ar-1016/1260, a one-point calibration for the remaining Aroclors may be used for calibration and quantitation.

For congeners, ICAL %RSD must be <20% or R squared > 0.99 for linear regression

8.3 Calibration Verification

8.3.1 A 500 ppb Mix 1016/1260 standard made from a source external to that of the calibration standards is analyzed following instrument calibration. The sole purpose of this standard is to validate the concentration of the calibration standards, therefore it is only analysed when a new calibration is performed. The percent recovery must not exceed +/-20%. If the standard fails to meet these criteria, a fresh standard should be made and analyzed. If the standard passes, analysis may continue. If the standard fails again, new calibration standards should be made and a new calibration performed.

8.3.2 For congeners – a 50ppb standard is made from 6.13.1 as described in 6.15.1 - The percent recovery must not exceed +/-20%.

8.4 Continuing Calibration

8.4.1 A continuing calibration standard should be injected every ten samples and must be analyzed at least every twelve hours. For PCBs, a 600 ppb Mix 1016/1260 standard is used for this purpose and for congeners; a 50ppb standard is used. All samples must be bracketed between check standards. The percent recovery for the calibration check standard must not exceed +/-15%. If the recovery fails to meet these criteria, a fresh standard should be made and analyzed. If the recovery meets the +/-15% criteria, analysis may continue. If the recovery fails again, a new calibration must be performed and data preceding the failed standard may not be reported. If the check standard recovery is greater than 15%, samples that are non-detects may be reported. Additionally, a 1000 ppb Ar-1254/100 ppb Ar-1221 mix calibration check standard is analyzed at the beginning and end of each run to verify change in ECD response.

8.5 Method Blank

8.5.1 A method blank for each batch of extractions is to be run along with the samples. A method blank must be extracted daily for

each type of extraction and at least every 20 samples. Analyte recovery should not exceed the reporting limit for that analyte.

8.6 Matrix Spike / Matrix Spike Duplicate

8.6.1 One MS/MSD per matrix must be analyzed every 20 samples. Recoveries should fall between the laboratory control limits of 40-140%. Spiked samples do not require re-analysis if matrix interferences are visibly present.

8.7 Laboratory Control Sample (LCS/LCSD)

8.7.1 One LCS/LCSD per matrix is to be run every 20 samples. Recoveries should fall between the laboratory control limits of 40-140%.

8.8 Sample Preparation

8.8.1 The sample extract for soils and waters will have a final volume of 5mL in hexane (if LDL analysis is desired, final volume will be 1mL). If the extract is to be analyzed for PCBs only, add 3 to 5 mL of concentrated sulfuric acid. If the extract is also to be analyzed for pesticides, split the extract and acidify one split. Concentrated sulfuric acid is used to remove matrix interferences such as elemental sulfur and phthalate esters. If concentrated sulfuric acid is added to the extracts, it must be added to associated QC samples as well.

8.8.2 Other sample clean-ups may need to be performed to remove interferences. They are as follows:

8.8.2.1 For samples that contain a high baseline and for all oil and caulk matrices, the sample is taken through a florisil cleanup. Restek Resprep disposable Florisil extraction columns are used. The sample is passed through using vacuum, and when necessary re-concentrated to avoid sample dilution. Label the vial with an "F" after the sample # on the vial to designate the sample is a florisil cleanup. In addition, put an "F" after the sample # in the instrument sequence. Any QC attached to this sample must also go through the clean-up procedure.

8.8.2.2 For samples that contain large amounts of sulfur, a copper cleanup must be performed. Place an aliquot of the sulfuric treated sample and place into a 2 ml glass vial and add 2-5 grams of the treated copper. Shake vigorously for approximately one minute and allow the layers to separate and reanalyze. Label the vial with an "Cu" after the sample # on the vial to designate the sample is a sulfur cleanup. In addition, put an "Cu" after the sample # in the instrument sequence. Any QC attached to this sample must also go through the clean-up procedure.

8.8.2.3 Additional cleanup methods may be used as deemed necessary by the analyst including; $KMnO_4$, additional sulfuric acid, or more florisil.

8.8.3 Make appropriate dilutions for matrix and required reporting levels.

9.0 Calculations

9.1 Samples are calculated by average response factor over a 7-point or 8-point curve for Ar-1016/1260 and 1 point for the remaining Aroclors, as needed. Samples are matched to the Aroclor that has a similar fingerprint pattern. Sample concentrations that are above the calibration curve must be diluted to the middle of the calibration curve with hexane.

9.2 From the Turbochrome report, determine the concentration of the individual compounds in the sample from the raw amount using the calculations below.

9.3 Final Concentration in ppb (µg/Kg):

$$\frac{\text{ppb from TC} * \text{Final Volume (mL)} * \text{Dilution Factor} * 100}{\text{Initial Weight (g)} * \% \text{ solids}}$$

9.4 Final Concentration in ppb (µg/L):

$$\frac{\text{ppb from TC} * \text{Final Volume (mL)} * \text{Dilution Factor}}{\text{Initial Volume (mL)}}$$

10.0 Quality Control

10.1 All standards are labeled, where applicable, with date received, date opened, analyst initials, expiration date, analyte name, analyte concentration and lot number.

10.2 An initial demonstration of capability must be performed to prove the generation of acceptable data with regard to accuracy and precision. An initial demonstration must be done for each new analyst, whether in the prep or instrument departments.

10.3 A continuing demonstration of capability or accuracy and precision study must be performed annually. Four standards at a level approximately ten times higher than the detection limit are evaluated for accuracy (% recovery) and precision (standard deviation). The RSD must be <20%.

10.4 A Limit of detection study is run on each instrument annually. A blank, spiked at the limit of detection is prepared through each extraction technique. This is run on each instrument and must recover 50%-200%.

10.5 Prep Blanks- Before processing any samples, the analyst must demonstrate that all glassware and reagent interferences are under control. Each time a batch of samples is extracted, a prep blank must be analyzed. If within the retention time window of any analyte of interest the LRB produces a peak that would prevent the determination of that analyte, determine the source of contamination and eliminate the interference before processing samples.

10.6 Matrix Spike and Matrix Spike Duplicate- Two fortified environmental samples (labeled MS and MSD) are analyzed every batch of 20 samples. Thus 10% of all samples are represented by a fortified sample. Calculate the percent recovery for each analyte by dividing the result by the true value. The recovery must be between 40-140% for MS/MSD. If the recovery of any such analyte falls outside the control limits and the LCS for that analyte is shown to be in control, the recovery problem encountered with the MS/MSD is judged to be matrix related, not system related. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

10.7 Laboratory Control Sample and Laboratory Control Duplicate- Two fortified blank samples (labeled LCS and LCSD) are analyzed every batch of 20 samples. Calculate the percent recovery for each analyte by dividing the result by the true value. The recovery must be between 40-140% for

LCS/LCSD. If the recovery of any such analyte falls outside the control limits, the batch needs to be re-preped.

10.8 Surrogate Recoveries

10.8.1 Surrogate recoveries should be 30% - 150% for both surrogates. When surrogate recovery from a sample or prep blank is <30%, it must be re-preped. If a surrogate recovery is >150%, it must be narrated.

10.8.2 If sample extract reanalysis meets the surrogate recovery criterion, report only data for the reanalyzed extract. If sample extract reanalysis continues to fail the surrogate recovery criterion, report all data for that sample as suspect.

10.9 Secondary Column Confirmation- Positive results must be confirmed on a secondary column. The higher of the two results must be reported.

11.0 Safety

11.1 The toxicity and carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.

11.2 Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures

12.0 Pollution Prevention

12.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

12.2 Reagents and standards should be purchased and/or prepared in volumes consistent with laboratory use to minimize the volume of disposal.

13.0 Waste Management

13.1 It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the

hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

14.0 Method Performance

14.1 This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples, matrix spikes and duplicate sample analysis.

14.2 See Section 10.0 Quality Control in this SOP for acceptable limits.

15.0 Corrective Action for Out-of-Control or Unacceptable Data

15.1 See Section 10.0 Quality Control in this SOP for corrective actions.

16.0 References

16.1 EPA Test Methods for Evaluating Solid Waste, SW-846, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography", Third Edition, Update IV, 2/07, Method 8082A.

16.2 EPA/625/R-96.010b Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, 2nd Edition, Method TO-10A, "Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam(PUF) Sampling followed by Gas Chromatographic/Multi-Detector Detection".

16.3 40 CFR Part 136 Appendix A, "Test Procedures for Analysis of Organic Pollutants, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", Method 608: Organochlorine Pesticides and PCBs.

Revision History Log for SOP #622.PCB

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
9/20/13	8	Make surrogate differently, CVS & LCS true values changed, updated control limits, clarified calibration of other aroclors	Kathy Cressia	Adam Werner	9/20/13
7/21/14	9	Added PCB in oil reference EPA 600, added oil to Sec. 1 & 2, added 1221 to mix CCAL	Kathy Cressia	Adam Werner	07/21/14
10/22/14	10	Added oil RL, more info on CVS Std and dilutions, ICAL RSD for 608 added, Sec. 8.8.2.3 added for additional clean ups, added 608 method reference	Adam Werner and Kathy Cressia	Keith Aloisa	10/22/14
5/10/16	10.1	Sec 5.2- new column supplier, Sec 5.5- new florisil column supplier, Sec 6.6.1- added low level standard to table, added Sec 8.1.3.1- determining dead space, Sec 8.1.4- changes to temperature program, Sec 8.2.1 – added low level standard, added Sec 10.4- LOD study, fixed assorted formatting issues throughout	Adam Werner	Keith Aloisa	5/10/16
3/20/17	10.2	Added Section 6.8 to reagents- purchasing activated Cu now, deleted part of section 8.8.2.2 on how to activate the copper for clean up	Adam Werner	Kathy Cressia	3/20/17
4/20/17	10.3	Deleted reference to EPA 600 method in Section 1.0 and 16.0 pertaining to oils	Kathy Cressia	Adam Werner	4/20/17
12/7/17	10.4	This update added all the information on the congeners. See sections	Adam Werner	Keith Aloisa	12/7/17

		1.0, 5.0, 6.0, & 8.0 for the analysis of congeners			
10/17/22	**10.5**	**Added section 10.9**	**Kathy Cressia**	**Keith Aloisa**	**10/17/22**

PHOENIX
Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
Telephone: 860.645.1102 • Fax: 860.645.0823

Effective Date: 03/31/20
Version Number: 1.5
Initiated By: Kathy Cressia
Approved By: Jeff Bull

SOP No.: **506**

Title: Determination of Trace Elements by Inductive Coupled Argon Plasma (ICP) Spectroscopy

Scope: This procedure describes the determination of dissolved and total recoverable elements in ground water, drinking water, waste water, and air by ICP. This procedure is also applicable to total recoverable elements in sediment, sludges, biological tissue and solid waste samples. This procedure is the general ICP procedure, which covers the requirements of the SW-846 6010, EPA 200.5 and EPA 200.7 as approved in 40 CFR part 136 and 40 CFR part 141.

1.0 Summary

1.1 This procedure describes the simultaneous analyses of metals on the following elements (Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, P, K, Se, Ag, Au, Zr, Na, S, Sn, Sr, Ti, Tl, V, and Zn) by ICP. All samples are digested prior to analysis. For sample digestion information, refer to SOP #205, #219, #224, #231, #238 and #358.

1.2 When using EPA method 200.5 (Lead and Copper) all samples are digested and preconcentrated prior to analysis. Preconcentrating samples increases analytical sensitivity for meeting method detection limit (MDL) requirements. Therefore, when using this method, the need to measure sample turbidity prior the analysis is eliminated.

2.0 Sample Collection, Preservation and Storage

2.1 Samples can be collected in plastic, Teflon or glass. When using EPA method 200.5 it is recommended that only plastic or PTFE should be used to eliminate the possibility of contamination of boron or silica. They must be preserved with HNO_3 to a pH < 2. They must be stored at 4°C until the time of analysis.

2.2 Samples have a holding time of 6 months.

2.3 Digestions holding time is 180 days.

2.4 ICP analysis is performed on sample digests only.

3.0 Interferences

3.1 Spectral interferences are minimized by the optimization of background points and the use of inter-element correction factors.
3.2 Physical interferences are associated with viscous samples and can be minimized by diluting the sample.
3.3 Memory interferences result when analytes from the previous sample contribute to a signal in the new sample.
3.4 An internal standard measured to increase the reproducibility of measurements of high concentrations or help correcting for samples with varying viscosities.

4.0 Equipment and Supplies

4.1 100 mL Volumetric Flasks
4.2 100 mL Graduated Cylinders
4.3 250 mL Beakers
4.4 1000, 500, and 250 mL Plastic Storage Bottles
4.5 20.0 L Nalgene Carboy
4.6 10 and 50 mL Polystyrene Disposable Beakers
4.7 Pipettes and Pipette Tips
4.8 10.0 mL Oxford
4.9 1 mL Eppendorf
4.10 0.1 mL Eppendorf
4.11 Pump Tubing (Cole-Palmer and Glass Expansion)
4.12 Argon Gas (Liquid)

5.0 Instrumentation

5.1 SPECTRO Model BLUE CCD ICP-EOP, Laboratory computer and printer; and ESI SC4DX Autosampler.
5.2 SPECTRO Model ARCOS CCD ICP-EOP, Laboratory computer and printer; and ESI SC-4DX Autosampler.
5.3 SPECTRO ARCOS II FHX22 ICP-OES, Laboratory computer and printer; and Elemental Scientific FAST 4DX Autosampler
5.2 Sample induction system consists of these major components: Modified Lichte High Solids Nebulizer, cyclonic spray chamber and torch.
5.3 Fiber Optics use as optical transfer devices.
5.4 Elemental Scientific oneFAST sample introduction system

6.0 Reagents and Standards

6.1 Argon Gas
6.2 Deionized water, ASTM Type or equivalent

6.3 Concentrated Hydrochloric Acid, Trace Metals Grade.

6.4 Concentrated Nitric Acid, Trace Metals Grade.

6.5 2.5% Nitric Acid (HNO_3) and 1% Hydrochloric Acid (HCL) - used in standards preparations. Prepare fresh daily. Fill 1000 mL volumetric flask half-full with DI water, add 25 mL concentrated HNO_3 and 10 mL concentrated HCL, dilute to 1000 mL with Di water. Use this solution in the preparation of all standards and dilutions of all samples.

6.6 Standards – Metal Plasma Grade Standards, assorted vendors

6.7 Calibration Verification (ICV), Calibration Verification Sample (CVS), and Interference check sample (ICS) standards are currently supplied by Inorganic Ventures and Environmental Express.

GENERAL PRECAUTIONS:

- Avoid contamination of Stock Standards. Always pour out a small volume of standard stock solution into a new beaker before taking an aliquot.
- Never insert a pipet directly into the bottle.

7.0 Definitions

7.1 Calibration Verification Standard (CVS) – A purchased standard of a known concentration that is from a different source than the calibration standards. The CVS is used to determine laboratory performance.

7.2 Preparation Blank (Prep Blank) – An aliquot of reagent water that is treated exactly as a sample. The prep blank is used to determine if method analytes are present in the lab environment, reagents, or glassware.

7.3 Calibration Standard – A solution prepared from the dilution of stock standard solutions. The Calibration solutions are used to calibrate instrument response with respect to analyte concentration.

7.4 Matrix Spike (MS) – An aliquot of environmental sample to which a known quantity of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.

7.5 Laboratory Control Sample (LCS) or Blank Spike- the LCS is analyzed exactly like a sample and uses the same source and concentration as the MS but added to a clean (control) matrix (DI water). For soils, it is a purchased soil matrix standard.

7.6 Maximum contaminant level (MCL) – the maximum permissible level of a contaminant in water which is delivered to any user of a public water system.

7.7 Linear Range (LR) – the concentration range over which the instrument response to an analyte is linear.

7.7 Limit of Quantitation (LOQ) -- the minimum levels, concentrations, or quantities of a target analyte that can be reported with a specified degree of confidence.

8.0 Procedure

8.1 Preparation of Standards

8.1.1 Blank (2.5% HNO_3 and 1% HCL) – 25 mL of trace metal grade nitric acid and 10 mL trace metal grade hydrochloric acid diluted to 1000 mL DI water.

8.1.2 CLP 1 + 3 – 10 mL of Environmental Express CLP-CAL-1 and 10 mL of Environmental Express CLP-CAL-3 diluted to 1000 mL with 2.5% HNO_3 and 1% HCL.

8.1.3 ICP High CAL STD (CRDL_S) -- 20 mL of Inorganic Ventures PEL STD 1A Rev1 + 20 mL of Inorganic Ventures PEL STD 2 to 1L with 2.5% HNO_3 and 1% HCL.

8.1.4 ICP CAL CHK (ICV) -- 10 mL of Inorganic Ventures PEL STD 1A Rev1 + 10 mL of Inorganic Ventures PEL STD 2 to 1L with 2.5% HNO_3 and 1% HCL.

8.1.5 CRQL – 10 mL of 1:100 PEL STD CRDL-1 and 10 mL of 1:100 PEL STD CRDL-2 diluted to 1000 mL of 2.5% HNO_3 and 1% HCL.

8.1.6 CRDL -- 50 mL of 1:100 PEL STD CRDL-1 and 50 mL of 1:100 PEL STD CRDL-2 diluted to 1000 mL of 2.5% HNO_3 and 1% HCL.

8.1.7 CVS – Second source- 10 mL of Absolute Standard 200.7/6010/200.5 Custom Mix (50692) diluted to 1000 mL with 2.5% HNO_3 and 1% HCL.

8.1.8 ICSA – 10 ml of 2007ICS-4 diluted to 1000 mL 2.5% HNO_3 and 1% HCL.

8.1.9 ICSAB – 10 mL of 2007ICS-4 + 1 mL of CGSB1-1 + 1 mL 2007 ICS-1 + 1 mL 2007 ICS-3 diluted to 1000 mL with 2.5% HNO_3 and 1% HCL.

8.1.10 All Standards are prepared and transferred to Teflon storage bottles and labeled.

8.1.11 Interference Check Standards: Cr, Mn, Ti, Zn (10.0 ppm) are prepared with 1.0 mL of a 1000 ppm stock standard from Environmental Express and diluted to 100 mL with 2.5% HNO_3 and 1% HCL. A 1.0 ppm standard of As is prepared with 0.1 mL of 1000 ppm stock from Environmental Express and diluted to 100 mL with 2.5% HNO_3 and 1% HCL. Other interfering check standards are Ca 200.0 ppm, Al 50.0 ppm, Fe 200.0 ppm, and Na 500.0 ppm.

8.1.12 Spiked Sample Analysis: Inorganic Ventures QCP-QCS-1 & 2 - The spike is added before the digestion. Take 0.5 mL of each spiking solution and add to the sample. The final volume is 50 mLs. When performing a post-digestion spike, take 0.08 mL of each QCP-QCS-1 and QCP-QCS-2 and bring up to 8.0 grams of sample. For soil matrix post digestion spike take 0.16 mL of each QCP-QCS-1 and QCP-QCS-2 and dilute to 8.0 grams of sample.

8.2 Tuning and Calibration of the ICP

8.2.1 Torch Maintenance - Open the Torch box and visually inspect the glass, and RF load coils for cleanliness and any oxidation. Check all connections – Drain hose, nebulizer cap, Argon lines, and cooling water.

8.2.2 Peristaltic Pump - Install new pump tubing if the old one shows signs of flattening or stretching and connect to the nebulizer with capillary tubing. Pump DI water and adjust pressure on tension arms to obtain a smooth flow.

8.3 Start-Up

8.3.1 Turn the main switch on the unit to "ON". Switch on the computer allowing the operating system to boot up.

8.3.2 Open ESI Software.

8.3.3 Start the unit software "Smart Analyzer Vision" after the operating system has been completely booted. Allow they unit to stabilize for at least 60 minutes after switching it on. Also make sure that the operating temperature (15°C) has been reached

8.3.4 On the Smart Analyzer Vision software select the Analysis view. Flush the sample introduction system with argon for approximately 5 minutes by clicking the "flush" button. The following conditions should be set:

Coolant: 13.4 L/min
Auxiliary: 0.70 L/min – 1.0 L/min
Nebulizer: 0.9 L/min

8.3.5 To ignite the plasma click on the "plasma" button.

8.3.5.1 The sample introduction system is automatically flushed with argon again at this point. In the status line "Plasma is starting" should be displayed. It takes about 2 minutes for the plasma to ignite.

8.3.6 Start the peristaltic pump by clicking the "pump" button

8.3.6.1 Allow rinse water (5% HNO_3+ 5% HCl) mixture to run through the system for 30 minutes to allow the unit to heat up and stabilize the components.

8.4 Optimization

8.4.1 Make sure the optical system is "ICAlized". The system needs to be "ICALized":

- After a software message (wavelength drift).
- After switching on the unit and temperature stabilization.
- In the event of changes concerning the sample induction system (torch or nebulizer changed).
- Before setting up a new method.
- Before carrying out high-precision measurements.

8.4.2 To perform an "ICALization"

8.4.2.1 After the plasma has been ignited and the system flushed and rinsed select the function "ICALization" in the "System" menu. Follow the instructions displayed by the software.

8.5 Measuring the Samples:

8.5.1 Perform the calibration using:

8.5.1.1 Blank (2.5% HNO3 and 1% HCL), CRQL, CRDL, IPC 1:10, IPC, CLP 1 + 3, and ICSA.

8.5.1.2 After completing the calibration a regression sheet is printed. The correlation coefficient should be no less than 0.9975 for each element.

8.5.1.3 To control the quality of results measure:

- Calibration verification standard (IPC 1+2) ± 5% for ICV
- CVS (second source) ± 10% (± 5% 200.7 DW & WW)
- CRQL (Quantitation limit standard)
- CRDL (Detection limit standard)
- ICB (Initial Calibration blank)
- Interference check standard- ICSA ± 20%
- Interference check standard- ICSAB ± 20%
- CRDL_S- High standard check
- IPC 1+2 (CCV) ± 10%
- CCB

8.5.2 Fill the autosampler trays with samples.

8.5.2.1 Sequence Generator

8.5.2.1.1 In the Phoenix LIMS program open the QA/QC Batching, ICP Sequence. Once the sequence page is open select the instrument you are running and press "new", click yes for the prompt to build a new sequence.

8.5.2.1.1.1 At this point you can manually type in the sample number you are running and select the matrix.

8.5.2.1.1.2 You can add a whole batch by clicking "Add Batch". Type in the whole batch (ex. TMD-SM-XXXXXX). Click add batch to sequence.

8.5.2.1.1.3 Once all the samples/batches have been added to the sequence click on "build sequence" click ok. Go to the software and paste into the tray position.

8.5.2.2 Analyze prep blank, blank spike, sample duplicate, sample spike and samples. Analyze calibration verification standard and calibration blank every 10 samples to ensure the calibration is still consistent.

8.5.3 EPA 200.5 samples

8.5.3.1 When analyzing preconcentrated drinking water samples, this method can only be used to report analysis on samples that fall into the requirements listed below.

8.5.3.1.1 Operative matrix effects can occur from elevated dissolved solids therefore when calcium (Ca) concentrations are greater than 125 mg/L or the combined concentrations of calcium (Ca), magnesium (Mg) sodium (Na) and Silica (Si) are greater than 250 mg/L and the primary contaminant concentration exceeds 80% of the established maximum contaminant level (MCL) or action level. If a sample exceeds these criteria the sample will then be analyzed using 200.7 or 200.9 (sample run on AA).

8.5.3.1.2 When determining Lead by 200.5 the instrument must be able to analyze silica as well. When the levels of silica exceed 30 mg/L (when preconcentrated 2X) a suppressive effect on the lead has been observed. For samples containing silica above 30 mg/L and lead concentrations >10 ug/L, lead must be determined by

another approved method. These samples would then be run using EPA method 200.9, and run by AA.

8.5.3.2 When determining Lead and Copper by 200.5, the following additional quality considerations must be met:

8.5.3.2.1 Any routine samples where the Lead and Copper concentrations are determined to be ≥80% of the established MCL or action level must be reanalyzed by another approved method if the required matrix spike analysis does not verify the absence of a matrix interference. (Pb @ ≥0.012 ppm, and Cu @ ≥1.04 ppm)

8.5.3.2.2 Prior to analyzing Lead and Copper by 200.5, the calibration standards must be verified by the analysis of 3 LCS samples, and their determined mean concentrations must be within ±5% of true value. Although a second source is analyzed daily, this procedure must be performed on an annual basis and after the preparation of stock or calibration standards.

8.6 Switching off the Instrument

8.6.1 Flush the sample introduction system for approximately 10 minutes with the rinse solution. This is to eliminate the potential for crystallization within the sample introduction system.

8.6.2 Select the analysis view. Switch off the plasma by clicking the "Plasma" button.

8.6.3 Exit the software.

8.6.4 Shut down the computer.

8.6.5 Switch of the exhaust and the coolant water.

8.6.6 Release the pressure clamps on the peristaltic pump and release the pump tubing.

8.6.7 To switch off the unit completely turn the main switch to "Off".

9.0 Calculations

9.1 Calculation of Lead in Air

$$C\ (\mu g/m^3) = \frac{C_s\ V_s}{V}$$

where:

C_s = lead in sample (µg/mL)
V_s = sample volume (mL)
V = air volume sampled (m^3)

10.0 Maintenance

10.1 Daily Checks

10.1.1 Clean OPI. Using DI water and a Q-Tip clean the face of the OPI.
10.1.2 Change Torch.
10.1.3 Check Elbow. Make sure the elbow has no water or condensation in it. Change the elbow if any water droplets are present.
10.1.4 Change peristaltic pump tubing.
10.1.5 Check all tubing and feed hoses. Change anything that is kinked, flattened, or stretched.
10.1.6 Make sure you have enough rinse water in the carboy.
10.1.7 Check discharge container: if more than ¾ full empty and replace.
10.1.8 Change all QC and Calibration standards.
10.1.9 Check peaks on all elements you are calibrating. Au, Zr, and S need to be checked also.
10.1.10 Check Sc counts during calibration and check the spray chamber to verify nebulizer flow. A drastic drop in Sc count could be a partially clogged nebulizer. Clean or replace the nebulizer if you suspect it to be clogged.

10.2 Weekly Checks

10.2.1 Inspect torches. Remove all torches that appear to have deposits or residue in aerosol tube. Torches with excessively pitted ends of the tube should also be removed.
10.2.2 Clean torches by soaking them in Aqua Regia (mix 1 part concentrated nitric acid and 3 parts concentrated hydrochloric acid.) After allowing the torches to soak to remove residue thoroughly rinse all torches with DI water and allow to dry. Once dry, cover the ends of the torch with Parafilm.
10.2.3 Clean elbows by running a small brush with soap through it and rinsing with hot tap water followed by a DI water rinse. Dry the elbows and cover the ends with Parafilm.
10.2.4 The spray chamber needs to be cleaned and rinsed. They spray chamber does not need to be dried. High levels of sodium can be rinsed out faster with DI water than with the normal rinse operation.
10.2.5 Chiller water levels should be checked weekly to ensure proper OPI cooling.

10.3 Monthly Checks

10.3.1 Inspect window for spotting or debris. Sudden drops in low intensity lines should warrant this check. Sb, Se, Pb, Cd and As are pretty good indicators.

10.3.2 All introduction tubing should also be changed. Torch tubing should be checked and replaced if it becomes too easy to remove from the torch. Nebulizer tubing should also be changed if it's stretched from over pressurization.

10.3.3 Filter media should be cleaned or replaced.

11.0 Trouble Shooting and Corrective Action

10.1 Problem: Stable plasma won't start.
Action: Check system for leaks.

10.2 Problem: Generator cannot be started.
Action: Check power supply (fuses, circuit breaker).

10.3 Problem: ARGON lamp is lit.
Action: Increase argon admission pressure, or replace argon supply if empty.

10.4 Problem: WATER lamp is lit.
Action: Check for and remove blockages (dirt, lime buildup) to the system.

10.5 Problem: Melting of the torch tubes.
Action: Re-adjust torch, remove and clean blockages in the aerosol tube, search for and repair leaks, increase auxiliary gas flow rate, check that exhaust is turned on.

10.6 Problem: Plasma does not ignite.
Action: Re-adjust torch, flush the system with argon by pressing <+> and <-> key wrong argon quality (purity >99.996%).

10.7 Problem: Plasma trembles or pulsates.
Action: Clean and dry torch the aerosol tube, increase power slightly, clean nebulizer.

10.8 Problem: Nebulizer gas flow too low.
Action: Clean nebulizer.

10.9 Problem: Sparking-over at the coil.
Action: Dry coil with tissue paper, clean any dirt, adjust torch (must not touch coil).

10.10 Problem: CV fails for an element.
Action: The instrument may need to be re-calibrated.

10.11 Problem: ICSA or ICSAB fails for an element.
Action: Check background points for that element.
**** SEE SECTION 18: TABLE I FOR MORE TROUBLESHOOTING INFORMATION**

12.0 Quality Control

12.1 Instrument Detection Limits (IDLs)- IDLs are useful means to evaluate instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. IDLs, in ug/L, can be estimated as the mean of the blank results plus three times the standard deviation of 10 replicate analysis of the reagent blank solution. Use zero for the mean if the mean in negative. IDLs must be determined once on new equipment, and after any major instrument maintenance.

12.2 Initial Demonstration of Capabilities (IDOC)- IDOCs are obtained by generating data of acceptable precision and bias for target analytes in a clean matrix. IDOCs are analyzed by each analyst and repeated when significant changes are made to the instrumentation. Continuing Demonstrations of Capabilities (CDOCs) are analyzed and collected annually for each analyst. This information is kept in the analysts training file.

12.3 Linear Range (LR)- The linear range establishes the highest concentration that may be reported without diluting the sample. Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. The standard must recover within 10% of the true value, and if successful, establishes the linear range. The linear range standards must be run daily in order to report to that concentration. If a linear range standard is not analyzed, the highest standard in the calibration becomes the linear range. Annually, the high standard is analyzed after calibration, where the recovery should be within 5% of true value.

12.4 Method Detection Limit (MDL) studies are established for each element per the guidelines established in 40 CFR part 136 Appendix B.

12.5 Sample QC for preparation and Analysis (Batch QC)-

12.5.1 Prep Blanks (PB) or Method Blank- For each batch of samples digested and analyzed, a prep blank must be carried throughout the entire process

to aid in identifying when and/or if sample contamination is occurring. The prep blank is considered acceptable if it does not contain any target analytes at concentrations above the reporting level. Blanks my contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e. targets are not present in the samples or sample concentrations are >10x the blank concentration). If the prep blank is associated with affected samples, the analyst must order a 're-prep' on the entire batch. Prep Blanks are matrix matched.

12.5.2 Laboratory Control Sample (LCS) or Blank Spike- One LCS is digested in every batch of samples, and consists of a clean matrix (DI water) spiked with the same analytical spike and concentration as the matrix spike (MS). When the results of the MS analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the analysis is acceptable in a clean matrix. The LCS acceptance criterion is ± 15%, except for EPA method 200.5, which is ± 10%. In the case of soil matrices, the LCS consists of a purchased spiked soil control sample and the recovery criteria is 75-125%.

12.5.3 Matrix Spike (MS)- Assessing the effect of matrix should include the analysis of one matrix spike per batch of non-potable water or soil samples, or an MS every 10 samples for potable water analysis. The matrix spike evaluates both sample matrix affect and efficiency of sample preparation. For non-potable or soil samples, if recovery falls outside of ± 25%, the analyst must narrate. For drinking waters, the recovery must fall within 85 – 115%.

12.5.4 Sample Duplicates (Dup)- One field sample duplicate must be prepared and analyzed with every batch for precision information. The RPD between the sample and sample duplicate should not exceed 20%. Exceedences must be reanalyzed or narrated.

12.6 To evaluate the instrument calibration, the following are analyzed:

11.6.1 The calibration verification (ICV, CCV) standard is the mid-level standard which is analyzed after calibration and every 10 samples (acceptance criteria is ± 5% for Initial verification and ±10% for continuing verifications.)

11.6.2 The Calibration verification standard (CVS) is a second source standard that is analyzed prior to the samples to verify the validity of the calibration standards (acceptance criteria is ± 10% and ± 5% for drinking water and waste water by 200.7).

12.7 Lower level quantitation check standard (CRQL) is the lowest standard in the calibration curve, and 5x lower than the reporting level. It is analyzed at the beginning and end of each run, and every 8 hours.

12.8 Reporting level standard (CRDL) is analyzed at the beginning and end of each run, and every 8 hours. The acceptance criteria is ± 30%.

12.9 The spectral interference check standards (ICSA, ICSAB) are analyzed to verify the instrument background corrections and the interelement correction factors. These checks are analyzed at the beginning of each run, every 8 hours, and at the end of the run. The acceptance criteria is ± 20%.

12.10 Dilution Test- If the analyte concentration is within the linear range and sufficiently high, an analysis of a 1:5 dilution should agree within ± 20% or ± 10% for DW/WW by 200.7). A dilution sample is analyzed in every batch.

12.11 Post Digestion Spike- A post spike is analyzed every batch of samples. When performing a post-digestion spike, take 0.08 mL of each QCP-QCS-1 and QCP-QCS-2 and bring up to 8.0 mLs of sample. For soil matrix post digestion spike take 0.16 mL of each QCP-QCS-1 and QCP-QCS-2 and dilute to 8.0 mLs of sample. The acceptance criteria is ± 25% of the true value. Narrate if the post digestion spike fails criteria.

13.0 Safety

13.1 The toxicity and carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.

13.2 Always wear safety glasses for eye protection as well as a lab coat.

13.3 Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures.

14.0 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

14.2 Standards should be purchased in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

15.0 Waste Management

15.1 It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hood and bench operations. Compliance with all sewage discharge permits and regulations is also required.

16.0 Method Performance

16.1 This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.

16.2 See Section 11- Quality Control in this SOP for acceptance limits.

17.0 Corrective Action for Out-of-Control or Unacceptable Data

16.1 Should the calibration curve have a correlation coefficient of <0.9975, remake and reanalyze curve before processing samples.

16.2 Should the preparation blank, LCS, or in-house standards fail acceptance criteria, re-digest and re-analyze batch.

16.3 Should the matrix spike or sample duplicate analysis fail acceptance criteria, a non-conformance report must be generated or the sample QC must be reanalyzed. If a 200.5 (Lead and Copper) matrix spike fails criteria, the sample must be rerun by another approved method.

18.0 References

17.1 "Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Environmental Monitoring Systems Laboratory – Cincinnati (EMSL-CL), EPA-600/4-79-020, Revised March 1994 (Method 200.7 version 4.4).

17.2 "Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma- Atomic Emission Spectrometry", Environmental

Protection Agency – Cincinnati, EPA-600/R-06/115, October 2003 (Method 200.5 version 4.2).

17.3 "Test Methods for Evaluating Solid Waste (SW-846), Third Edition, EPA Office of Solid Waste, Final Update IV, (2/07), Method 6010C, "Inductively coupled plasma- atomic emission spectrometry".

17.4 "Test Methods for Evaluating Solid Waste (SW-846), EPA Office of Solid Waste, Final Update V, (7/14), Method 6010D, "Inductively coupled plasma- optical emission spectrometry".

17.5 SPECTRO Analytical Instruments Operator's Manual.

17.6 USEPA Method 29 - Determination of Metals Emissions from Stationary Sources, 8/2017

19.0 Tables

Table I: Troubleshooting

Fault	Cause	Remedy
Generator does not start	Power Supply	Check instrument power supply (fuses, main switch)
Error Message: Argon Pressure too low	Argon supply interrupted	
Error Message: Insufficient OPI cooling flow	Cooling agent supply interrupted.	• Check to ensure system (tubing etc) is not blocked (dirt, lime, deposits) • Check flow amount.
	Cooling agent supply pressure too low.	Increase inlet pressure (approximately 4 bar/60 psi)
Error Message: Unit door not closed.	Door not properly closed	Check closing mechanism

Error Message: Plasma exhaust error	Exhaust capacity too low	Check the unit filter for soiling or check the exhaust capacity.
Error Message: Current error on start	Error during start routine	Repeat start procedure

Plasma Torch melts	Torch incorrectly adjusted within the load coil.	Check whether the torch is positioned correctly.
	Aerosol tube blocked	Clean the torch.
	Leak in the argon connection to the torch.	Search for the leak with soapy solution (Snoop) and seal.
	Insufficient auxiliary gas flow setting.	Increase the auxiliary gas flow.
	Leak in the nebulizer system.	Search for leak with soapy solution (Snoop) and seal.
Plasma cannot be ignited	No Tesla discharge	Ignition cable not connected correctly
		Check the insulation for correct installation
	Torch not correctly centered within the load coil	Check the load coil setting
	Oxygen in sample intro-duction system	Flush the sample introduction system with argon
	Wrong argon quality	Argon with a quality of 4.6 or better is required (purity = 99.996%)

Plasma asymmetrical	Argon flush out of the OPI/SPI too high	Reduce the flow rate
	Plasma chamber extraction set too high	Reduce extraction
Plasma flickers or pulsates	Humidity or deposits in the aerosol tube of the torch.	Clean and dry the torch or replace it.
	Aerosol pulsates	• Clean the nebulizer • Replace the pump tube.
Plasma too bright	Power set too high	Decrease power
	Oxygen in sample intro-duction system.	Check for leaks and seal
Plasma extinguishes when nebulizer gas flow is switched on	Sample introduction tube not connected to the nebulizer.	Connect the tube and check fitting
	Oxygen in sample intro-duction system	Flush the system with argon before plasma ignition
	Leak in the sample intro-duction system	Find the leak and seal
No channel visible in the plasma	Nebulizer gas flow setting too low	Optimize the nebulizer gas flow
	Nebulizer blocked	Clean the nebulizer
	Leak in spray chamber outlet	Find the leak and seal
Error Message: Waste container level	Waste container is full	Remove the cover of container and properly dispose of the

indicator		content
Sparking-over at the load coil	Humidity	Turn off generator and dry the load coil with tissue paper (if humidity is visible)
	Dirt on the load coil	Switch off the generator and clean the load coil using a toothbrush.
Error Message: A new ICALization is required	Control peak drift detection	ICALize
Error Message: ICALization failed	Incorrect sample or incorrect flush time	Use the correct sample. Check the measurement parameters. Repeat the ICALization
	In the current method, no ICAL reference spectrum is defined	Select an ICAL reference spectrum for the method under method development
Error Message: ICAL reference does not fit to current transformation data.	A method with other ICAL basic parameters was loaded	ICALize the new method with the correct nebulizer type.
Error Message: Network Connection No communication between the PC and the unit.	IP address incorrect or used for a different purpose	Enter the correct IP address (online help). Only qualified IT staff should enter the address.

Revision History Log for SOP #506

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
8/7/14	1	New SOP for Blue instrument, broken out from SOP#505	Laura Kinnin	Kathy Cressia	8/7/14
7/29/15	1.1	Sections 11.2.2 & 11.3.3- fixed 200.9 typo, Section 8.5.1.3- clarified ICV limits +/- 5% and CCV limits +/- 10%, Section 8.5.1.2- made CC match Section 16.1, which is >0.9975 and fixed formatting issues throughout SOP	Kathy Cressia	Phyllis Shiller	7/29/15
3/15/16	1.2	Added HCl throughout the entire SOP, CLP 1+ 3 changed volumes, CVS new source, deleted section 8.3.6.2	Laura Kinnin	Kathy Cressia	3/15/16
5/18/16	1.3	Combined SOP 505/506 together to have one ICP SOP. Added Arcos to Instrument list. Changed LCS second source to a CVS throughout SOP, and LCS now a blank spike. Section 8.5.1.1- put ICAL standards in order, Section 11.0- rewrote entire section using the terms we call QC in the batches and in the runs. Added information on where they come from and their criteria. Section 17.4- added 6010D method reference	Kathy Cressia	Phyllis Shiller	5/18/16
1/10/20	1.4	Added air to scope Added section 9 and 9.1 for Lead in air calculation. Added USEPA Method 29 to references	Kathy Cressia	Phyllis Shiller	1/10/20

Expiration Date

3/31/20	**1.5**	**Added section 5.3 for new ICP Arcos 2 instrument**	**Kathy Cressia**	**Phyllis Shiller**	**3/31/20**

PHOENIX
Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
Telephone: 860.645.1102 • Fax: 860.645.0823

Effective Date: 8/28/19
Version Number: 6.8
Initiated By:
Approved By:

SOP No.: 621.8081

Title: Determination of Organo-chlorine Pesticides in soil and water matrices by GC-ECD

1.0 Scope and Application

This procedure covers the determination of certain Organo-chlorine pesticides. The following parameters that are determined by this procedure are as follows:

Alachlor	Endosulfan II
Aldrin	Endosulfan sulfate
Alpha-BHC	Endrin
Beta-BHC	Endrin Aldehyde
Delta-BHC	Endrin Ketone
Gamma-BHC	Heptachlor
Alpha Chlordane	Heptachlor Epoxide
Gamma Chlordane	cis-Nonachlor
Chlordane	trans-Nonachlor
4,4'-DDD	Methoxychlor
4,4'-DDE	Oxychlordane
4,4'-DDT	Toxaphene
Dieldrin	Hexachlorobenzene
Endosulfan I	Mirex

This gas chromatographic (GC) procedure is applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR part 136.1, and in groundwater, surface water, and soil, as described by EPA method 8081B. This procedure describes analytical conditions for a second gas chromatographic column that is used to confirm measurements made with the primary column. In addition, 40 CFR part 136.1, PCBs are also evaluated. In the presence of PCBs, refer to SOP 622-8082 for method specific criteria.

2.0 Summary of Method

2.1 A measured volume of sample, approximately 1 L for waters or 15-30 g for soils, is extracted with methylene chloride. The methylene chloride extract is dried and exchanged to hexane and brought to a final volume

of 5.0 mL. The extract undergoes clean up, if necessary, and is separated by gas chromatography and the concentrations are measured with an electron capture detector. Target compounds are confirmed by dual column analysis.

3.0 Interferences

3.1 Interferences may by caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interference under the conditions of the analysis by running laboratory reagent blanks. A blank is prepared daily for each batch of extractions.

3.2 Interferences by phthalate esters can pose a major problem in pesticide analysis when using the electron capture detector. These compounds generally appear in the chromatogram as large late eluting peaks, especially in the 15 and 50% fractions from Florisil. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding the use of plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

3.2 Matrix interference may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled.

4.0 Sample Collection, Preservation and Storage

4.1 Sample extracts must be stored at 4°C and analyzed within 40 days of extraction.

5.0 Equipment and Supplies

5.1 Also refer to extraction SOP 239.Sep, SOP 240.Liq/Liq or SOP 235.ASE.

5.2 Gas Chromatograph

5.2.1 Perkin Elmer Auto-system GC with Dual Electron Capture Detectors.

5.3 Columns:

5.3.1 Column 1: RTX-CLPesticides, 30m X 0.32mm ID X 0.32 micron film thickness (Restek #11141).
5.3.2 Column 2: RTX-440, 30m X 0.32mm ID X 0.25 micron film thickness (Restek #12924).
5.3.3 Restek Siltek Guard Column, 5m X 0.53mm ID (Restek #10028)
5.3.4 Siltek MXT Connector Kit (Restek #21388)

5.4 Gas – Carrier and ECD Makeup

5.4.1 Ultra High Purity Helium (carrier gas)
5.4.2 Liquid nitrogen (ECD makeup)

5.5 Repipettor- VWR, 5 mL
5.6 Hamilton Gas Tight syringes, assorted
5.7 Vials, 16 mL teflon cap, Kimble or equivalent
5.8 Vials, 2 mL, crimp, Proline or equivalent

6.0 Reagents and Standards

6.1 Hexane-Pesticide Quality
6.2 Organochlorine Pesticide Mixture (Ultra PPM-808C)
6.3 Pesticides Stock Surrogate Standard Solution (Ultra ISM-320)
6.4 Pesticide Degradation Standard (Ultra ISM-450)
6.5 Toxaphene at 1000 ug/mL (Absolute 79178)
6.6 Toxaphene, 2nd source at 1000 ug/mL (Restek 32005)
6.7 Chlordane at 100 ug/mL (Ultra PP-150)
6.8 Chlordane, 2nd source at 1000 ug/mL (Restek 32021)
6.9 Alachlor at 5000 ug/mL (Ultra EPA-1068)
6.10 Dieldrin at 100 ug/mL (Ultra PP-190)
6.11 g-BHC at 100 ug/mL (Ultra PP-140)
6.12 Aldrin at 100 ug/mL (Ultra PP-110)
6.13 Pesticide Laboratory Control Sample (Restek #32291)- Prep Dept.
6.14 Organochlorine Pesticide Mix AB #3 (restek 32415) (CVS stock standard)
6.15 Internal Standard- 1-Bromo-2-nitrobenzene in acetone, 5000ug/mL (Ultra PPS-351)
6.16 Hexachlorobenzene at 1000 ug/mL (Absolute 70195)
6.17 Hexachlorobenzene 2nd source at 1000 ug/mL (Ultra EPA-1125)

6.18 Mirex 2nd source at 100 ug/mL (Absolute X9232)
6.19 Mirex at 100 ug/mL (Ultra PST-720M100A01)
6.20 cis-Nonachlor at 1000 ug/mL (Absolute 71237)
6.21 trans-Nonachlor at 1000 ug/mL (Absolute 70235)
6.22 Oxychlordane isomer at 100 ug/mL (Accustandard P-3315)
6.23 cis-Nonachlor at 100 ug/mL (Ultra PP-490)
6.24 trans-Nonachlor at 100 ug/mL (Ultra PP-500)
6.25 Oxychlordane at 100 ug/mL (Ultra PP-541)

Stock standard solutions must be replaced every 3 – 6 months and always before manufacturer's expiration.

7.0 Definitions

7.1 Internal Standard (IS) -- A pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes and surrogates that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

7.2 Surrogate -- A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

7.3 Laboratory Preparation Blank -- An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The laboratory prep blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

7.4 Sample Matrix Spike / Matrix Spike Duplicate (MS/MSD) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations.

7.5 Stock Standard Solution -- A concentrated solution containing one or more method analytes prepared in the laboratory using reference materials purchased from a reputable commercial source.

7.6 Calibration Standard -- A solution prepared from the dilution of stock standard solutions. The calibration standard solutions are used to calibrate the instrument response with respect to analyte concentration.

7.7 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD) -- A solution of method analytes of known concentrations which is used to fortify an aliquot of reagent blank. The LCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

8.0 Calibration and Standard Curves

8.1 Internal Standard Solution (ECD IS)- Dilute 100 uL of Ultra PPS-351 to 100 mL Hexane.

8.2 Surrogate Solution (1:10)- Dilute 100 uL of Ultra ISM-320 to 1 mL Hexane.

8.3 Pesticide Working Standard- Dilute 50 uL of Ultra PPM-808C, 50 uL of Absolute 70195, and 500 uL of Ultra PST-720M100A01 to 10 mL Hexane.

8.4 Mirex Solution (1:10)- Dilute 100 uL of Absolute X9232 to 1 mL Hexane.

8.5 Toxaphene – Alachlor Working Standard- Dilute 0.5 mL Ultra EPA-1068 and 1.0 mL Absolute 79178 to 5 mL Hexane.

8.6 Pesticide Calibration Verification (CVS) Working Standard– Dilute 100uL Organochlorine Pesticide Mix AB #3 (restek 32415) plus 200uL Hexachlorobenzen 2nd source (Ultra EPA=1125) to a final volume of 10ml in hexane.

8.7 Pesticide Calibration Verification (CVS)- Dilute 25uL CVS working standard, 40uL surrogate solution (1:10), and 50ul mirex solution (1:10) to a final volume 10ml in hexane plus 100ul ECD IS.

8.8 Prepare Calibration Curves

8.8.1 Mixed Pesticide Calibration Curve:

Std #	uL Working Standard (section 8.3)	uL Surrogate (section 8.2)	uL Internal Std. (section 8.1)	Final Vol. mL (Hexane)	Final Conc. ppb
1A	4	5	100	10	2
1	10	10	100	10	5
2	20	20	100	10	10
3	50	30	100	10	25
4	100	40	100	10	50
5	150	50	100	10	75
6	200	60	100	10	100

8.8.2 Degradation Standard PreMix- An additional standard (1G) is prepared for Aldrin, Dieldrin, g-BHC, and Mirex. Make a 1:10 dilution of Ultra PP-110, Ultra PP-190, Ultra PP-140, and Ultra PST-720M100A01 by adding 10 uL of each to a final volume of 1 mL Hexane.

8.8.3 Degradation/1G Standard- Dilute 500 uL Ultra ISM-450, 15 uL of Degradation PreMix, 40 uL of Surrogate to a final volume of 10 mL in Hexane plus 100ul internal standard solution. This yields a final concentration of 1.5 ppb for Aldrin, Dieldrin, g-BHC, and Mirex.

8.8.4 Chlordane CVS- Dilute 100 uL Restek 32021 to a final volume of 1.0 mL Hexane. Take 30 uL of this solution, add 30 uL Surrogate Solution, and dilute to 10 mL Hexane plus 100ul internal standard solution.

8.8.5 Chlordane Standard Curve:

uL Chlordane Standard (section 6.6)	uL Surrogate (section 8.2)	uL Internal Std. (section 8.1)	Final Vol. mL (Hexane)	Final Conc. ppb
2	5	100	10	20
10	10	100	10	100
20	20	100	10	200
30	30	100	10	300

40	40	100	10	400
50	50	100	10	500
60	60	100	10	600

8.8.6 Toxaphene CVS- Dilute 200 uL Restek 32005 to a final volume of 1.0 mL Hexane. Take 50 uL of this solution, add 30 uL Surrogate Solution and dilute to 10 mL Hexane plus 100ul internal standard solution.

8.8.7 Toxaphene / Alachlor Standard Curve:

Std #	uL Working Standard (section 8.4)	uL Surrogate (section 8.2)	uL Internal Std. (section 8.1)	Final Vol. mL (Hexane)	Final Conc. Alachlor ppb	Final Conc. Toxaphene ppb
5	100	50	100	10	50	2000
4	80	40	100	10	40	1600
3	50	30	100	10	25	1000
2	20	20	100	10	10	400
1	10	10	100	10	5	200

8.8.8 Pest Supplemental CVS Primary Standard: Dilute 10 uL each of cis-Nonachlor (Absolute 71237), trans-Nonachlor (Absolute 70235), and 100 uL of Oxychlordane isomer (Accustandard P-3315) to a final volume of 1.0 mL in Hexane.

8.8.9 Pest Supplemental CVS: Dilute 50 uL of Supplemental CVS Primary Standard (Section 8.7.8), 40 uL Surrogate solution, and 100 uL of internal standard to a final volume of 10 mL in Hexane.

8.8.10 Pest Supplemental Working Standard: Dilute 50 uL each of cis-Nonachlor (Ultra PP-490), trans-Nonachlor (Ultra PP-500), and Oxychlordane isomer (Ultra PP-541) to a final volume of 1.0 mL in Hexane.

8.8.11 Pest Supplemental Curve for cis-, trans-Nonachlor, Oxychlordane:

uL Supplemental Standard (section 8.7.10)	uL Surrogate (section 8.2)	uL Internal Std. (section 8.1)	Final Vol. mL (Hexane)	Final Conc. ppb
5	5	100	10	2.5
10	10	100	10	5.0
20	20	100	10	10.0
50	30	100	10	25.0
100	40	100	10	50.0
150	50	100	10	75.0
200	60	100	10	100

9.0 Procedure

9.1 Gas Chromatograph Conditions

9.1.1 Injector Temperature: 250 degrees C

9.1.2 Detector Temperatures: 380 degrees C

9.1.3 Carrier Gas: UHP Nitrogen at 14.0 psi

9.1.4 Make Up Gas: UHP Nitrogen at 80-100 mL/min

9.1.5 Temperature program:

Rate	Temp	Hold
	130°	0 min
23.0	240°	0 min
9.0	260°	0 min
25	330°	3.20 min

Total Run Time = 13.00 minutes

9.1.6 Injection Volume: 1uL

9.1.7 Detector "A": CLP; Detector "B": RTX-440

9.2 Daily Degradation Standard Check

9.2.1 Inject 2uL of degradation standard. Measure the degradation of 4,4'-DDT to its degradation products 4,4'-DDE and 4,4'-DDD. Measure the degradation of Endrin to its degradation products Endrin Aldehyde and Endrin Ketone. See Sec. 10 for calculation formula. The degradation products should be no more than 20%. These criteria must be met at the beginning of each 12-hour interval of sample analysis.

Corrective Action: If the products exhibit a degradation of greater than 20%, replace liner in the injection port along with the septum and cut off

6 inches of the column. If it passes after reinjection, proceed with analysis, if it does not, see department supervisor.

9.3 Initial Calibration

9.3.1 Prepare the four calibration curves described in section 8.7. The % RSD for each analyte of interest must be below 20% for method 8081.

Corrective Action: If the % RSDs are greater than allowed, clean injector port, clip a loop off the column from the injector side and recalibrate.

9.4 Continuing Calibration

9.4.1 A calibration check standard is to be run, at a minimum, every 12 hours or 20 injections, whichever occurs first. The check standard that should be used is that of level #4, which lies at the middle of the curve. The percent difference cannot exceed 20%.

9.5 Method Blank

9.5.1 A method blank for each batch of extractions is to be run along with the samples. A method blank must be extracted daily for each type of extraction and every 20 samples.

9.6 Matrix Spike/Matrix Spike Duplicate

9.6.1 One MS/MSD per matrix must be run every 20 samples. Recoveries should fall between the laboratory guidelines. Spiked samples do not require re-analysis if matrix interferences are visibly present. Spike level is at 500ppb for soils and 50ppb for waters.

9.7 Laboratory Control Spike

9.7.1 One LCS/LCSD sample per matrix is to be run every 20 samples. LCS/LCSD spike level is at 500ppb for soils and 50ppb for waters.

9.8 Analysis Summary

9.8.1 Run solvent blank (Hexane).
9.8.2 Run degradation standard; calculate degradation products.
9.8.3 Run calibration check standard.

9.8.4 Run Supplemental calibration check standard.
9.8.5 Run Chlordane & Toxaphene check standards.
9.8.6 Run method blank(s), samples and quality control (10)
9.8.7 Run solvent blank (Hexane).
9.8.8 Run calibration check standards.
9.8.9 Run Supplemental calibration check standard.
9.8.10 Run Chlordane & Toxaphene check standards.
9.8.11 Run samples and closing calibration check standards within 12 hour clock from degradation standard.

9.9 Sample Quantitation and Validation

9.9.1 Samples are to be calculated by average response factors over a curve as described in Section 8.6. Sample concentrations greater than 10% of the highest standard must be diluted to fall in the midpoint range of the curve. Dilutions are to be made with hexane or MTBE. For Toxaphene and Chlordane, identification is based on a characteristic fingerprint. Chlordane quantitation is based on the sum of 2-3 signature peaks. Toxaphene quantitation is based on the sum of all peaks falling within the reference Toxaphene retention time window, determined at calibration.
9.9.2 Samples are quantitated from the response on Channel A by retention time compared to that of the known standard. The positive hits are confirmed on Channel B.

10.0 Calculations

10.1 Determine the concentration of the individual compounds in the sample from Turbochrom in (ppb) from the report.

10.2 Final Connection in ppb (ug/Kg or ug/L):

$$\frac{\text{ppb from TC} * \text{Final Volume (mL)} * \text{Dilution Factor} * 100}{\text{Initial Weight (g) or Volume (mL)} * \% \text{ solids}}$$

11.0 Quality Control

11.1 All standards are labeled, where applicable, with date received, date opened, analyst initials, expiration date, analyte name, analyte concentration and lot number.

11.2 An initial demonstration of capability must be performed to prove the generation of acceptable data with regard to accuracy and precision. An initial demonstration must be done for each new analyst, whether in the prep or instrument departments.

11.3 A continuing demonstration of capability or accuracy and precision study must be performed annually. Four standards at a level approximately ten times higher than the detection limit are evaluated for accuracy (% recovery) and precision (standard deviation). The RSD must be <20%.

11.4 A Method Detection Limit or a Limit of Detection Study is performed annually. For a MDL, at least seven blanks are spiked at a level 2-5 times the expected MDL. The standard deviation of the seven or more analyses is multiplied by the degrees of freedom to obtain the calculated method detection limit. Refer to 40 CFR Part 136 Appendix B. The Limit of Detection Study requires analyzing a standard that is below the Reporting Level (LOQ) and calculating its recovery. LOD standards must be 50 – 150%.

11.5 Prep Blanks- Before processing any samples, the analyst must demonstrate that all glassware and reagent interferences are under control. Each time a batch of samples is extracted, a prep blank must be analyzed. If within the retention time window of any analyte of interest the LRB produces a peak that would prevent the determination of that analyte, determine the source of contamination and eliminate the interference before processing samples.

11.6 Matrix Spike and Matix Spike Duplicate- Two fortified environmental samples (labeled MS and MSD) are analyzed every batch of 20 samples. Thus 10% of all samples are represented by a fortified sample. Calculate the percent recovery for each analyte by dividing the result by the true value. The recovery must be between 30-150% recovery for MS/MSD. If the recovery of any such analyte falls outside the control limits and the LCS for that analyte is shown to be in control, the recovery problem encountered with the MS/MSD is judged to be matrix related, not system related. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

11.7 Laboratory Control Sample and Laboratory Control Duplicate- Two fortified blank samples (labeled LCS and LCSD) are analyzed every batch of 20 samples. Calculate the percent recovery for each analyte by dividing the result by the true value. The recovery must be between 40-140% recovery in the LCS/LCSD. If the recovery of any such analyte falls outside the control limits, the batch needs to be reprepped.

11.8 Surrogate Recoveries

11.8.1 When surrogate recovery from a sample or prep blank is <30%, it must be reprepped. If a surrogate recovery is >130%, it must be narrated.

11.8.2 If sample extract reanalysis meets the surrogate recovery criterion, report only data for the reanalyzed extract. If sample extract reanalysis continues to fail the surrogate recovery criterion, report all data for that sample as suspect.

11.9 See Appendix I for criteria summary chart for more information.

12.0 Safety

12.1 The toxicity and carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.

12.2 Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures

13.0 Pollution Prevention

13.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

13.2 Reagents and standards should be purchased and/or prepared in volumes consistent with laboratory use to minimize the volume of disposal.

14.0 Waste Management

14.1 It is the laboratory's responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

15.0 Method Performance

15.1 This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.

15.2 See Sections 9.0, 10.0, and 11.0 in this SOP for acceptable limits.

16.0 Corrective Action for Out-of-Control or Unacceptable Data

16.1 See Sections 9.0, 10.0, and 11.0 in this SOP for corrective actions.

17.0 References

17.1 EPA Test Methods for Evaluating Solid Waste, SW-846 Third Edition, Update IV, February 2007, Method 8081B.

17.3 State Of Connecticut Department of Environmental Protection, Recommended Reasonable Confidence Protocols (RCP), Quality Assurance and Quality Control Requirements Pesticides by Method 8081, SW-846 Version 2.0 July 2006.

17.4 Quality Control Requirements and Performance Standards for the Analysis of Chlorinated Pesticides by Gas Chromatography (GC) in Support of Response Actions under the Massachusetts Contingency Plan (MCP), WSC–CAM, Final July 2010.

Revision History Log for SOP #621

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
9/25/14	6	Added new curves, added 1G Std, added LOD study, RCP/MCP to reference, Appendix I	Carol Eddy	Kathy Cressia	9/25/14
12/18/15	6.1	Added Mirex to SOP, new column, new conditions, Mirex solutions and standards	Carol Eddy	Kathy Cressia	12/18/15
05/05/16	6.2	Added Sec. 5.3.3 and 5.3.4- new guard column	Carol Eddy	Keith Aloisa	05/05/16
3/24/17	6.3	Section 9.4.1- added 20% for method 8081	Carol Eddy	Kathy Cressia	3/24/17
5/16/17	6.4	Section 6 & Section 8- added Toxaphene and Chlordane 2nd sources	Carol Eddy	Kathy Cressia	5/16/17
2/23/18	6.5	Deleted all references to method 608, changed column 2 name & part number, added sections 6.20 – 6.25 for cis, trans nonachlor & oxychlordane standards and second sources, added 8.7.8 – 8.7.11 for these extra pests- CVS and curve, section 9.3.1- changed 3 curves to 4, section 9.8- added supplemental pests Cal checks to run order	Carol Wohlmuth	Keith Aloisa	2/23/18
03/14/18	6.6	Section 8.7.1- mixed Pest standard conc of 1A changed to 2.0 ppb	Carol Wohlmuth	Keith Aloisa	03/14/18
5/13/19	6.7	Section 8.7.5 – Changed first row of table	Carol Wohlmuth	Keith Aloisa	5/13/19
8/28/19	6.8	Section 6.7, 6.8, 6.14, 6.16, 6.17 were updated. Section 8.6 & 8.7 were added for CVS info. Section 8.8.2, 8.8.3 & 8.8.4 were updated w/	Adam Werner	Kathy Cressia	8/28/19

		more info on internal standard solution. Section 9.1.5 Temp program was added.			

PHOENIX
Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
Telephone: 860.645.1102 • Fax: 860.645.0823

Effective Date: 11/17/16
Version Number: 4.1
Initiated by:
Approved by:

SOP No.: 251.Soxhlet

Title: Soil/Caulk Extraction by Soxhlet Method 3540C

1.0 Scope and Application

1.1 Method 3540 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, solids (including caulks) and wastes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

1.3 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 Summary of Method

2.1 A representative sample size, usually 15 g of soil sample or 3g of caulk, is prepared for extraction by mixing the sample with 10 g of sodium sulfate. The sample is then ground and loaded into the thimble, and extracted using an appropriate solvent in a Soxhlet extractor.

2.2 The extract is then dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

3.2 Phthalate esters contaminate many types of products commonly found in the

laboratory. Plastics, in particular, must be avoided.

3.3 Soap residue, which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Specifically, Aldrin, Heptachlor, and most organophosphorus pesticides will degrade in this situation.

4.0 Apparatus and Materials

4.1 Soxhlet extractor unit with condensers

4.2 Wooden Tongue Depressor (One Use)

4.3 Razor blades (One Use)

4.4 Weigh / transfer dishes (One Use)

4.5 Analytical Balance-capable of weighing to 0.01g.

4.6 250-mL round bottom flask with covers/wraps

4.7 Beakers, 250-mL

4.8 Boiling chips

4.9 Thimbles (One Use)

4.10 Buchi Syncore Concentration system

5.0 Reagents

5.1 Drying Agent- Sodium Sulfate (granular anhydrous), Na_2SO_4-Brand-Nu (337509) or equivalent.

5.2 Extraction Solvents-The extraction solvent to be employed depends on the analytes to be extracted, as described below. All solvents are pesticide quality or equivalent.

Hexane-BJ217 or equivalent
Acetone-BJGC010 or equivalent
Methylene Chloride-BJ300 or equivalent

5.2.1 Soil, sediment, and aqueous sludge samples are extracted with 1:1 acetone/hexane.

5.2.2 Other matrixes (like caulks and paint) are extracted in methylene chloride.

5.3 Nitrogen: Air Gas 0.999 Pure

5.4 Surrogates

5.4.1 SVOA= Ultra CUS-8804. Use 0.5mL.

5.4.2 PCB/Pest= Ultra ISM-320- Dilute 1mL Ultra ISM-320 into 100mL Acetone. Use 1.0mL.

5.4.3 PCB/Pest Massachusetts samples= Ultra ISM-320- Dilute 1.0mL Ultra ISM-320 into 100mL Acetone. Use 0.50mL.

5.4.4 NPD= Ultra PPS-100- Dilute 1mL of Ultra PPS-100 into 20mL of Acetone. Use 0.5mL of this solution.

5.4.5 EPH and NJEPH= Ultra ISM-581X- Use 2mL.

5.4.6 TPH= Aldrich 286931-5G, Pentacosane, 99%. Dilute 100mg pentacosane into 1 Liter 1:1 acetone/methylene chloride. Use 1mL.

5.4.7 PCB homologs (680)= 50uL of Ultra IST-440 into 1mL Acetone. Use 50uL.

5.5 Matrix Spike Solutions

5.5.1 SVOA= Supplied by instrument laboratory. Use 1mL of each.

5.5.2 Pest= Dilute 100ul of Restek #32415, 200uL of Absolute #79431, and 200uL of Absolute #70195 into 200mL of 1:1 hexane/acetone. Use 500uL of this solution.

5.5.3 PCB= Ultra CUS-14078. Use 500uL.

5.5.4 EPH= Dilute 4mL of Ultra SMA-300 + 4mL of Ultra SMA-310 into 100mL acetone. Use 2mL of this solution.

5.5.5 TPH, ETPH, and NJEPH= Dilute 1.0mL Restek #566330 into 100mL 1:1 acetone/methylene chloride. Use 1.0mL of this solution.

5.5.6 NPD= Dilute 100uL of Absolute #96127 into 20mL acetone. Use 500uL of this solution.

5.5.7 PCB homologs (680)= Ultra CB-681MN. Use 100uL.

6.0 Sample Collection, Preservation, and Handling

6.1 Soil samples are received in 4-8 oz jars with Teflon lids.

6.2 Caulk samples are received in baggies (the caulk can be wrapped in aluminum foil to minimize contact with the plastic baggie)

6.3 Hold Time is 14 days from day of collection.

7.0 Procedure

7.1 Sample preparation

7.1.1 Soils/Sediments: Weigh 15 g (or 10 g for EPH) soil into a 250mL beaker. Add approx. 10 g $NaSO_4$ and mix the sample thoroughly.

7.1.2 Caulk Samples: Approximately 3g of caulk is transferred on the disposable weigh dish. (Dish is tared and weight of caulk is transferred into the prep batch). The caulk sample is sliced into small sections with a disposable razor blade (one blade per sample).

7.1.3 Gummy, fibrous or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The analyst may add anhydrous sodium sulfate, palletized diatomaceous earth, sand, or other clean, dry reagents to the sample to make it more amenable to grinding.

7.2 Label round bottom flask with sample number. Add 90mLs of appropriate solvent through extractor and into a 250-mL round bottom flask containing one or two boiling chips. Check for cracks/leaks.

7.3 Transfer prepared sample into thimble. With the caulk samples, sodium sulfate is added directly into the thimble after the caulk.

7.4 Add the surrogates listed in sec. 5.4, to each sample. Add the matrix spike/matrix spike duplicate compounds listed in sec. 5.5 to the two additional aliquots of the sample selected for spiking.

7.5 Attach the condenser to the extractor and water supply. Wrap with covers. Extract the sample for 16 -24 hours at 4-6 cycles/hour at 175° for acetone/hexane, and 130° for methylene chloride.

7.6 Allow the extract to cool after the extraction is complete.

7.7 The extract is now ready for concentration, cleanup, solvent exchange or analysis, depending on the extent of interferents, and the determinative method to be employed. Refer to Method 3600 for guidance on selecting

appropriate cleanups methods. Excess water present in extract may be removed by filtering the extract through a bed of anhydrous sodium sulfate. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or sample analysis.

8.0 Quality Control

8.1 With every batch, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a solid matrix method blank (e.g., clean sand). Each time samples are extracted, and when there is a change in reagents, a method blank must be extracted and analyzed for the compounds of interest.

8.2 A matrix spike/matrix spike duplicate, or matrix spike and duplicate sample analysis, and a laboratory control sample are prepared and analyzed with every 20 samples or within 24 hours, which ever is more frequent.

8.3 Surrogate standards are added to all samples.

9.0 Definitions

9.1 Surrogate Analyte (SA) -- A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

9.2 Preparation Blank (PB) -- An aliquot of diatomaceous earth (DE) that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

9.3 Laboratory Fortified Sample Matrix and Matrix Duplicate (MS/MSD) -- An aliquot of an environmental sample to which known quantities of the method analytes are added. The MS/MSD samples are extracted and analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in

the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations.

10.0 Safety

10.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be minimized. The laboratory has a safety manual and MDSD volumes located in the general laboratory. It is important to read and understand these as well as the chemical hygiene plan.

10.2 Hexane is an extremely flammable liquid and harmful if swallowed, inhaled or absorbed through the skin. Methylene chloride is harmful if swallowed, inhaled or absorbed through the skin.

11.0 Pollution Prevention

11.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

11.2 Standards should be purchased in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

12.0 Waste Management

12.1 It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

13.0 Method Performance

13.1 This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.

14.0 Corrective Action for Out-of-Control or Unacceptable Data

14.1 The Organics Department will order a "re-prep", should batch QC or surrogate recoveries be unacceptable.

15.0 References

15.1 EPA SW-846 Test Methods for Evaluating Solid Waste, 3rd Edition, Update III, Dec 1996, Method 3540C

Appendix I – Prep Department Concentration Programs

Buchi Concentration Programs for Blow downs and Solvent Exchanges

Solvent	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6
ASE DCM	425 Mbar for 5 Mins	375 Mbar for 3 Mins	325 Mbar for 3 Mins	275 Mbar for 3 Mins	225 Mbar for 3 Mins	150 Mbar for 5 Mins
ASE Hex	200 Mbar for 3 Mins	150 Mbar for 2 Mins	100 Mbar for 4 Mins	50 Mbar for 3 Mins		
L/L DCM	450 Mbar for 2 Mins	400 Mbar for 5 Mins	350 Mbar for 6 Mins	300 Mbar for 9 Mins	250 Mbar for 8 Mins	100 Mbar for 3 Mins
SOX Hex	200 Mbar for 6 Mins	150 Mbar for 2 Mins	100 Mbar for 2 Mins	50 Mbar for 1 Mins		
SOXDCM	425 Mbar for 2 Mins	375 Mbar for 1 Mins	325 Mbar for 1 Mins	275 Mbar for 2 Mins	225 Mbar for 2 Mins	150 Mbar for 9 Mins

TurboVap Concentration Programs

Method	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8
#1 (all but 525)	2 Mins to 35 PSI	Hold for 45 mins						
#2 (for EPA 525)	8 PSI for 2 mins	10 PSI for 2 min	15.5 PSI- 5 min	21.5 PSI- 5 min	28 PSI- 5 min	32.2 PSI for 3 mins	35.7 PSI for 2 mins	Hold for 30 mins

NOTE: Any deviation from a program must to be documented in the associated Prep Batch for the affected sample(s).

Revision History Log for SOP #251.Soxhlet

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
11/12/13	4	Custom surrogates and spike solutions changed, added PCB homologs	Kathy Cressia	Tara Banning	11/12/13
11/17/16	4.1	Took Zymark out and added Buchi system, new surrogate volume for MA samples, new spiking solutions for Pesticides, new 680 surrogate solution, section 5.2 broke down between soil and caulk	Tara Banning	Phyllis Shiller	11/17/16

Expiration Date

SOP No.: 243.SPLP

Title: Synthetic Precipitation Leaching Procedure (SPLP)

Scope: The SPLP is designed to determine the mobility of both organic and inorganic analytes present in solids and waste materials. If a total analysis of the material demonstrates that individual analytes are not present in the material or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the SPLP need not be run.

1.0 Summary of Method

1.1 For liquid materials (i.e., those containing less than 0.5% dry solid material), the material, after filtration through a 0.6 to 0.8μm glass fiber filter, is defined as the SPLP extract.

1.2 For materials containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a pH 4.2 ±0.05 solution. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8μm glass fiber filter.

1.3 If compatible, the initial liquid phase of the sample is added to the liquid extract, and they are analyzed together.

2.0 Interferences

2.1 Potential interferences that may be encounter during analysis are discussed in the individual analytical methods.

3.0 Sample Collection, Preservation and Storage

3.1 Preservatives shall not be added to samples before extraction.

3.2 Samples may be refrigerated unless refrigeration results in irreversible physical change to the material. If precipitation occurs, the entire sample (including precipitate) should be extracted.

3.3 SPLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs. Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyses shall not be allowed to come into contact with the atmosphere to prevent losses.

3.4 Samples must undergo SPLP extraction within the following time periods:

	From: Field Collection To: SPLP extraction	From: SPLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis
Volatiles	14 days	NA	14 days
Semi-volatiles	14 days	7 days	40 days
Mercury (Hg)	28 days	NA	28 days
Metals, except Hg	180 days	NA	180 days

3.5 If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a sample does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the sample exceeds the regulatory level.

4.0 Equipment and Supplies

4.1 Agitation apparatus: The agitation apparatus is a wooden box with a motor that is capable of rotating the extraction vessel in an end-over-end fashion at 30+/- 2 rpm.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE)- This device is for use only when the sample is being tested for the mobility of volatile analytes. The ZHE allows for liquid/solid separation within the device, and effectively precludes headspace. The vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. The vessel has an internal volume of 500-600mL, and is equipped to accommodate a 90-110 mm filter. The devices contain Viton O-rings, which should be replaced frequently.

The ZHE should be checked for leaks on a weekly basis. Pressurize the device to 50psi, allow it to stand unattended for 1 hour, and recheck the pressure. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary.

4.2.2 Bottle Extraction Vessel- When the sample is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel. Plastic bottles shall not be used if organics are to be investigated.

4.3 Filtration Devices- It is recommended that all filtrations be performed in a hood.

4.3.1 Filtration flasks (acetoned rinsed for organics)
4.3.2 Buchner funnels (glass acetone rinsed for organics)
4.3.3 Filter paper, pore size of 0.6 to 0.8mm

4.4 pH Meter- The meter should be calibrated and accurate to $\pm$ 0.05 units.

4.5 VOA vials (for volatile extract collection)

4.6 Extraction bottles, 2L

4.7 Laboratory Balance

5.0 Reagents and Standards

5.1 Reagent Water (DI)

5.2 Sulfuric acid / Nitric acid (60/40 mixture)- Cautiously mix 60mL of concentrated sulfuric acid with 40mL of concentrated nitric acid. Record in reagent logbook.

5.3 Extraction fluid #1- In a PFTE carboy, add the 60/40 mixture of sulfuric and nitric acids to reagent water until a pH of 4.20 $\pm$ 0.05 is reached. Record date and pH in reagent logbook.

5.4 Extraction fluid #3- DI water only.

NOTE: The extraction fluid should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. **If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.**

6.0 Definitions

6.1 Reagent Water (DI)- Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest.

6.2 Synthetic Precipitation Leaching Procedure (SPLP)- a procedure used to evaluate the potential for leaching analytes or contaminants into ground and surface waters. This method provides a realistic assessment of mobility under actual field conditions. The extraction fluid is intended to simulate precipitation (acid rain).

6.3 Extraction Fluid #1- The fluid that is used to determine the leachability of soil or material from a site that is east of the Mississippi River. This extraction fluid has a final pH of 4.20.

7.0 Procedure

7.1 Determine whether the sample requires particle size reduction, which is required unless the solid has a surface area per gram of material equal to or greater than 3.1cm^2, or is smaller than 1cm in its narrowest dimension. If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the material to a surface area or particle size as described above. If the solids are prepared for organic volatile extraction, special precautions must be taken. See section 7.3.

7.2 Procedure when Volatiles are not Involved:

7.2.1 For liquid samples, filter enough sample to support the analyses to be performed on the SPLP extract. This should be done with Buchner funnels and filtering flasks. Make sure the glassware used for organic analyses is rinsed with acetone.

7.2.2 For solid samples, weigh out 100g per 2L bottle. Make sure enough sample is extracted to support the analyses to be performed on the SPLP extract. Also, make sure the glassware

used for organic analyses is rinsed with acetone. PFTE bottles are used for metals and mercury analyses only.

NOTE: Some samples, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.3 Determine the amount of extraction fluid to add to the extractor vessel by multiplying the amount of sample by 20.

7.2.4 Slowly add this amount of extraction fluid to the extractor bottle. Close the extractor bottle tightly, secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18± 2 hours. Ambient temperature shall be maintained during the extraction period. Record room temperature in the SPLP batching program.

7.2.5 Following the 18 ± 2-hour extraction, stop tumbler and record time and room temperature in the batching program. Also, determine the final pH of the sample and record.

7.2.6 Separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter. For final filtration of the SPLP extract the glass fiber filter may be changed, if necessary, to facilitate filtration.

7.2.7 Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. All other aliquots must be stored under refrigeration until analyzed. The SPLP extract shall be prepared and analyzed according to appropriate analytical methods.

7.3 Procedure when Volatiles are involved

7.3.1 Use the ZHE device to obtain SPLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes.

7.3.2 Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

7.3.3 Do not allow the sample, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary.

7.3.4 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample. Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.5 If the sample contains <0.5% solids, the liquid portion of sample, after filtration, is defined as the SPLP extract. Pre-weigh the (evacuated) filtrate collection container and set aside. Filter sample through a 0.6 – 0.8mm filter into a VOA vial. There should be no headspace.

7.3.6 If the sample is a solid: if needed, prepare for extraction by crushing, cutting, or grinding the solid portion of the material. Samples and appropriate reduction equipment should be refrigerated, if possible, to 4°C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in and of itself.

7.3.7 Weigh 20g of sample and quickly transfer to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

7.3.8 Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) closed, begin applying gentle pressure. When it reaches of 50 psi, open valve to bleed air out. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

7.3.9 After SPLP fluid #1 has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check

the ZHE to ensure that all valves are in their closed positions. Position the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi and slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are close.

7.3.10 Place the ZHE in the rotary agitation apparatus and rotate at 30 ±2 rpm for 18±2 hours. Ambient temperature shall be maintained during agitation. Record room temperature in batching program.

7.3.11 Check the ZHE to make sure pressure has been maintained. If it has not, perform the extraction again with new sample. If it has, the material in the extractor vessel is once again separated into its component liquid and solid phases. Filter through the glass fiber filter. Collect in a VOA vial.

7.3.12 Following collection of the SPLP extract, immediately prepare the extract for analysis and store with no headspace at 4°C until analyzed.

7.4 Procedure for Cyanide bearing material or Cyanide analysis

7.4.1 Extraction fluid #3 (reagent water) must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

8.0 Quality Control

8.1 A minimum of one blank, using the same extraction fluid as used for the samples, must be analyzed for every batch that have been conducted in an extraction vessel.

8.2 Matrix spikes are to be added after filtration of the SPLP extract and before preservation. Matrix spikes should not be added prior to SPLP extraction of the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The ambient room temperature shall be recorded when a batch of SPLP's are started and when they are taken down.

8.5 The agitation apparatus must be checked monthly to ensure that it is rotating at 30+/- 2 rpm. Record rotation per minute in logbook.

9.0 Safety

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be minimized. The laboratory has a safety manual and MSDS volumes located in the general laboratory. It is important to read and understand these as well as the chemical hygiene plan.

9.2 Always wear safety glasses for eye protection as well as nitrile gloves and lab coats. Refer to Phoenix SOP #805: Hazardous Chemical and Laboratory Safety Procedures.

10.0 Pollution Prevention

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

11.0 Waste Management

11.1 It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

12.0 Method Performance

12.1 This method is validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates for the individual analytes tested.

13.0 Corrective Action for Out-of-Control or Unacceptable Data

13.1 The Organics or Metals Department will order a "re-prep", should batch QC or surrogate recoveries be unacceptable.

14.0 References:

14.1 USEPA Test Methods for Evaluating Solid Waste, SW846, Synthetic Precipitation Leaching Procedure, Method 1312.

PHOENIX
Environmental Laboratories, Inc.

587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
Telephone: 860.645.1102 • Fax: 860.645.0823

SOP# 653.8260C/D

Standard Operating Procedure
for
VOA by GC/MS

Version 2.4

Initialized by:

Harry Mullin
12/12/19

Approved by:

Kathy Cressia
12/12/19

1. VOA by GC/MS

1.1 Title: MEASUREMENT OF VOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GAS CHROMATROGRAPHY/MASS SPECTROMETRY

2. Applicable Matrix or Matrices

2.1 This procedure is used for the identification and simultaneous measurement of volatile organic components in soils, ground waters, monitoring wells, surface waters, waste water, and wastes.

3. Detection Limit

3.1 The 25 ml purge has reporting levels between 0.5 and 2.5 ppb. The standard 5 ml purge has reporting levels between 0.7 and 25 ppb.

4. Scope and Application

4.1 This procedure is used for the identification and simultaneous measurement of volatile organic components in soils, ground waters, monitoring wells, surface waters, waste water, and wastes. Table 23.6 lists the compounds that the laboratory can report by this method.

5. Summary of Method

5.1 An inert gas is bubbled through the solution at ambient temperature and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column interfaced to a mass spectrometer. A temperature program is used in the gas chromatograph to separate the organic compounds that are then detected by the MS. Compounds eluting from the GC column are identified by comparing their measured mass spectra and retention times to reference spectra and retention times in a database.

5.2 Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. The concentration of each analyte is measured by the comparison of the relative response factor of the compound in the sample to the average relative response of the compound in the calibration standard. Surrogate components are measured with the same internal calibration procedure.

6. Definitions

6.1 Internal Standard (IS) -- A pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other

method analytes and surrogates that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

6.2 Surrogate -- A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the surrogate is to monitor method performance with each sample.

6.3 Laboratory Duplicates -- Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. The analyses of duplicates indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

6.4 Laboratory Reagent Blank -- An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The laboratory reagent blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.5 Field Blank (FB) -- An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

6.6 Sample Matrix Spike / Matrix Spike Duplicate (MS/MSD) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations.

6.7 Stock Standard Solution -- A concentrated solution containing one or more method analytes prepared in the laboratory using reference materials purchased from a reputable commercial source.

6.8 Calibration Standard -- A solution prepared from the dilution of stock standard solutions. The calibration standard solutions are used to calibrate the instrument response with respect to analyte concentration.

6.9 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD) -- A solution of method analytes of known concentrations which is used to fortify an aliquot of reagent blank. The LCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

7. Interferences

7.1 Major contaminant sources are volatile materials in the laboratory and impurities in the purging gas and sorbent trap. Analysis of calibration blanks, reagent blanks and trip blanks provide information about the presence of contaminants. Extra precautions are taken in the laboratory to eliminate common laboratory solvent contamination, such as methylene chloride.

7.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of these compounds. The autosampler is programmed to rinse between each sample numerous times to prevent this from occurring. Suspect samples are reanalyzed.

7.3 The purging device is cleaned and baked before each run to prevent contamination from samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high concentration samples.

8. Safety

8.1 The toxicity and carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. A reference file of material data handling sheets are available to all personnel involved in the chemical analysis.

8.2 Refer to SOP #805: Hazardous Chemical and Laboratory Safety Procedures

9. Equipment and Supplies

9.1 Syringes (microliter)

9.2 Syringe valve (2 way with Luer ends)

9.3 5mL syringe (gastight with shutoff valve)

9.4 Glass scintillation vials (40mL) with screw tips and Teflon liners

9.5 Volumetric flasks (100mL)

9.6 Disposable Pasteur pipets

9.7 Gas Chromatography/Mass Spectrometer/Data Systems and auto samplers

9.7.1 Agilent 5973 MSD with 6890 GC, Centurion Auto sampler, EST Encon Evolution Purge and Trap concentrators, HP Chemstation and Enviroquant software.

9.7.2 Agilent 5973 MSD with 6890 GC, Centurion Auto sampler, EST Encon Evolution Purge and Trap concentrators. HP Chemstation and Enviroquant software

9.7.3 Agilent 5972 MSD with 5890 GC, Centurion Auto-sampler, EST Encon Evolution Purge and Trap concentrators, HP Chemstation and Enviroquant software.

9.7.4 Agilent 5975 MSD with 7890 GC, Centurion autosampler, two Encon Evolution Purge and Trap concentrators.

9.7.5 Agilent 5973 MSD with 6890 GC, Centurion Auto-sampler, two EST Encon Purge and Trap concentrators, PT2 switching valve box.

9.8 Trap (VOCARB 3000 K)

9.9 Column RTX-VMS Restek Length: 20 meters; ID: 0.18 mm; Film: 1.0micron

10 Reagents and Standards

10.1 Organic free reagent water (Prepared with Barnstead water purification system)

10.2 Methanol (Purge and Trap Grade) demonstrated to be free of analytes.

10.3 Stock Standards- see section 7.1.1 for standard information.

11 Sample Collection, Preservation, Shipment and Storage

11.1 Samples must be collected in clear or amber glass VOA vials, 40-mL capacity, with polyethylene screw caps with fluoropolymer-lined (PTFE, Teflon) silicone septa. Aqueous samples must contain HCl preservative. Two VOA vials must be collected for each sample with no headspace.

11.2 For soil samples, collect one vial for high level and two vials for low level volatiles;

11.2.1 For low concentration soils- collect approximately 5-g sample, weighed in the field at the time of collection, and place it in a pre-weighed vial that already contains a stirring bar and 5 ml of water.

11.2.2 For high concentrations soils, collect approximately 10-g sample in a pre-weighed vial that contains 10 mL of methanol.

11.3 Samples must be stored at 4°C in the Volatile Laboratory refrigerators until the time of analysis. The holding time for samples is 14 days from collection. See SOP#102.5035. For unpreserved soil samples that are collected with EnCore samplers, analyze within 48 hours of sample collection, or freeze to below -7°C and analyze within 14 days.

12 Quality Control

12.1 Calibrate the mass and abundance scales of the MS with the BFB that is injected into each sample and acquire the mass spectra. If the spectrum does not meet all the criteria in Table IV, the MS must be retuned before running the initial calibration. The MS tune must be verified every 12 hours during which analyses are performed and adjusted to meet all criteria before the continuing calibration standard is analyzed.

12.2 Before running any samples, it should be demonstrated through the analysis of a reagent blank that interferences from the analytical system, glassware and reagents are under control. Each time there is a change in organic free reagent water, a blank should be run to ensure against chronic laboratory contamination. The blank samples are carried through all stages of the sample preparation and measurement steps. Blanks must also be analyzed at intervals throughout the sequence to demonstrate a continually contaminant free system. The frequency of these blanks should be at least 5% of all samples analyzed. Method blanks should contain no compounds over the reporting limit. When detected above the reporting level, all the samples have a "b" qualifier in the margin for that compound and a footnote explaining the non-conformance.

12.3 Initial Calibration

An initial calibration curve is constructed with a minimum of five concentrations.

The %Relative Standard Deviation criteria is <20%. Due to the large number of compounds, up to 10% of the compounds can have a RSD > 20% as long as the RSD is <40%.

12.4 After a calibration curve has been constructed, the curve must be checked with Laboratory Control Samples (LCS/LCSD). The LCS/LCSD is obtained from a vendor different than the calibration standards. The recovery for each analyte in the LCS/LCSD must be within 70-130% of the true value with no more than 10% of the analytes out of criteria in order to demonstrate that the curve is accurate. Do not proceed with analysis if the LCS/LCSD does not meet these criteria.

12.5 The calibration relationship established during the initial calibration must be verified at the beginning of each 12-hour period during which analyses are performed. Continuing calibration standards are prepared at a concentration near the midpoint of the initial calibration range

After analysis of the CCAL, the data file is loaded into the instrument batch. State specific criteria is evaluated and the batch is flagged green (passing) or red (failing).

RCP:
All 8260B CCCs and SPCCs must pass; 10% of other compounds can exceed a % deviation of 30 (as long as less than 40%).

MCP:
20% of compounds can exceed a % deviation of 20% (as long as less than 40% or curve 0.99).

If these criteria cannot be met, instrument maintenance (such as cleaning the ion source) and recalibration may be necessary.

12.6 The absolute areas of the quantitation ions for the internal standard of the samples are compared to the areas measured in the most recent continuing calibration check; any area that is <-50% or >+100% is

flagged red.

If this happens to an aqueous sample, the sample is re-analyzed at a dilution.

If this happens to a soil sample, the other low level is analyzed and/or the high level is analyzed. Sample data is reported from runs with passing IS area criteria.

Sample comments are made to explain why a dilution was required.

12.7 Sample surrogate recovery criteria is 70-130%. Should surrogate recoveries fall outside of range, re-analyze sample or narrate non-conformance.

12.8 For each analytical batch (up to twenty samples) a spike and spike duplicate must be analyzed. The acceptance criterion for spiked samples is 70-130% recovery with no more than 10% of the analytes out of criteria. The relative percent difference (%RSD) between the spike and spike duplicate must be <20% to demonstrate acceptable precision. If the MS/MSD does not meet this criteria, reanalyze or narrate non-conformance.

12.9 Manual integration is used only when a peak is not identified correctly. This may be a peak next to a similar peak and the software had identified the wrong peak or it may be poorly resolved peak. Manual integration can never be used to increase or decrease the area of a response.

12.10 A demonstration of capability is performed yearly and with each new employee. Four standards at a level approximately ten times higher than the detection limit are evaluated for accuracy (%recovery) and precision (standard deviation).

13 Calibration and Standardization

13.1 An extra blank, prior to tune and CCAL, is analyzed verify that the system is free of contamination from previous runs.

13.2 Change the trap after every 6-8 weeks or whenever low response of aromatics or tailing of late eluting compounds is observed.

13.3 Flow rates must be checked once in a month or whenever the trap is changed. Turn the standby feed pressure on and set system pressure to 20psi. Turn the standby feed pressure off and adjust the standby flow exiting of the vent. Set the standby flow to 40-45 ml/minute, set pressure to 3-5 psi keeping the standby flow at 40 ml/minute.

13.4 Cleaning of Ion Source is usually done after 6-8 weeks or as needed (e.g. when response drops and increasing multiplier voltage results in increase in multiplier noise resulting in a noisy baseline or when calibration peaks are deformed and ratios of ion spectra for PF43 cannot be properly adjusted by changing the MS Tune parameters.

13.5 Check mechanical pump oil every three months.

13.6 Gas purifiers no longer need to be changed: Helium gas tank has built in purifiers to provide the cleanest possible He gas for the instrument

14 Procedure

14.1 Standard Preparation

14.1.1 Preparation of Initial Calibration Standards

For each analyte of interest, prepare calibration standards at a minimum of five concentrations. The following is a list of the stock standards and concentrations used to prepare the calibration curve. This compound list represents the current standards; but is subject to change.

	Concentration	Part Number	Vendor
Oxygenate Custom	Various	64281	Absolute
Custom Std (6 analytes)	Various	CUS-11008	Ultra
1-4 Dioxane	10,000 ug/mL	95426	Absolute
Methylcyclohexane	1000 ug/mL	71627	Absolute
Methyl Acetate	1000 ug/mL	71031	Absolute
Carbon disulfide	1000 ug/mL	70060	Absolute
MTBE	1000 ug/mL	70209	Absolute
Ketones	2000 ug/mL	82402	Absolute
Cyclohexane	2000 ug/mL	96162	Absolute
502/524mix 54compounds	2000 ug/mL	32001	Absolute
502/524 6 compounds	2000 ug/mL	30058	Absolute
2-Chloroethylvinyl ether (2-CEVE)	1000 ug/mL	70074	Absolute
Vinyl Acetate	10000 ug/mL	RCC-218	Ultra
2-Nitropropane	5000 ug/mL	91102	Absolute

Working Calibration Standard: VOA-50PPM STOCK STANDARD

Component	Volume = 4mL
Meoh	380 uL
Oxygenate Custom	200 uL
Custom Standard	1000 uL
Vinyl Acetate	20 uL
1,4 Dioxane	400 uL
Methyl Cyclohexane	200 uL
Methyl Acetate	200 uL
Carbon Disulphide	200 uL
MTBE	200 uL
Ketones	100 uL
Cyclohexane	100 uL

Volatile 502/524	100 uL
Volatile Gases	100 uL
2-Nitropropane	40 uL

Table II lists concentration of analytes in the calibration curve.

14.1.2 Table III lists the volumes of stock standard used to prepare a 50ppm primary dilution standard.

14.1.3 Table IV lists the volumes of the primary dilution standard injected into 100mL of reagent water in a volumetric flask to achieve the concentrations listed in TABLE II. The contents of the volumetric flask are then poured into 2 VOA vials with zero headspace.

14.1.4 Table IV-A lists the volumes of Primary dilution standard injected into 5.0 ml of reagent water in low bleed septa VOA vials to achieve the concentrations listed in Table II. Standards prepared in VOA vials are analyzed with the soil sample for heated purge to comply with the method.

14.1.5 Laboratory Control Standard (LCS) Preparation

	Concentration	Part Number	Vendor
Oxygenate Custom	Various	64281	Absolute
Custom Standard	Various	CUS-11008	Ultra
1-4 Dioxane	2000 ug/mL	30287	Restek
Methyl Cyclohexane	1000 ug/mL	S-12469M4	Chem Service
Methyl Acetate	10000 ug/mL	N-12411	Chem Service
Carbon Disulfide	5000 ug/mL	EPA-1012	Ultra
MTBE	2000 ug/mL	STS-44051	Ultra
VOA calibration mix	5000 ug/mL	30006	Restek
Cyclohexane	1000 ug/mL	S-11526M4	Chem Service
VOC mix (54 compound)	2000 ug/mL	DWM-589N-1	Ultra
VOC Gas Mix	2000 ug/mL	DWM-544-1	Ultra
2-CEVE	1000 ug/L	70074	Absolute
Vinyl Acetate	10000 ug/mL	N-13746-1G	Chem Service
2-Nitropropane	5000 ug/mL	91102	Absolute

Tert-butanol and Methyl Acetate standards at a concentration of 10,000 ppm are prepared by weighing 0.1000 grams of neat compound and diluting to 10 mL with methanol.

LCS Working Standard: LCS 50PPM STOCK STANDARD

Component	Volume = 4mL
Meoh	1580 uL
Oxygenate Custom	200 uL
Custom Standard	1000 uL
Vinyl Acetate	20 uL
1,4-Dioxane	400 uL
Methyl Cyclohexane	200 uL
Methyl Acetate	20 uL
Carbon Disulphide	40 uL
MTBE	100 uL
Ketones	40 uL
Cyclohexane	200 uL
Volatile 502/524	100 uL
Volatile Gases	100 uL

14.1.6 Internal Standard/Surrogate Standard Preparation

Internal Standard Mixture 2000ppm(ULTRA CAT# STS-341N)
Internal Standard Mixture 2000ppm(ULTRA CAT# STS-210N)
Surrogate Standard Mixture2000ppm (ULTRA CAT# STM-330N)

1ml of each is added to 5ml of methanol to produce 250ppm stock.

14.2 Tuning the MS

Calibrate the mass and abundance scales of the MS. Purge 250ng of BFB and acquire MS data. The sample is split at the injector at least at a 1:25 ratio so the actual amount of BFB injected on column is comparable to 25ng. (Refer to Table V of this SOP for criteria). If spectrum does not meet the mass abundance criteria, the MS must be retuned before running initial calibration. The MS tune must be verified every 12 hours during which analyses are performed and adjusted to meet all criteria before the continuing calibration standard is analyzed.

14.3 Initial Calibration/Continuing Calibration

The initial calibration must meet appropriate requirements before samples are run. Continuing calibration checks must be run at the beginning of each 12 hour period during which analyses are performed.

14.4 Loading Samples/Standards

Place the appropriate VOA vial into the autosampler. The appropriate vial is determined by the ion Tiger portable PID screener. If the HL vial is to be used, it is vortex shaken for two minutes prior to taking an aliquot.

14.5 Purge and Trap Conditions (or equivalent)
Purge Volume: 5mL or 25 ml depending on instrument
Purge Ready Temp.:35°C
Purge Gas:Helium
Purge Time:8 minutes
Desorb Preheat:245°C
Desorb Time: 1 minutes
Desorb Temp.:250°C
Bake Time:6 minutes
Bake Temp:280°C
MCS Bakeout Temp.: 280°C
Transfer Line Temp.: 150°C

14.6 GC Temperature Program (or equivalent)
45°C hold for 2 minutes
Ramp 18°C/min to 130°C
Hold for 0.10 minutes
Ramp 40°C/min to 230°C
Hold for 1 minute
Constant flow through column is 0.8 ml/min., split ratio 1:40

15 Data Analysis and Calculations

15.1 Response Factor
Calculate the response factor (RF) for each analyte in each calibration standard using the internal standard.

$$RF = \frac{(Ax)\,(Qis)}{(Ais)\,(Qx)}$$

where:
Ax=Integrated abundance of the quantitation ion of the analyte in the sample
Ais =Integrated abundance of the quantitation ion of the internal standard in the sample
Qis=Total quantity (in micrograms) of internal standard added to the water sample
Qx =Quantity of analyte purged in concentration units

For each analyte and surrogate, calculate the average RF from analyses of calibration standards. Calculate the standard deviation (SD) and the relative standard deviation (RSD) from each average.
RSD = 100 (SD/average RF)

15.2 Internal Standard Calibration Technique
The internal standard calibration technique is used to calibrate the chromatographic system. The concentration of each compound in the sample is calculated using the results of the initial calibration.

$$\text{Concentration (ug/L)} = \frac{(Ax)(Qis)(1000)}{(Ais)(RF)(V)}$$

where:
V=Original water sample volume in mL

15.3 Q edit files prior to sending results.

15.4 Pick instrument run in the instrument batching screen.
Verify that CCALs and LCSs passed criteria.

Refresh all the RR files for the batch
Select sample for QC codes
Select RR files for data transfer and make any necessary comments.
"Over-range/carryover" causes that compound to be put on hold for re-run or dilution.
Transfer rr files
Go to result entry to drive the calculation spreadsheets and review results.

16 Method Performance

16.1 This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.

17 Pollution Prevention

17.1 No solvents are utilized in this method except the extremely small volumes of methanol needed to make calibration standards. The only other chemicals used in this method are the neat materials in preparing standards and sample preservatives. All are used in extremely small amounts and pose no threat to the environment.

18 Corrective Actions for Out-Of-Control Data

18.1 See the Quality Control section in this SOP for corrective actions.

19 Contingencies for Handling Out-of-Control or Unacceptable Data

19.1 The utmost care must be taken not to load samples if the instrument is not in complete control. However, there are circumstances, like a power outage, that may require the reporting of data that has not met all of the quality control criteria. In such cases, the problem and the affect on the data is recorded in the instrument batch, which becomes part of the narrative and/or a Corrective/Preventive Log entry.

20 Waste Management

20.1 Refer to SOP 703: Waste Disposal and SOP 807: Chemical Hygiene Plan

21 References

21.1 Test Methods for Evaluating Solid Waste (SW-846), Third Edition, EPA Office of Solid Waste, Final Update III December 1996 USEPA, SW-846 online- New Methods, "Volatile Organic Compounds by Gas Chromatography/Mass Spectroscopy (GC/MS)" 8260C Revision 3 August 2006.

21.2 Test Methods for Evaluating Solid Waste (SW-846), Third Edition, EPA Office of Solid Waste, Update VI, USEPA, SW-846 online- New Methods, "Volatile Organic Compounds by Gas Chromatography/Mass Spectroscopy (GC/MS)" 8260D Revision 4 June 2018.

22 Tables, Diagrams, Flowcharts and Validation Data

22.1 TABLE I

Surrogate Recovery Criteria

COMPOUND	% REC. WATER	% REC. SOIL
Dibromofluoromethane	70-130	70-130
Toluene-d8	70-130	70-130
4-Bromofluorobenzene	70-130	70-130
1,2-Dichloroethane-d4	70-130	70-130

22.2 TABLE II

CONCENTRATIONS OF ANALYTES IN 6 LEVEL CURVE for 5ml Purge

	Stryene and o-xylene	Acrolein	THF and trans-1,4-Dichlorobutene	ALL OTHER COMPOUNDS
Low STD	4 ppb	10 ppb	5 ppb	2.0ppb
LEVEL 1	10 ppb	25ppb	12.5 ppb	5.0ppb
LEVEL 2	20 ppb	50 ppb	25 ppb	10ppb
LEVEL 3	40 ppb	100ppb	50 ppb	20ppb
LEVEL 4	100 ppb	250 ppb	125 ppb	50ppb
LEVEL 5	200 ppb	500 ppb	250 ppb	100ppb
LEVEL 6	400 ppb	1000 ppb	500 ppb	200ppb

CONCENTRATIONS OF ANALYTES IN 6 LEVEL CURVE for 25ml purge

	Stryene and o-xylene	Acrolein	THF and trans-1,4-Dichlorobutene	ALL OTHER COMPOUNDS
Low STD	1 ppb	2.5 ppb	1.25 ppb	0.5 ppb
LEVEL 1	4 ppb	10 ppb	5 ppb	2 ppb
LEVEL 2	8 ppb	20 ppb	10 ppb	4 ppb
LEVEL 3	20 ppb	50 ppb	25 ppb	10ppb
LEVEL 4	40 ppb	100 ppb	50 ppb	20ppb
LEVEL 5	60 ppb	150 ppb	75 ppb	30 ppb

22.3 TABLE III

STOCK STANDARD VOLUMES USED TO PREPARE PRIMARY DILUTION STANDARD A

STANDARD	CONCENTRATION	VOLUME
Tert Butanol	2000 ug/mL	1.0mL
Custom Standard	Various concentrations	1.0mL
1-4 Dioxane	10000 ug/mL	400 uL
Methyl Cyclohexane	1000 ug/mL	200 uL

Methyl Acetate	1000 ug/mL	200 uL
Carbon disulfide	1000 ug/mL	200 uL
MTBE	1000 ug/mL	200 uL
Ketones	2000 ug/mL	100 uL
Cyclohexane	2000 ug/mL	100 uL
502/524 Mix (54 Comp)	2000 ug/mL	100 uL
502/504 (6 comp)	2000 ug/mL	100 uL
Methanol	Pure	380 uL
Vinyl Acetate	10,000 ug/mL	20 uL

22.4 TABLE IV

PRIMARY DILUTION STANDARD VOLUMES USED TO PREPARE THE SIX LEVEL CURVE FOR 5mL WATERS

uL of Primary Dilution	Final Volume in DI	Final Concentration
4 uL	100 mL	2 ppb
10 uL	100 mL	5 ppb
20 uL	100 mL	10 ppb
40 uL	100 mL	20 ppb
100 uL	100 mL	50 ppb
100 uL	50 mL	100 ppb
200 uL	50 mL	200 ppb

TABLE IV-A
PRIMARY DILUTION STANDARD VOLUMES USED TO PREPARE THE SEVEN LEVEL CURVE FOR SOIL SAMPLES (5g SOIL)

Standard # (Level)	Standard Volume (uL)	Final Volume H_2O (mL)	Volume (uL)in 5g Soil
2	4	100	2
5	10	100	5
10	--	--	1
20	--	--	2
50	--	--	5
100	--	--	10
200	--	--	20

TABLE IV-B
PRIMARY DILUTION STANDARD VOLUMES USED TO PREPARE THE SIX LEVEL CURVE FOR 25 ML PURGE

LEVEL DL	LEVEL 0.5PPB	LEVEL 2 PPB	LEVEL 4 PPB	LEVEL 10 PPB	LEVEL 20 PPB	LEVEL 30 PPB
uL of std	1.0 uL	4 uL	8 uL	20 uL	40 uL	60 uL

22.5 TABLE V

ION ABUNDANCE CRITERIA FOR BFB

MASS (M/z)	RELATIVE ABUNDANCE CRITERIA
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	100% Relative Abundance
96	5 to 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 to 9% of mass 174
176	> 95% but < 101% of mass 174
177	5 to 9% of mass 176

22.6 Table VI Analyte List for Method 8260

Compound	CAS Number
1,1,1,2-Tetrachloroethane	630206
1,1,1-Trichloroethane	71556
1,1,2,2-Tetrachloroethane	79345
1,1,2-Trichloroethane	79005
1,1-Dichloroethane	75343
1,1-Dichloroethene	75354
1,1-Dichloropropene	563586
1,2,3-Trichlorobenzene	87616
1,2,3-Trichloropropane	96184
1,2,4-Trichlorobenzene	120821
1,2,4-Trimethylbenzene	95636
1,2-Dibromo-3-chlorpropane (DBCP)	96128
1,2-Dibromoethane (EDB)	106934
1,2-Dichlorobenzene	95501
1,2-Dichloroethane	107062
1,2-Dichloropropane	78875
1,3,5-Trimethylbenzene	108678
1,3-Dichlorobenzene	541731
1,3-Dichloropropane	142289
1,4-Dichlorobenzene	106467
1,4-Dioxane	123911
2,2-Dichloropropane	594207
2-Butanone (MEK)	78933

2-Chloroethyl vinyl ether	110758
2-Chlorotoluene	95498
2-Hexanone	591786
2-Isopropyltoluene	527844
4-Chlorotoluene	106434
4-Isopropyltoluene	99876
4-Methyl-2-pentanone (MIBK)	108101
Acetone	67641
Acrylonitrile	107131
Benzene	71432
Benzyl chloride	100447
Bromobenzene	108861
Bromochloromethane	74975
Bromodichloromethane	75274
Bromoform	75252
Bromomethane	74839
Carbon Disulfide	75150
Carbon Tetrachloride	56235
Chlorobenzene	108907
Chloroethane	75003
Chloroform	67663
Chloromethane	74873
cis-1,2-Dichloroethene	156592
cis-1,3-Dichloropropene	10061015
Cyclohexane	110827
Dibromochloromethane	124481
Dibromomethane	74953
Dichlorodifluoromethane	75718
Diethyl ether	60297
Ethylbenzene	100414
Ethanol	64175
Hexachlorobutadiene	87683
Isopropylbenzene (Cumene)	98828
Methyl Acetate	79209
Methylcyclohexane	108872
Methylene Chloride	75092
Methyl-tert-butylether (MTBE)	1634044
m-Xylene [2]	108383
Naphthalene	91203
n-Butylbenzene	104518
n-Propylbenzene	103651
o-Xylene [2]	95476
p-Xylene [2]	106423

p-Isopropyltoluene	99876
Sec-Butylbenzene	135988
Styrene	100425
tert-Butanol (TBA)	75650
Tert-Butylbenzene	98066
Tetrachloroethene (Perc)	127184
Tetrahydrofuran (THF)	109999
Toluene	108883
trans-1,2-Dichloroethene	156605
trans-1,3-Dichloropropene	10061026
trans-1,4-Dichloro-2-butene	110576
Trichloroethene (TCE)	79016
Trichlorofluoromethane	75694
Trichlorotrifluoroethane (Freon-113)	76131
Vinyl Acetate	108054
Vinyl Chloride	75014
Tert-amyl alcohol	75854
Di-isopropyl ether	108203
Ethyl-Tert butyl ether	637923
Tert amyl methyl ether	994058
2-Nitropropane	79469

2. May be reported as total xylenes or any combination of the three isomers.

Changes Since Prior Version:

See revision history sheet for later versions.

Version 2.1

The SOP was taken out of LIMS MDL writer and back into word document form.
Changes were made to the following:
Section 3.1: 5mL range 0.7 – 25ppb
Section 11.3: Added information on hold time for EnCores
Section 14.1.5: 1,4-Dioxane LCS = new concentration, part number, and supplier
Section 14.4: New procedure for loading HL
Table II: Concentration change of low and Level 6 standards in 5mL purge
Table IV & IVA: Added new standard dilutions and levels
Table VI: Added 2-CEVE, Vinyl acetate, and Benzyl chloride

Version 2.0

Changes were made to the following:
Equipment (section 9.7)

Soil sample preservation (section 11.2)
The quality control section (section 12) was made to reflect the LIMS controls
Section 13.1
Section 13.2 was removed
Section 14.1.1 and section 14.1.5 were updated with the currently used standards
The GC Temperature Program (Section 14.6)
Section 14.7 was removed.
Sections 22.1, 22.3 and 22.4 were removed
Table 5 (spelling corrections)

Revision History Log for SOP # 653

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
10/19/16	2.1	See previous page	Kathy Cressia	Phyllis Shiller	10/19/16
2/23/17	2.2	Section 12.5- under MCP added or if curve is 0.99, section 14.1.1- added vinyl acetate, added 50ppm working standard table, section 14.1.5- added vinyl acetate and added 50ppm LCS table, Table III- added vinyl acetate, added CAS#	Harry Mullin	Phyllis Shiller	2/23/17
7/8/19	2.3	Added 8260D to method references	Kathy Cressia	Harry Mullin	7/8/19
12/12/19	2.4	Section 14.1.1 - Added general statement for list Added new compounds to table VI Added 2-Nitropropane to standards and LCS list	Harry Mullin	Kathy Cressia	12/12/19

Expiration Date

PHOENIX
Environmental Laboratories, Inc.
587 East Middle Turnpike, P.O. Box 370, Manchester, CT 06040
Telephone: 860.645.1102 • Fax: 860.645.0823

SOP#: 246

Title: Wipe Extraction by Soxhlet Method 3540C

1.0 Scope and Application

1.1 Method 3540 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wipes. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

1.3 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 Summary of Method

2.1 The entire wipe along with any residue solvent loaded into the thimble, and extracted using an appropriate solvent in a Soxhlet extractor.

2.2 The extract is then dried, concentrated (if necessary), and, as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

3.2 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided.

3.3 Soap residue, which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Specifically, Aldrin, Heptachlor, and most organophosphorus pesticides will degrade in this situation.

4.0 Sample Collection, Preservation, and Handling

4.1 Wipe samples must be collected in glass containers.

4.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction. All samples must be extracted within fourteen days of collection and completely analyzed within 40 days of extraction.

5.0 Apparatus and Materials

5.1 Soxhlet extractor with condenser
5.2 250-mL round bottom flask with covers/wraps
5.3 Boiling chips
5.4 Thimbles, disposable
5.5 Zymarks
5.6 Wooden tongue depressors, single use
5.7 Buchi Syncore concentration system

6.0 Reagents

6.1 Drying Agent - Sodium Sulfate (granular anhydrous), Na_2SO_4-Brand-Nu (337509) or equivalent.

6.2 Extraction Solvents- Methylene Chloride – BJ300 or equivalent, pesticide quality.

6.3 Nitrogen: Air Gas 0.999 Pure

6.4 Surrogates

6.4.1 SVOA= Ultra CUS-8804- Use 0.5mL.
6.4.2 PCB/Pest= Ultra ISM-320- Dilute 1mL Ultra ISM-320 into 100mL Acetone. Use 0.5mL of this solution.
6.4.3 NPD= Ultra PPS-100- Dilute 1mL of Ultra PPS-100 into 20mL of Acetone. Use 0.5mL of this solution.
6.4.4 EPH= Ultra ISM-581X- Use 2mL.
6.4.5 TPH= Aldrich 286931-5G, Pentacosane, 99%. Dilute 100mg pentacosane into 1 Liter methylene chloride. Use 1mL.

6.5 Matrix Spike Solution

6.5.1 SVOA= Supplied by SVOA Instrument Lab (Sec.6.6 SOP # 601.8270/624r12). Use 1mL.

6.5.2 Pest= Dilute 100ul of Restek #32415 + 200uL Absolute #70195 into 200mL of 1:1 hexane/acetone. Use 500uL of this solution.

6.5.3 PCB= Ultra CUS-14078. Use 100uL.

6.5.4 EPH= Dilute 4mL of Ultra SMA-300 + 4mL of Ultra SMA-310 into 100mL acetone. Use 2mL of this solution.

6.5.5 TPH+ETPH= Dilute 1.0mL Restek #566330 into 100mL 1:1 acetone/methylene chloride. Use 1.0mL of this solution.

6.5.6 NPD= Dilute 100uL of Absolute #96127 into 20mL acetone. Use 500uL of this solution.

7.0 Procedure

7.1 Label round bottom flask with sample number.

7.2 Add 80 mLs of appropriate solvent to a 250-mL round bottom flask containing 3 or 4 boiling chips by pouring it through the extractor to check for cracks.

7.3 Transfer wipe into thimble. With the wipe samples, sodium sulfate is added directly into the thimble after the wipe. Rinse the wipe jar with solvent and add to extraction solvent.

7.4 Add the surrogates listed in sec. 6.4 to each sample. Add the matrix spike/matrix spike duplicate compounds listed in sec. 6.5 to the two additional sample aliquots (if available) selected for spiking.

7.5 Attach the condenser to the extractor and water supply and wrap extractor with cover. Extract the sample for 12 -24 hours at 4-6 cycles/hour at the following temperatures: Hexane/acetone= 175°, DCM= 130°.

7.6 Allow the extract to cool after the extraction is complete.

7.7 The extract is now ready for concentration, cleanup, solvent exchange or analysis, depending on the extent of interferents, and the determinative method to be employed. Refer to Method 3600 for guidance on selecting appropriate cleanups methods. Excess water present in extract may be removed by filtering the extract through a bed of anhydrous sodium sulfate. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or sample analysis.

8.0 Quality Control

8.1 With every batch, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank (e.g., a newly opened wipe). Each time samples are extracted, and when there is a change in reagents, a method blank must be extracted and analyzed for the compounds of interest.

8.2 A LCS/LCSD are prepared and analyzed with every 20 samples or within 24 hours, which ever is more frequent.

8.3 Surrogate standards are added to all samples.

9.0 Definitions

9.1 Surrogate Analyte (SA) -- A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

9.2 Preparation Blank (PB) – A new wipe that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

9.3 Laboratory Fortified Sample Matrix and Matrix Duplicate (MS/MSD) -- An aliquot of an environmental sample to which known quantities of the method analytes are added. The MS/MSD samples are extracted and analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations.

10.0 Safety

10.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be minimized. The laboratory has a safety manual and MDSD volumes located in the general laboratory. It is important to read and understand these as well as the chemical hygiene plan.

10.2 Hexane is an extremely flammable liquid and harmful if swallowed, inhaled or absorbed through the skin. Methylene chloride is harmful if swallowed, inhaled or absorbed through the skin.

11.0 Pollution Prevention

11.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

11.2 Standards should be purchased in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

12.0 Waste Management

12.1 It is the laboratories responsibility to comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

13.0 Method Performance

13.1 This method was validated through internal QA/QC monitoring, including annual method detection limit studies, precision and accuracy studies, initial and continuing calibration verifications, blank analysis, laboratory control samples and matrix spikes and duplicates.

14.0 Corrective Action for Out-of-Control or Unacceptable Data

14.1 The Organics Department will order a "re-prep", should batch QC or surrogate recoveries be unacceptable.

15.0 References

15.1 EPA SW-846 Test Methods for Evaluating Solid Waste, 3rd Edition, Update III, Dec 1996, Method 3540C.

Appendix I – Prep Department Concentration Programs

Buchi Concentration Programs for Blow downs and Solvent Exchanges

Solvent	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6
ASE DCM	425 Mbar for 5 Mins	375 Mbar for 3 Mins	325 Mbar for 3 Mins	275 Mbar for 3 Mins	225 Mbar for 3 Mins	150 Mbar for 5 Mins
ASE Hex	200 Mbar for 3 Mins	150 Mbar for 2 Mins	100 Mbar for 4 Mins	50 Mbar for 3 Mins		
L/L DCM	450 Mbar for 2 Mins	400 Mbar for 5 Mins	350 Mbar for 6 Mins	300 Mbar for 9 Mins	250 Mbar for 8 Mins	100 Mbar for 3 Mins
SOX Hex	200 Mbar for 6 Mins	150 Mbar for 2 Mins	100 Mbar for 2 Mins	50 Mbar for 1 Mins		
SOXDCM	425 Mbar for 2 Mins	375 Mbar for 1 Mins	325 Mbar for 1 Mins	275 Mbar for 2 Mins	225 Mbar for 2 Mins	150 Mbar for 9 Mins

TurboVap Concentration Programs

Method	Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7	Step 8
#1 (all but 525)	2 Mins to 35 PSI	Hold for 45 mins						
#2 (for EPA 525)	8 PSI for 2 mins	10 PSI for 2 min	15.5 PSI- 5 min	21.5 PSI- 5 min	28 PSI- 5 min	32.2 PSI for 3 mins	35.7 PSI for 2 mins	Hold for 30 mins

NOTE: Any deviation from a program must to be documented in the associated Prep Batch for the affected sample(s).

Revision History Log for SOP #246 Wipe sox

Date:	Revision #:	Summary of Changes:	Submitted By:	Approved By:	Effective Date:
11/12/13	3	Updated new surrogate and spike solutions	Tara Banning	Kathy Cressia	11/12/13
1/25/17	3.1	Added sec 5.7- Buchi system, sec 5.2- 250mL flask, sec 6.5- updated SV, Pest, PCB spikes solutions and amounts used, sec 7.2- 80mL to 250 mL	Tara Banning	Phyllis Shiller	1/25/17

Expiration Date

Quality Systems Manual

Alpha Analytical, Inc.

D/B/A

Alpha Analytical
Eight Walkup Drive
Westborough, MA 01581-1019
Telephone: (508) 898-9220
Facsimile: (508) 898-9193

Alpha Analytical
Woods Hole Labs Facility
320 Forbes Blvd
Mansfield, MA 02048
Telephone: (508) 822-9300
Facsimile: (508) 822-3288

Email: info@alphalab.com
Web site: www.alphalab.com

Quality Assurance Officer: James Todaro, Ext 508-439-5101
Laboratory Technical Manager (Director) Organics-Westboro: Marco Soares 508-439-5144
Laboratory Technical Manager (Director) Inorganics-Westboro: Elena Dayn 508-439-5131
Laboratory Technical Manager (Director) Mansfield: John Trimble, 508-844-4134
Laboratory Technical Manager (Director) Air-Mansfield: Andy Rezendes, 508-844-4181

Approvers:
Jim Todaro Approved on 11/18/2022 2:13:40 AM, Elena Dayn Approved on 11/17/2022 2:40:13 PM, John Trimble Approved on 11/17/2022 2:58:48 PM, Andrew Rezendes Approved on 11/17/2022 4:46:26 PM, Marco Soares Approved on 11/17/2022 6:40:52 PM

1 Mission Statement

The mission of Alpha Analytical is quite simply to provide our customers with the greatest value in analytical service available. For the 'greatest value' is not only found in the data that is delivered, it is also found in the services provided.

- Data must be of the highest integrity, accuracy and precision.
- Consultation and educational services must be provided to support the customer in establishing data quality objectives and interpretation of the final data package.
- Support services such as sample containers, courier service and electronic data deliverables must be available to the customer.

Alpha's mission continues with an established commitment to our community and environment. We must ensure that we do not produce any additional contamination to our environment or harm our neighbors and community in any way.

The value of Alpha's product is in the honesty and integrity with which each chemist, courier, login staff member, or office staff member performs their tasks. The customer or employee must always feel satisfied that they received the greatest value in their lab experience at Alpha.

Alpha Analytical will vigorously pursue its mission into the next millennium.

Mark Woelfel

President

2 Table of Contents

3 Introduction

The Quality Systems Manual, referred to as Corporate Quality Systems Manual (CQSM) of Alpha Analytical describes the quality program in use at the laboratory for both Westboro and Mansfield facilities. This Quality Systems Manual provides employees, customers and accrediting agencies with the necessary information to become familiar with how the quality system operates within Alpha Analytical. The quality program includes quality assurance, quality control, and the laboratory systems including feedback mechanisms for the automated continuous improvement of the laboratory operations to meet customer needs.

Implementation of the laboratory operations is by documenting procedures, training personnel and reviewing operations for improvement. Written procedures are maintained as Standard Operating Procedures (SOPs). The SOPs are available to the staff as a controlled, electronic, secure copy. The provisions of the QSM are binding on all temporary and permanent personnel assigned responsibilities. All laboratory personnel must adhere strictly to the QSM and SOPs.

All policies and procedures have been structured in accordance with the NELAC Institute (TNI) Standards), DOD QSM 5.4 and applicable EPA requirements and standards.

Twenty-five (25) sections comprise the QSM. Related quality documentation including the listing of SOPs, forms, floor plan, equipment, personnel and laboratory qualifications are available. The QSM sections provide overview descriptions of objectives, policies, services and operations.

3.1 Scope

The QSM describes the requirements of the Laboratory to demonstrate competency in the operations for performing environmental tests for inorganic, organic, air and microbiological testing. The basis for the environmental tests is the methods found in documents published by the United States Environmental Protection Agency (EPA), ASTM, AOAC, APHA/AWWA/WEF, Standard Methods, and other procedures and techniques supplied by customers.

The QSM includes requirements and information for assessing competence and determining compliance by the laboratory to the quality system. When more stringent standards or requirements are included in a mandated test method, by regulation, or specified in a project plan the laboratory demonstrates achievement of the customer specified requirements through its documented processes.

The QSM is for use by Alpha Analytical for developing and implementing the quality system. Accrediting authorities and customers use the QSM for assessing the competence of Alpha Analytical. Alpha Analytical is committed to continually improving the quality system. Meeting customer needs, operating within regulatory requirements and adhering to Alpha's Data Integrity and Ethics policy are several of the mechanism used to continually improve the quality system.

3.2 Policy Statement

This Quality Systems Manual summarizes the policies, responsibilities and operational procedures associated with Alpha Analytical. This manual applies to all associates of the laboratory and is intended for use in the on-going operations at Alpha Analytical. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, corrective action, and reporting are described. All policies and procedures have been structured in accordance with the NELAC Institute (TNI) Standards, DOD QSM(which includes 17025 standards), applicable EPA requirements, regulations, guidance, and technical standards. This Quality Systems Manual, laboratory Standard Operating Procedures (SOPs), and related documentation describe the quality systems, policies and procedures for Alpha Analytical.

Alpha Analytical performs chemical analyses for inorganic and organic constituents in water, seawater, soil, sediment, oil, tissue and air matrices. Alpha Analytical's goal is to produce data that is scientifically valid, technically defensible, and of known and documented quality in accordance with standards developed by The NELAC Institute (TNI) Standards and any applicable state or EPA regulations or requirements. It is the commitment of the President, Operations Director, Laboratory Technical Manager and Quality Assurance Officer to work towards continuous improvement of the operation, and towards meeting our customer's needs, requirements, and intended data usage. This continued commitment is built into every activity of the laboratory. It is the responsibility of Senior Management and the Department Managers to ensure that all associates familiarize themselves with, and comply at all times with, the quality systems, procedures and policies set forth in this manual, laboratory SOPs, and related documentation.

Alpha Analytical analyzes Proficiency Test (PT) samples, in accordance with the NELAC Institute (TNI) Standards and other regulatory programs, from a National Institute of Standards and Technology (NIST)-approved PT provider for the analytes established by EPA for water samples, and for other analytes and matrices. The specific analytes and matrices analyzed are based on the current scope of the laboratory services as documented in the laboratory SOPs and state certifications.

The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that analyses can be performed correctly and within the expected schedule. All measurements are made using published reference methods or methods developed by Alpha Analytical. Competence with all methods is demonstrated according to the procedure described in SOP/ 1739 prior to use.

Alpha Analytical has developed a proactive program for prevention and detection of improper, unethical or illegal actions. Components of this program include: internal proficiency testing, electronic data audits and post-analysis data review by the QA Officer; a program to improve employee vigilance and co-monitoring; and Ethics Training program identifying appropriate and inappropriate laboratory practices, instrument manipulation practices and consequences. Additionally, all associates are required to sign the Alpha Analytical *Ethics Agreement* form upon commencement of employment and complete annual refresher Ethics Training thereafter. This form clearly outlines the possible consequences of unethical or improper behavior, or data misrepresentation. All staff are required to report any suspected unethical conduct to management. Management will then investigate and determine if the situation was considered unethical and will take appropriate action as described in the Alpha Ethics policy.

It is the policy of the laboratory to discourage and reject all influence or inducements (whether commercial, financial or personal) offered either by customers or suppliers, which might adversely affect results or otherwise compromise the judgment or impartiality of the staff. It is the responsibility of the Operations Director and Laboratory Technical Manager to inform customers and suppliers of this policy when necessary.

In the event that any such influences or inducements are encountered, the staff is instructed to inform management immediately. It is the responsibility of the Operations Director and the Laboratory Technical Manager to take appropriate action to prevent recurrence.

3.3 References

External reference documents are available electronically in the Qualtrax system for staff to access the latest edition or version of the reference methods, regulations or national standards. The Quality Assurance Department maintains the electronic files in the Qualtrax system. Management purchases automated update services, where available, to provide the laboratory with the latest hardcopy edition, where electronic means is not available.

3.4 Definitions

Appendix A lists the definitions as adopted by the laboratory. The definitions are mostly from the 2016 TNI standards and other sources.

4 Organization and Management

4.1 Legal Definition of Laboratory

Alpha Analytical is a full service analytical laboratory. Testing services include Drinking Water, Waste Water, Ground Water, Waste material and Air. Alpha Analytical is a privately held corporation incorporated in the state of Massachusetts. Alpha Analytical, Inc. does business as (D/B/A) Alpha Analytical.

Alpha Analytical has been in business since 1985. The types of businesses served include:

Consulting firms,
Engineering firms,
Waste Management Companies,
Industrial sites,
Municipal agencies
Department of Defense projects.

4.2 Organization

The laboratory operates a quality system approach to management in order to produce data of known quality. The laboratory organization provides effective communication and lines of authority to produce analytical data meeting customer specifications. The organizational design provides open communication while ensuring that pressures and day to day operating circumstances do not compromise the integrity of the reporting of the final data. See Appendix B for Organizational Chart.

The President is responsible for directing all areas of the company. The following job functions report to the President:

Operations Manager
Quality Assurance Officer
Marketing / Business Development / Sales
Financial Services
Human Resources

The Operations Manager is responsible for directing all laboratory operational areas of the company. The following job functions report to the Operations Manager:

Laboratory Technical Manager(s)

Customer Services Manager

Department Managers

The Laboratory Technical Manager(s) is(are) responsible for the laboratory data generated by the organics testing, inorganics testing and metals testing areas and the Air Technical Director is responsible for laboratory data generated by air analyses.

The Departmental Managers (Supervisors) have the following responsibilities:

The organics managers direct personnel in the organics extraction and instrumental laboratories.

The wet chemistry manager directs personnel and team leaders in the wet chemistry and/or microbiological testing areas.

The metals manager directs personnel and team leaders in the metals sample preparation and instrumental laboratories.

The Quality Assurance Officer is a member of the staff and reports directly to the President and has defined responsibility and authority for ensuring that the quality system is implemented and adhered to at all times. The Quality Assurance (QA) Officer is responsible for interacting and communicating certification requirements, implementing the Quality Systems Manual and reporting to the Laboratory Technical Manager and Senior Management the status of the quality program. The QAO oversees the Quality Systems Specialists and is responsible for oversight and/or review of quality control data and function independently from laboratory operations.

The Customer Services Manager is responsible for customer interactions, project coordination and laboratory personnel notification of project requirements.

The Marketing, Business Development and Sales personnel are responsible for increasing the volume of work from current customers and adding new customers to the base business of Alpha Analytical. The Marketing and Business Development personnel review all new work with the Laboratory Technical Manager, Operations Manager, President and/or Quality Assurance Officer before contractual commitment.

The CFO is responsible for maintaining and reporting on the financial status of the company. The CFO directs financial personnel on proper accounting procedures and maintaining the list of approved suppliers and subcontractors. The CFO reports directly to the President.

The Human Resource Director is responsible for personnel recruitment, hiring, performance reviews.

Personnel job descriptions define the operational function duties and responsibilities. Administration and Laboratory personnel assignments may include cross-functional training and work performance in multiple areas of the operations. Multiple function training ensures laboratory back up personnel during peak workloads.

During the absence of any staff member, assignment of alternative personnel occurs by memo or e-mail. The Manager or Supervisor authorizes the assignment. The naming of alternative personnel assures the continuing performance of critical tasks during the primary person's absence and ensures that lines of communication remain open for continued decision making. The deputy for the Laboratory Technical Manager is the Quality Assurance (QA) Officer. The deputies for the Quality Assurance (QA) Officer are the Quality Systems Specialists.

For the purposes of the NELAC Institute (TNI) Standards the Lead Laboratory Technical Manager is the Laboratory Technical Manager. The deputies for the Lead Technical Manager are the Quality Assurance (QA) Officer, and the Departmental Managers. The Laboratory Technical Manager meets the requirements specified in the Section 4.1.7.2 Volume 1, Module 2 of the 2016 TNI standards. If the Laboratory Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, a full-time staff member meeting the qualifications of Laboratory Technical Manager will be designated to temporarily perform this function. The primary Accrediting Body shall be notified in writing if the Technical Manager's absence exceeds 35 consecutive calendar days.

4.3 Business Practices

Alpha maintains certification for the programs and analytes required by regulatory programs. The listing of qualifications from the various certifications, registrations and accreditation programs are available upon request. Alpha Analytical operates Monday to Friday from 7:30 a.m. to 5:30 p.m. Management prepares and posts the holiday schedule for the year indicating closed operations. Sample delivery occurs during normal operating hours unless arranged in advance.

Alpha's reputation depends upon timely reporting and quality data. The standard turnaround time for engineering and consulting firms is five business days from time of sample receipt. Standard turnaround for all other customers is ten business days from time of sample receipt. The time of sample receipt is when the verification of the chain of custody and samples meets the laboratory sample acceptance policy. Laboratory management must approve any special arrangements for rush or expedited turnaround time. The basis for data quality depends on customer, regulation and method performance criteria. Accuracy, precision, sensitivity and comparability are expressions of method performance criteria.

All work is performed in the strictest confidence. New and contract employees must review corporate policy and practice requirements for protecting customer confidentiality and proprietary rights. The review occurs during orientation and ethics training. It is the policy of the laboratory to release data to the customer authorized contact. Personnel assigned the duties of interacting with customers review project files and discuss data related only to the project. Personnel whose duties do not include routine customer contact must check with the customer service manager before discussing data with regulators or third parties

5 Quality System

Establishment, Audits, Essential Quality Controls and Data Verification

5.1 Establishment

The Mission Statement presents the policy and objectives for Alpha Analytical. The Quality Systems Manual provides the framework for the processes and operations to implement the Mission. The Quality Systems Manual and documentation controlled by the laboratory system detail the management authorized operations for achieving the objectives of the company.

The laboratory operates a quality system approach to management in order to produce data of known quality. Alpha Analytical is a full service laboratory designed to provide its customers with accurate, precise and reliable data within the best turn-around time and at the most reasonable prices. Alpha employs chemists of the highest training, ethics and caliber in the field of analytical chemistry. This and state-of-the-art instrumentation and automation combine to insure data of known and documented quality.

5.2 Quality Systems Manual

The QA Officer is responsible for the publication and distribution of the Quality Systems Manual and annual review. Management reviews and authorizes the manual. Implementation of major changes in the quality system occurs after revision of the appropriate Quality Systems Manual section and authorization by management.

The authorization of the Quality Systems Manual is documented electronically in Qualtrax. Updates of this manual occur at any time throughout the year. Document control procedures (SOP1729) apply to the distribution of the Quality Systems Manual. Controlled copies of the manual are maintained electronically within Qualtrax. Persons or organizations outside of Alpha Analytical may receive uncontrolled copies. Copies are distinctly indicated “Uncontrolled Documents” within the footer of each page.

5.3 Audits

Laboratory audits, both internal and external, review and examine the operations performed in the laboratory. Internal audits are conducted by qualified QA Specialists and external audits are reviews by external organizations to evaluate the ability of the laboratory to meet regulatory or project requirements. Internal audits are conducted on a frequency of annually, or method required.

A QA designee schedules internal process audits to ensure the completion of the annual audit of each operational area. The process audits are a more detailed review of the operations. Personnel from areas other than the one audited perform process audits.

The internal system audit is a review of the implementation of the documented quality system. The system audit includes sample tracking from receipt to disposal, a data audit of a completed report, and all operations not audited during the process audit.

The purpose of the internal system audit is:

Verification that adequate written instructions are available for use;

Analytical practices performed in the laboratory are consistent with SOPs;

The quality control practices are applied during production;

Corrective actions are applied as necessary;

Deviations from approved protocols are occurring only with proper authorization and documentation;

Reported data is correct and acceptable for reporting;

SOPs, quality records, analytical records, electronic data files are maintained properly; and

Personnel training files and records are satisfactory and current.

Before a scheduled internal audit, the assigned auditor reviews checklists, if used, and/or the SOP specific to the area. The checklist may be from an external source or prepared by the auditor. . After the audit, the auditor submits a summary or notes from the audit to the Laboratory Technical Manager or QAO as part of the audit report. The summary identifies discrepancies found during the audit. Technical personnel are responsible for the inspection and monitoring of in-process and final data. Personnel independent of those having direct responsibility for the work performed audit the quality system and processes.

Representatives sent by customers and government or accrediting agencies often perform external audits. These audits are most often announced inspections, but sometimes are not announced. The Quality Assurance Officer, Laboratory Technical Manager or assigned deputy, and/or appropriate Department Manager accompany the external audit team through the laboratory. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory staff and technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors usually provide a report on their findings shortly after the audit. The QA Officer receives the audit report and copies are provided to laboratory personnel for review. Corrective actions are identified and distributed to responsible parties for implementation in response to any cited deficiencies.

5.4 Audit Review

Management reviews internal and external audit reports to evaluate system effectiveness at the annual management review meeting. Tracking of the audit findings occurs through the nonconformance action process. The management and staff work together to establish a time line for resolving the audit findings. The Quality Assurance team tracks the time line and reports to the Laboratory Technical Manager on any outstanding audit findings. Approved corrective actions for DoD that are not implemented or avoided may result in loss of DoD ELAP accreditation and may result in work being discontinued until implementation is verified by DOD ELAP AB.

5.5 Performance Audits

Alpha Analytical participates in inter-laboratory comparisons and proficiency test programs required by customers and certifying agencies. The performance audits provide information on the data comparability of results generated by the laboratory. Test samples received by the laboratory are handled following routine laboratory procedures. Proficiency test samples are unpacked, checked against the packing slip and examined for damage. Reporting requirements and deviations to routine practices are noted as would be required for any project.

Analysts demonstrate proficiency by analyzing either an external proficiency test sample, an internally prepared blind test sample or Initial Demonstration of Capability (IDC) before independent operation of a test method. The results of performance audits serve several purposes. The QA Officer may use performance audits for evaluating analyst proficiency, laboratory performance in a specified area to facilitate laboratory improvement efforts, and/or to provide information to an accrediting agency on correction of past performance of an external performance audit.

5.6 Corrective Actions/Preventative Actions (CAPA)

The corrective action process at Alpha Analytical is detailed in SOP 1736. The corrective action program at Alpha Analytical uses the Quality Nonconformance workflow in Qualtrax to document and follow through the corrective action/preventative action process for three main areas: nonconformance's within the laboratory, customer complaints and failed PT studies. The process ensures continuous improvement of company performance by preventing the recurrence of quality problems.

Nonconformance reports are tracked for closure date and the type. Reports to management include the listing of open nonconformance reports and the frequency of the type of nonconformance occurring. A QA designee monitors the completeness of the forms, as well as verifies the actions are complete and acceptable.

Customers will be notified within 5 days of any question(s) regarding validity of results.

5.7 Managerial Review

The management review occurs at least once per year as part of the strategic planning process. Documentation of the management review meeting is by recording the meeting minutes and listing the attendees. The focus of the quality management review is the frequency of the type of nonconformance, closure status, audit progress and other quality assurance actions. Meetings include discussion and progress on quality system initiatives since the last meeting.

Prior to the meeting, an agenda is distributed to all personnel expected to be in attendance. The meeting is chaired by the President. Minutes are taken and distributed at the conclusion of the meeting by a QA designee. If action is necessary on any issue, a Summary Report is generated and distributed to responsible parties for implementation. Actions are monitored by the QAO or designee until completion.

5.8 Essential Quality Control Procedures

The following general quality control principles apply to all tests. The manner implemented is dependent on the type of test performed. The laboratory SOP presents the specific quality control checks undertaken to ensure precision, accuracy and sensitivity of each test method. Deviations from the existing SOP are allowed only upon approval of the deviation by the department manager and Quality Assurance Officer. This documentation must be either in form of written notice or email.

Alpha Analytical uses quality control samples to evaluate the following:

1. Adequate positive and negative controls to monitor blanks, spikes, reference toxicants, zero blanks;
2. Adequate tests to define the variability and/or reproducibility of laboratory results;
3. Measures to ensure the accuracy of the test data including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples;
4. Measures to evaluate test performance, such as detection limits and quantitation limits or range of applicability such as linearity;
5. Selection of appropriate formulae to reduce raw data to final results such as linear regression, internal standards, or statistical packages;
6. Selection and use of reagents and standards of appropriate quality;

7. Measures to assure the selectivity of the test for its intended purpose;
8. Measures to assure constant and consistent test conditions for the method such as temperature, humidity, light, or specific instrument conditions.

   Note: All quality control samples are treated in the same manner as field samples.

All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance limits are used to determine the usability of the data. Control charts and/or calculated control limits monitor the long-term method performance by analyte, by instrument for water matrices. Routine evaluation and reporting of the control chart performance provides supervisors and management with additional performance measures to ensure data comparability. Control limits are recalculated when trends are observed.

Where no reference method or regulatory criteria exist, the laboratory specifies the acceptance/rejection criteria in the SOP. The test SOP specifies the QC samples performed per batch of samples. The quality control samples are categorized into the following, as appropriate to the method

- Method Blank
- Laboratory Duplicate
- Laboratory Control Sample (LCS)
- Laboratory Control Sample Duplicate (LCSD)
- Matrix Spike (MS)
- Matrix Spike Duplicate (MSD)

Selection of samples for Duplicate, Matrix Spike (MS) & Matrix Spike Duplicate (MSD)

2. Duplicate samples
   a. Samples will be selected if identified and requested by customer
   b. If no samples are identified by the customer then random samples will be analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.

3. Matrix Spike (MS) / Matrix Spike Duplicate (MSD) samples
   a. Samples will be selected if identified and requested by customer
   b. If no samples are identified by the customer then random samples will be selected and analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.
   c. If MS/MSD is not required, LCS/LCSD may be substituted for

precision and accuracy evaluation.
All DOD projects require MS/MSD.

The frequency is dependent on the reference method and test protocol. The following is the default requirement for quality control checks in lieu of any other guidance. The frequency for each quality control sample is generally one (1) per every 20 samples.

5.9 Data Reduction

After completion of the test procedure, the data reduction process begins.

Chromatography data may require the manual integration of peak areas or heights before reporting of results. The analyst must perform manual integration when software does not properly integrate or identify the peak. Manual integration must not occur for the purpose of achieving acceptable quality control or calibration. Signatures of analyst performing manual integrations can be found by electronic entry of analysts initials that can be traced to original signatures in the "Employee Signature Register". The analyst notes the rationale for performing the manual integration using the M-Codes listed in the manual integration SOP 1731 and ensures the "TIC" marks from the software represent the integration area used for reporting the results. The analyst must minimize and avoid manual integration. The establishment of the proper integration parameters in the software reduces the number of manual integration occurrences.

The SOP for each test presents the formulas used for the specific test method. The formulas for the data calculations used throughout the laboratory are the following:

% Recovery (LCS)

$$\frac{MV}{TV} * 100 = \%R_{LCS}$$

where:
MV = Measured Value
TV = True Value

% Recovery (MS or MSD)

$$\frac{MV - SV}{TV} * 100 = \%R_{MS}$$

where:
MV = Measured Value
TV = True Value
SV = Amount found in sample

Average ($\overline{X}$)

$$\sum_{i-1}^{n} X_i \Big/ n = \overline{X}$$

where:
$\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Relative Percent Difference (% RPD)

$$\frac{R_1 - R_2}{\left(R_1 + R_2\right)/2} * 100 = \%RPD$$

where: R_1 = Larger of two observed values
R_2 = Smaller of two observed values

% Difference (%D)

$$\frac{X - \overline{X}}{\overline{X}} * 100 = \%D$$

where: $\overline{X}$ = Average of all values
X = Result of measurement

Standard Deviation of the sample (S_x)

$$\sqrt{\frac{\sum\left(X - \overline{X}\right)^2}{n-1}} = S_x$$

where: $\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Relative Standard Deviation (%RSD)

$$\frac{S_x}{\overline{X}} * 100 = \%RSD$$

where: $\overline{X}$ = Average of all values
Sx = Standard Deviation (n - 1)

Range of Logs (for microbiological enumeration analysis)

10% of routine samples are analyzed in duplicate and the range of logs is determined.

MDL (See 40CFR Part 136 for details)

where: *MDL* = The method detection limit

$$\left[\sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2 \Big/ n}{n-1}}\right] * t_{0.99} = MDL$$

X = Result of each measurement
n = Number of values
t(n-1,1 = .99) = The students' T value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. (See Students t Test Table)

Reporting Limit (RL)

Lowest calibration standard or greater

Control Limits

Upper Control Limit: $\overline{X} + 3 * S_x = UCL$
Lower Control Limit: $\overline{X} - 3 * S_x = LCL$

Warning Limits

Upper Warning Limit: $\overline{X} + 2 * S_x = UWL$
Lower Warning Limit: $\overline{X} - 2 * S_x = UWL$

Method of Standard Additions (MSA): (See EPA 7000A for details)

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume Vx, are taken. To the first (labeled A) is added a known volume Vs of a standard analyte solution of concentration Cs. To the second aliquot (labeled B) is added the same volume Vs of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration Cx is calculated:

$$C_x = \frac{SB\ V_S\ C_s}{(SA - SB)\ V_X}$$

where SA and SB are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_s should be chosen so that SA is roughly twice SB on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume.

For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. A linear regression program may be used to obtain the intercept concentration.

5.10 Document Control

The Document Control Procedure (SOP/1729) describes the process for controlled and uncontrolled documents. The use of the revision number allows for the retention of a previous document for historical information purposes.

Every document is assigned a unique identification number, which is present on each page of the document. A master list of documents includes the unique identification. Each controlled copy includes the revision number, published date and page number.

Full document control includes the status of each document: active, inactive or superseded/archived. Inactive documents are procedures not currently requested, but may be in the future. Archived documents are procedures replaced with a later revision. Authorized personnel must review and approve each document and any subsequent revisions before use in the laboratory. Personnel authorized to review and approve a document have access to all necessary information on which to base their review and approval. The history section of the document in Qualtrax includes a description of the nature of the document change.

Standard Operating Procedures (SOPs) are instructions for repetitive or standard operations performed by the laboratory. The SOP author is the person familiar with the topic. The standard format for writing SOPs is set-up as a template for administration and technical SOPs. Each SOP is peer reviewed, authorized by management, and QA before final publication and implementation. Authorized signatories for controlled documentation include one or more of the following personnel: Company President, Quality Assurance Officer, Laboratory Technical Manager, Department Manager, Department Team Leader. Personnel acknowledges approved documents as read, understood and agreed to through electronic attestation forms associated with each document as SOP Attestation Tests which reside in Qualtrax.

SOPs must receive evaluation and input by laboratory supervisors and key technical personnel. The content of each SOP must conform to applicable requirements of analytical methods and certification agencies. Within these constraints, the content of a SOP meets the needs of a particular area of the laboratory. A new or revised SOP is needed when regulatory programs update or add methods, the scope of the existing method is extended, or when activities are being performed without adequate documentation.

Updating, modifying and changing SOPs, forms and the contents of this QSM are prompt and part of the routine practices. The prompt modification of these documents ensures the documents reflect the current practices and operations of the laboratory. During annual review of a document, (including but not limited to: SOPs, Ethics Policy, Quality Systems Manual), requested changes are reviewed and the document reissued using the information and a new revision number is assigned and published in Qualtrax.

The laboratory maintains control over the possession and distribution of all documents that directly affect the quality of data. This includes, but is not limited to, documents such as the Quality Systems Manual, Standard Operating Procedures, customer instructions, Laboratory Work Instructions, data sheets, check lists and forms.

5.11 Detection Limits

Detection Limits (DLs), previously referred to as Method Detection Limits (MDLs), are determined for all analytes as specified in the Institute (TNI) Standards. DLs are determined for all new instrumentation, whenever there is a change in the test method or instrumentation that affects performance or sensitivity of the analysis. From these, detection limits, Reporting Limits (RLs), are established. The RL is the minimum concentration of an analyte that can be identified and quantified within specified limits of precision and bias during routine and analytical operating conditions.

Method Blanks are evaluated to determine an MDLb when performing an initial MDL study and annually thereafter.

Laboratory reporting limits lie within the calibration range, at or above the RL. For methods that require only one standard, the reporting limit is no lower than the low-level check standard, which is designed to verify the integrity of the curve at lower levels. If reporting limits are required below the lower level of the calibration curve, RL, or low-level check standard, method modifications are required. Refer to DL/LOQ SOP/1732. Note: ”J” Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Detection Limit (DL) with a “J” qualifier.

5.12 LOD/LOQ Studies

A. LOD (Limit of Detection) Verification - DOD only

1. LOD is required quarterly for all DOD projects. If there are no DOD projects for a particular quarter than LOD is not required for that quarter.

2. All sample-processing steps of the analytical method shall be included in the determination of the LOD.

3. The validity of the LOD shall be confirmed by ***qualitative*** identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests, and > 1X up to 4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data.

4. An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature. Where an LOD study is not performed, the laboratory may not report a value below the limit of quantitation.

B. LOQ (Limit of Quantitation) Verification

1. LOQ (Limit of Quantitation) verification is required quarterly for each target analyte. The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix. A successful analysis is one where the recovery of each analyte is within the established test

method acceptance criteria for accuracy

The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH)..

Refer to DL/LOQ SOP/1732

5.13 Range of Logs – Precision of Quantitative Methods - Microbiology

A. Precision of duplicate analyses is calculated for samples examined by enumerative microbiological methods according to the following procedure:

   a. Perform duplicate analyses on first 15 positive samples.

   b. Record duplicate analyses as D1 and D2 and calculate the logarithm of each result.

   c. If either of a set of duplicate results is <1, add 1 to both values before calculating the logarithms.

   d. Calculate the range (R) for each pair of transformed duplicates as the mean of these ranges.

6 Personnel

6.1 Laboratory Management Responsibilities

Management is responsible for communicating the requirements of the quality system, customer specifications and regulatory needs to all personnel. Management job descriptions detail the responsibilities of each position.

The H.R. Director has job descriptions for all positions in the laboratory defining the level of qualifications, training, and experience and laboratory skills. During initial training, management provides access to documented operations procedures, observes personnel performance, and evaluates personnel proficiency. Management documents technical laboratory staff's proficiency initially and on a continuing basis through use of laboratory control samples and purchased proficiency evaluation standards.

Management is responsible for verification of proper sample management and all aspects of data reporting. The communication of the operating practices of the laboratory is through the document control and attestation process.

Either the Quality Assurance Officer, Operations Director and/or Technical Managers have the authority to stop work due to non-conformances and have the authority to resume work after it has been stopped.

6.2 Laboratory Staff Requirements

Recruitment is the responsibility of the Operations Manager and HR Department, with input from other personnel as required. The Training Program procedure SOP/1565 details the process for completing requirements and training to ensure personnel have adequate skills and competence for the job function. Initial training includes ethics training, Qualtrax Training, QA Basics, IT/LIMs including computer security.

A job description details the necessary requirements for each job and includes position title, minimum educational requirements, skills, responsibilities and reporting relationships and any supervisory responsibility.

Initial training of new employees and contract staff includes laboratory ethics and quality policies, signing the Employee Signature Log, as well as execution of an Ethics Agreement. Any employee found to knowingly violate the Ethics Policy Agreement, report data values, that are not actual values obtained or improperly manipulated, or intentionally report dates and times of data analyses that are not the actual dates and times of analysis, will lead to disciplinary action, including termination, as outlined in Section V.K of the Employee Handbook. Each employee must report personally or anonymously to the Laboratory Technical Manager, QA Officer and/or Ethics Team Member any accidental or suspected intentional reporting of non-authentic data by others for follow up action. The review of the laboratory ethics and ethics training occurs annually with all personnel.

(DOD) All inappropriate and prohibited laboratory practices, as detailed in the DOD QSM 5.4, will be reported to the appropriate accrediting body within 15 business days of discovery. Records of corrective actions or proposed will be submitted within 30 business days. Failure to notify the AB within 15 business days will result in suspension of the DOD ELAP accreditation.

The Ethics program consists of the following key components:

- Ethics Policy /Agreement (Appendix E)
- Initial and annual ethics training

- Internal audits conducted annually
- Adherence to Manual Integration SOP/1731
- Ethical or Data Integrity issues reported to Lab Managers, QAO or HR Director
- Anonymous reporting to HR Director - This is accomplished by writing a detailed description of the suspected ethics breach and submitting the information, anonymously, to the Human Resource Director.
- "No-fault" policy encouraging reporting of incidences without fear of retribution
- Electronic tracking and audit trails through LIMs and instruments enabled where available.

6.3 Training

The Quality Systems Manual and related documentation is available to all employees. Cross training, supervisory training and other related training takes place on a scheduled and as-needed basis. Training ensures the communication and understanding of all personnel in the laboratory-documented procedures and practices.

All personnel undertake orientation-training sessions upon initial employment. Orientation training includes laboratory business practices, employment specifications, Ethics Policy, Quality Systems Manual, Chemical Hygiene Plan, and all SOPs required for the job function.

Managers ensure the training for new employees and review the continuing training for current employees. Training includes on-site and off-site programs presented by staff members, contractors, equipment manufacturers, and institutions of higher learning.

Training of new personnel to any job assignment takes place on-site according to the Training Program procedure. Laboratory personnel may perform their assigned methods/protocols without supervision only after documentation of acceptable proficiency. Training records lists the current training status.

On-the-job training includes demonstration of skills during job performance, initial demonstration of proficiency, and review of SOPs. Health and Safety training takes place on an annual basis with careful introduction to new principles. Personnel have access to the Chemical Hygiene Plan and Safety Data Sheets. On-site training includes side-by-side hands-on training, formal classroom type instruction on the SOP or a meeting to discuss procedural changes or to address questions related to the laboratory operation. All training is documented via the Training Attestation Form, which is signed by all in attendance that they understood and will implement what was presented to them.

Training is an on-going opportunity to evaluate the laboratory operations. The updating of SOPs, Quality Systems Manual and other related information documents all changes to the quality system. Training is documented via the Training Attestation Form or in Qualtrax with training test records.

Off-site training takes place on an as-needed basis. Recommendations and suggestions regarding educational programs come from all levels of staff. It is the employee's responsibility to present a copy of any certificates or attendance information to the HR Director. The information is added to the individual's training record.

6.4 Records

The QA Department is responsible for maintaining training records. Credintals including certificates, transcripts, diplomas, resumes, and other records of training are placed in the individual's training file. Demonstrations of Capabilities are kept either in Qualtrax or LIMS.

Appropriate personnel are notified through email and/or Qualtrax or by the QA department when a revision is complete for the controlled version of a document. The manager of the area determines when a change is significant to require training.

Job descriptions are included in the training record files. The Human Resources Department reviews the job descriptions, Resumes and/or biosketches are kept on file with the Human Resources Department and the QA Department.

7 Physical Facilities – Accommodation and Environment

This laboratory facility has a total area of 25,000 square feet for each of the Westboro and Mansfield Facilities

The laboratory functional areas include:

Administration and offices
Sample receiving
Sample management
Air analysis (Mansfield Facility only)
Microbiological (Westboro Facility only)
General analytical chemistry
Metals sample preparation (Mansfield Facility only)
Organic sample preparation
Metals analysis (Mansfield Facility only)
Volatiles gas chromatography (GC)
Volatiles gas chromatography/mass spectrometry (GC/MS)
Volatiles air analysis (Mansfield Facility only)
Semivolatiles gas chromatography/mass spectrometry (GC/MS)
Semivolatiles gas chromatography (GC)
Emerging Contaminants (Mansfield Facility only)
Miscellaneous facility mechanical and storage areas.

All chemicals are stored in appropriate cabinets and properly disposed of as required. All flammable solvents are stored in OSHA and NFPA approved cabinets. Acids are stored in OSHA acid cabinets. Separate waste areas houses the sample and chemical waste before pickup by a licensed waste hauler.

7.1 Environment

Lighting, noise, humidity, heating, ventilation and air conditioning satisfy the needs of the testing performed on the premises. The laboratory building design ensures regulated temperature control for analytical equipment. Air-handling systems minimize airborne contaminants that may jeopardize sample integrity or analytical performance.

The analytical instrumentation is in separate rooms from laboratory activities that involve the use of large quantities of organic solvents or inorganic acids. A separate room, in the Westboro facility, provides the facilities for the microbiological testing.

Standards and other materials requiring below 0°C storage temperatures are placed in freezers and separated from samples or potential contaminating materials. Refrigerators provide cooling needs for samples and materials with temperature requirements of below room temperature and greater than freezing. Sample and standard storage areas are monitored and controlled for temperature and recorded in the data logger system. Sample storage areas for volatiles are separated from other samples and monitored for any effects due to cross contamination.

Bulk hazardous waste containers are located away from the testing activities. Waste disposal uses lab pack procedures and those designated by the regulatory authorities. The Chemical Hygiene Plan and the Waste Management and Disposal SOPs (Westboro: SOP/1728 and Mansfield SOP/1797)) include the procedures for handling and disposing of chemicals used in the laboratory.

The working and storage environments are maintained in a safe and appropriate manner. A Chemical Hygiene Plan details the requirements for safety and chemical handling. Safety measures that protect property and personnel from injury or illness include: fume hoods, fire extinguishers, fire blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes, and spill control kits.

7.2 Work Areas

Good housekeeping is the responsibility of all personnel. Each person is responsible for assuring clean and uncluttered work areas. The job descriptions list specific housekeeping duties. Records, samples and waste materials are the common cause for clutter in the laboratory.

. Removal of administration and laboratory records to the record storage area occurs to reduce clutter and ensure traceability. The individual filling the laboratory record box, labels the box with a number, the contents, date and laboratory area. Authorized personnel assign and record into a permanent record the box number, discard date and box contents. Authorized personnel review the box label for number, discard date and contents. Boxes are stored onsite and off-site for the record retention period identified in the NELAC Institute (TNI) Standards and EPA regulations, whichever is more stringent.

Sample management personnel remove samples to the sample storage area after all data is correct and complete. Sample coolers are removed to a designated storage area for recycling. Samples are stored in the designated process storage areas until testing is complete. Sample removal from the process storage occurs after mailing of the final report. The sample management staff places the samples in the archive storage area for thirty days after report release. The archive sample storage area is not controlled or monitored. Based on customer specifications, samples are properly disposed or returned to the customer.

Waste materials, expired reagents, expired standards and materials are disposed of and not stored in the laboratory. Hazardous waste labeled accumulation containers in the laboratory collect designated waste streams for later bulk disposal. Laboratory personnel remove the less than five-gallon accumulation containers when full from the laboratory and place the containers in the bulk hazardous waste area. Refer to the Waste Management and Disposal SOPS for Westboro: SOP/1728 and Mansfield SOP/1797. Personnel identifying out of date reagents and standards remove the materials to the proper disposal area.

7.3 Security

Alpha Analytical provides a secure environment for our employees, guests, customers, samples and analytical data. Security procedures require that all exterior doors remain locked unless manned. Access to the laboratory is limited to employees and contractors. Visitors not under signed contract are required to sign the Visitors Log and must be accompanied by a laboratory employee at all times within the testing areas.

The defined high security area is the sample management area. Identification card locks on the internal doors control entry into the laboratory area.

All doors are locked after hours and require a key for entry. The security alarm continuously monitors for smoke and fire related heat. When the alarm is activated, the appropriate emergency response officers are notified. The local emergency offices have the emergency contact list for the laboratory.

8 Equipment and Reference Materials

8.1 Maintenance

The laboratory has a proactive equipment maintenance program. The laboratory maintains service contracts for most major equipment, which include routine preventative maintenance visits by the service provider. Technical personnel perform manufacturer's specified maintenance on a routine basis to ensure equipment operates at peak performance.

A brief summary of some common preventive maintenance procedures is provided in Appendix D. All instrument preventative and corrective maintenance is recorded in the maintenance logbook assigned to the equipment. After maintenance or repair, the instrument must successfully calibrate following the method SOP. Laboratory personnel must demonstrate quality control performance before sample analysis.

The laboratory maintains a stock of spare parts and consumables for analytical equipment. Backup instrumentation for some analytical equipment is available on site for use in case of major equipment failure. The person discovering or suspecting an equipment maintenance problem or failure tags the equipment with 'out of service' tag. If routine maintenance measures do not eliminate the problem, the Laboratory Technical Manager or Operations Director is notified and the appropriate equipment service provider is contacted.

All major laboratory equipment has individual and traceable maintenance logbooks in which to document manufacturer's recommended maintenance procedures, specific cleaning procedures, comments on calibration, replacement of small worn or damaged parts, and any work by outside contractors. The person performing routine or non-routine maintenance signs and dates the maintenance logbook. If an instrument is down for maintenance, a complete record of all steps taken to put it back into service is recorded including reference to the new calibration and quality control checks. Any equipment service providers working on the equipment are recorded in the logbook.

Record repetitive or on-going equipment problems other than normal maintenance requirements on nonconformance action forms. The nonconformance action form notifies management and the Quality Assurance Officer of a problem affecting the performance and data quality.

The laboratory groups some equipment into a single laboratory equipment maintenance logbook. Examples include: autopipets, thermometer calibration. The identity of each item is by serial number or a laboratory-designated item number. The same data recorded for major equipment applies to this documentation.

The maintenance records shall include:

Equipment name;

Manufacturer's name, type identification, serial number or other unique identification;

Date received, date put into service, condition when received;

Current location;

Details of past maintenance and future schedule;

A history of any damage, malfunction, modification or repair;

Dates and results of calibration or verification.

The maintenance logbook may include the reference to the location of the equipment operational and maintenance manuals. The logbook may include the reference to laboratory run logbook or data files for the calibration and quality checks of daily or frequent calibrations.

The Courier Supervisor ensures that maintenance and records for transportation vehicles are complete. The purchasing process is used for ordering garage maintenance, the garage work order is reviewed, and the vehicle checked for condition. The Controller receives all paperwork for completion of the maintenance process.

8.1.1 Microbiology General Equipment Maintenance

Optics of the Quebec colony counter and microscope are cleaned prior to each use. The stage of the microscope is also cleaned and the microscope is kept covered when not in use.

Glassware is checked for residual alkaline or acid residue utilizing bromothymol blue (BTB) on each day of media preparation.

8.2 Equipment Listing

A listing of the major equipment used for testing is available upon request. The equipment list details the unique identification number, equipment location, serial number, model number, and purchase date. The unique identification number is attached to the piece of equipment.

The laboratory performs analyses using state of the art equipment. In addition to the major equipment, the most common equipment used in the laboratory are: thermometers, balances, autopipets, water baths, hot plates, autoclaves, pH meters, conductivity meters and a variety of labware. The SOPs list the calibration and verification requirements for all laboratory equipment used in measurements.

8.3 Laboratory Water

Laboratory water is purified from central DI and RO water systems and piped to all laboratory areas. The QA Department samples the laboratory grade water and submits the samples for analysis by the lab to document the water meets the drinking water certification criteria. The Laboratory Water Logbook lists the daily conductivity checks and acceptance criteria for the laboratory water. The laboratory documents the daily, monthly and annual water quality checks. Please refer to Table 8-1 for tested parameters, monitoring frequency and control limits for each parameter (SOP/1738). Additional parameters may be tested for at the laboratory's discretion.

When additional treatment occurs in the test area, that test area records the water quality checks from the most frequently used tap. At a minimum the quality of the laboratory grade water is monitored daily by conductivity measurements. Records of the daily checks are found in the Laboratory Water Logbook. If out of specification results occur, a nonconformance action form is submitted.

TABLE 8-1

Parameter	Monitoring Frequency	Control Limits
Conductivity	Daily	<2 µmhos/cm @ 25°C
pH	Daily	5.5 - 7.5
Total Organic Carbon	Monthly	< 1.0 mg/L
Total Residual Chlorine	Monthly	< detection limit
Ammonia	Monthly	< 0.1 mg/L
Metals: Cd, Cr, Cu, Pb, Ni and Zn	Monthly (Required Annually)	< 0.05 mg/L
Total Metals	Monthly (Required Annually)	< 0.1 mg/L

Heterotrophic Plate Count (Westboro only)	Monthly	< 500 CFU/mL
Water Quality Test (Biosuitability) (Westboro only)	Annually	0.8 – 3.0 ratio

8.4 Reference Materials

Reference materials include: Class 1 weights, NIST thermometers and reference standards. Timers used for DOD projects are NIST-certified. Logbooks record the reference materials used for calibration and verification. The Department Manager or QA Department maintains any certificates received with the reference materials. Laboratory personnel record in the standards logbook the reference standards date received, unique identification number, expiration date and number of containers. Each laboratory area records the unique identifier on the reference standard certificate and the Department Manager maintains the certificate. The identifier allows traceability from the certificate to the analytical data.

9 Measurement Traceability and Calibration

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before put into service and on a continuing basis. The results are recorded in the instrument specific logbook. The laboratory has a program for the calibration and verification of its measuring and test equipment. The program includes all major equipment and minor equipment such as balances, thermometers and control standards. The Quality Systems Manual and method SOP describe the calibration records, frequency and personnel responsibilities.

9.2 Traceability of Calibration

The program of calibration and/or verification and validation of equipment is such that measurements are traceable to national standards, where available. Calibration certificates indicate the traceability to national standards, provide the results, and associated uncertainty of measurement and/or a statement of compliance with identified metrological specifications. A body that provides traceability to a national standard calibrates reference standards. The laboratory maintains a permanent file of all such certifications.

9.3 Reference Standards and Materials

Alpha Analytical has a program for calibration and verification of reference standards. The results and program are recorded in the appropriate instrument logbook. Required in-service checks between calibrations and verifications are described in method SOPs and are recorded in the appropriate instrument logbook.

Calibration standards are maintained within the area of consumption. A logbook of use is maintained and use is limited strictly to method required calibrations. Each calibration standard is identified as to test method used, date received, date opened, and expiration date. Calibrations are verified by using a second source or lot number of the calibration standard. Calibration check procedures are stated in applicable test method SOPs.

Preparation of standards must be performed using Class A glassware. Class A glassware must be used for all processes involving quantitative analyses. The only exception to this is when the method specifically requires or recommends plastic (ie. EPA 537.1).

Reference standards of measurement in the laboratory's possession (such as calibration weights or traceable thermometers) are used for calibration only and no other purpose.

Standards and reagents are uniquely identified as outlined in Westboro SOP 1745 and Mansfield SOP 1816.

9.4 Calibration General Requirements

Each calibration record is dated and labeled with method, instrument, analysis date, analyst(s) and each analyte name, concentration and response. For electronic processing systems that compute the calibration curve, the equation for the curve and the correlation coefficient are recorded in the appropriate instrument logbook. This is also true for manually prepared curves. Calibrations are tagged to the specific instrument through use of the instrument logbook and or sequence file documentation.

Initial calibration requires a standard curve that brackets the expected sample concentration. Initial calibration generally uses three to five standards depending on the equipment and

reference method specifications. Before the start of each analytical sequence, initial calibration is verified by using a continuing calibration standard. Calibration verification or continuing calibration uses the same standard as the ICAL unless method specifies otherwise. The ICV is from a second source or lot number than that used for initial calibration. The acceptance criteria for the continuing calibration standard must meet acceptance criteria before analysis of any samples. When the acceptance criteria is not within limits, review maintenance protocols and perform any necessary maintenance before starting the initial calibration sequence.

9.5 Equipment Calibration

The SOP used for the analysis defines the instrument and equipment calibration required. The following defines the general practices for equipment calibration of selected equipment.

9.5.1 Gas Chromatography/Mass Spectrometry (GC/MS)

The GC/MS is hardware tuned before performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. For volatiles analyses, bromofluorobenzene (BFB) is used, and for semivolatiles analyses, decafluorotriphenylphosphine (DFTPP) is used for instrument tuning.

The mass spectrometer response is calibrated by analyzing a set of five or more initial calibration solutions, as appropriate, for each GC/MS method. Each solution is analyzed once, unless the method or the customer requires multiple analyses. The relative response factor for each analyte is calculated for internal standard calibration. The calibration factor for external standard calibration is calculated using the expressions found in the laboratory method SOP. Calibration is acceptable when all acceptance criteria are within method criteria.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated and must be less than the acceptance criteria stated in the method.

An acceptable continuing calibration run must have measured percent differences for the analytes within method specified ranges. If any criteria for an acceptable calibration are not met, either instrument maintenance must be performed until the continuing calibration analysis meets all criteria or a new initial calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Additional quality control samples are part of the GC/MS analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.2 Gas Chromatography (GC)

Internal standard calibration or external standard calibration is utilized for analysis by GC. The method-specified number of calibration standards is used. Each solution is analyzed once and the analyte relative response factors or calibration factors are calculated. The mean relative response factor for each analyte is then obtained by using the expression in the formula listed in the SOP. Integrated areas are utilized for these expressions.

For multiple response pesticides, PCBs or hydrocarbons the quantitation consists of the average of selected peaks or the integration of the area defined by a reference standard. The SOP details the integration criteria for each compound.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours or 20 samples. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated. The percent drift (%d) may be calculated when calibration factors are used for quantitation.

An acceptable continuing calibration must have measured percent differences or percent drift for the analytes within method specified ranges. Should any criteria for an acceptable calibration not be met, either instrument maintenance is performed until the continuing calibration analysis meets all criteria, or a new calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Other standard checks may be required for a specified reference method. Instrument performance checks specified in the reference method must be performed and be within the acceptance limits stated in the reference method. Additional quality control samples are part of the GC analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.3 Cold Vapor Atomic Absorption Spectrophotometry (CVAA)

An initial calibration is performed daily with freshly prepared working standards that bracket the expected concentration range of the sample. A minimum of a five-point calibration curve is acquired which must have a correlation coefficient of 0.995 or better. The initial calibration is verified at the beginning of the sequence and every 10 samples. The continuing calibration is required to be within method-defined criteria, depending on the analytical method employed. Continuing calibration blanks are run at the same frequency. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within ± 5% of the true value for EPA Method 245.1 or ±10% for EPA 7470A and EPA 7471B.

9.5.4 Inductively Coupled Plasma Emission Spectrophotometry-Mass Spectrometry (ICP-MS)

Initial calibration and instrument tune is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of a minimum of three standards and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within method-defined criteria. The continuing calibration is required to be within method-defined criteria. Interference check standards are performed at the beginning of the sequence. Acceptance criteria are stated in the SOP.

9.5.5 Inductively Coupled Plasma Emission Spectrophotometry (ICP)

Initial calibration is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of one standard and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 5% of the true value for EPA Method 200.7 and 10% for SW846 6010 methods. The continuing calibration is required to be within 10% of the true value. Interference check standards are performed at the beginning and end of the sequence. Acceptance criteria are stated in the SOP.

9.5.6 Thermometers

Laboratory thermometers are checked annually for accuracy against certified, NIST traceable thermometers. Correction factors derived from the annual calibrations are applied to temperature readings where applicable. The analyst records the corrected temperature for all observations.

NIST traceable thermometers are calibrated professionally and re-certified every year. Records of thermometer calibrations are retained by the QA Department. All thermometers are tagged with the ID number, correction factor to be applied and the expiration of the calibration check.

NOTE: Electronic-based thermometers are calibrated on an annual basis. Thermometers are tagged with calibration information by the vendor, including the ID number, correction factor to be applied and the expiration of the calibration check. Certificates are kept on file in the QA Department.

Thermometers are not used past the calibration expiration date or if the thermometer is not reading properly. Replacement thermometers are calibrated and the maintenance logbook is updated when a change in the thermometer is required due to breakage, damage or expired calibration.

9.5.7 Balances

Calibration checks are performed for each day of use, for each balance. The calibration consists of a minimum of two weights, which bracket the weight to be measured. Additional calibration check procedures are performed on balances utilized in Microbiology laboratory. This additional procedure consists of a deflection test, which is performed to ensure that 100mg is detectable at a weight of 150 grams.

The balance logbook lists the acceptance criteria and performance criteria for the various balances used in the laboratory. Calibration weight measurements must meet the acceptance criteria listed on the record form.

Each balance is serviced and calibrated by a professional semi-annually. Balances are labeled with the balance number, date of service and the expiration date for the annual service check. The balance number used for any measurements requiring traceability is recorded with measurement data. Balances are not used past the expiration date or when the weight check is not within acceptable criteria. The accuracy of the calibration weights used by Alpha Analytical is verified annually by an accredited calibration service.

9.5.8 Mechanical volumetric pipettes

Delivery volumes for the mechanical volumetric pipettes (i.e. Eppendorf) are checked and recorded gravimetrically before use and on a quarterly basis. The verification is performed at the volume of use or bracketing the volume range of use. The check must be within the criteria stated in the laboratory logbook. Pipettes failing acceptance criteria are tagged and removed from service until repaired and the criteria are met, or discarded and replaced. Automatic pipettes are labeled with a unique ID number, volumes verified and expiration date.

9.5.9 Ion Chromatography

The ion chromatograph calibration is by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. The concentrations must bracket the expected concentration range of the samples analyzed. Procedures for verifying the calibration curve are method specific. The initial calibration is performed at the start of each day. The calibration curve is verified at least after every 20 samples.

9.5.10 pH Meters

pH meters are calibrated prior to use for each day of use. The meter is calibrated following the procedure for pH analysis. The records of the calibration are recorded in an instrument logbook or in the raw data for the analysis being performed. At least two buffer solutions that bracket the measurement range for the analysis are used for calibration. A second source check standard is used at the end of a run to verify meter stability. Buffer solutions used for calibration are NIST certified. Standard buffer solutions are not retained or re-used. The lot number of the buffer solutions is recorded in the data record to ensure traceability of the measurement to NIST.

9.5.11 Conductivity Meters

Three calibration standards of potassium chloride (KCL) solutions are analyzed annually on each instrument range. The calibration standards are used to verify instrument performance. The acceptance criteria are defined in the test SOP. If unacceptable performance is found, the cell is cleaned and rechecked. The cell is not used until satisfactory performance is achieved.

A single KCL standard solution is used to calibrate each range of the instrument. A second standard is used to check the calibration each day the meter is used. The check standard is near the measurement range for the samples to be analyzed. The acceptance criterion is $\pm$ 20% of the true value. The meter is labeled with expiration date for the annual calibration. A check standard that is NIST traceable is used to allow traceability. The check standard is performed at the end of the analysis run or at least after every 20 samples.

9.5.12 Autoclave

The date, contents, sterilization time and temperature, total cycle time and analyst's initials are recorded each time the autoclave is used. Autoclave cycles must be completed within 45 minutes when a 15 minute sterilization time is used. Autoclave timing mechanisms are checked quarterly with a stopwatch to verify timing controls. A maximum temperature thermometer is used with each cycle to ensure the sterilization temperature is reached.

Spore strips or ampoules are used weekly to confirm sterilization. BTSure ampoules are utilized as follows: An indicator ampoule is placed in most challenging area of sterilizer. Load is processed according to standard operating instructions. Remove from sterilizer and allow to cool for a minimum of 10 minutes. (Chemical indicator on label changes from green to black when processed.) Place the autoclaved indicator and un-autoclaved control indicator in an upright position in the plastic crusher provided. Gently squeeze crusher to break glass ampoules. Incubate both indicators at 55-60^{o}C for 24 hours. Examine appearance for color change. Yellow color indicates bacterial growth. No color change indicates adequate sterilization.

Calibration is conducted and certified annually by an outside service provider and recorded. Certificates are kept on file. Routine maintenance includes cleaning the autoclave seal to ensure freedom of caramelized media and cleaning drain screens to remove any debris buildup. For the efficient operation of the unit, overcrowding is avoided.

10 Test Methods and Standard Operating Procedures

10.1 Methods Documentation

Analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations, monitoring instrument performance tests, analyzing prepared samples, and collecting data from the analyses. The test method SOP describes the instrumental analysis procedures, quality control frequencies and acceptance criteria. EPA accepted methods, national recognized methods or customer-specified methods are the basis for performance criteria, instrument conditions and the steps of the procedure. The method performance requirements of the published methods are followed unless otherwise specified by the customer.

The reference methods define the instrument operating conditions. In many of the reference methods, a range or general guidance on the operating conditions is defined. Documented modifications to the operating conditions clarify the reference methods or improve the quality of the results. In all cases where the method modifications are adopted, the performance criteria from the reference method must be met. Modifications to the operating conditions are stated in the SOP. Changes in the operating conditions made at the time of the analysis are documented in the appropriate laboratory or sequence log. A revision to the SOP takes place, when a day to day change in the operating condition improves performance for all matrices.

The laboratory SOPs include the operation of measurement equipment. The SOPs contain the - following information, as applicable:

- The equipment used in the procedure, including equipment type
- Equipment calibration and process for obtaining the measurement from the calibration
- The step by step instructions to perform the measurement
- Acceptance criteria for the calibrations
- Corrective action for failed acceptance criteria, including assessment of previous calibration results
- The basis used for the calibration standards such as traceability to NIST or EPA or demonstration of comparability
- Frequency at which the equipment will be calibrated, adjusted and checked
- The records maintained to document the calibration and use of measurement equipment
- The calibration status for the equipment
- The environmental conditions necessary before measurement equipment may be calibrated or used for measurement
- Allowed adjustments to measurement equipment, including software, which will not invalidate the laboratory analysis
- Maintenance of the equipment and record keeping to track performance before and after maintenance is completed
- Define the standards, reagents and sample handling, interferences, preservation, and storage in order to assure measurement performance

10.2 Standard Operating Procedures (SOPs)

Alpha Analytical maintains SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, nonconformance actions, handling customer complaints, sample receipt and storage, purchasing of all materials, and all test methods. These documents include equipment manuals provided by the manufacturer, internally written documents, and published methods with documented changes or modifications.

Copies of all SOPs are accessible to all personnel in electronic form through Qualtrax. Each SOP clearly indicates the published date of the document and the revision number.

10.3 Laboratory Method Manual (s)

All SOPs are posted as secure documents in the Alpha Qualtrax system. Directories are available for each laboratory area and administrative area in appropriate subfolders. Each SOP includes or references where applicable:

1) identification of the test method and where applicable;
2) applicable matrix or matrices;
3) method detection limit;
4) scope and application;
5) summary of method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies
10) reagents and standards
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for quality control measurements;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and
23) any tables, diagrams, flowcharts and validation data.

In cases where modifications to the published method have been made by the laboratory or where the referenced method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described in the SOP.

10.4 Test Methods

The laboratory uses appropriate methods and procedures for all tests and related activities within its responsibility (including sampling, handling, transport and storage, preparation of items, estimation of uncertainty of measurement and analysis of test data). The method and procedures are consistent with the accuracy required, and with any standard specification relevant to the calibrations or tests concerned. When the use of mandated methods for a sample matrix is required, only those methods are used. Where methods are employed that are not required, the methods are fully documented and validated and are available to the customer and other recipients of the relevant reports.

The customer requests the reference method for sample analysis usually based on the regulatory program. The customer services staff may assist the customer with method selection when the customer specifies the regulatory program, but is unsure of the correct method required. The Laboratory Technical Manager or Quality Assurance Officer recommends methods for non-regulatory programs. In all cases, recommendation of methods is based on customer-defined method performance criteria. Customer services may recommend a procedure that meets the customer method performance criteria.

10.5 Method Validation/Initial Demonstration of Method Performance

Before acceptance and use of any method, satisfactory initial demonstration of method performance is required. In all cases, appropriate forms are completed and retained by the laboratory and made available upon request. All associated supporting data necessary to reproduce the analytical results is retained. Initial demonstration of method performance is completed each time there is a significant change in instrument type, personnel or method.

10.6 Sample Aliquots

The aliquot sampling process from a submitted sample is part of a test method. The laboratory uses documented and appropriate procedures and techniques to obtain representative sub-samples. Sample aliquots removed for analysis are homogenized and representative portions removed from the sample container. Personnel record observations made during aliquot sampling in the test method logbooks.

10.7 Data Verification

Calculations and data transfers are subject to appropriate checks which is a 3 tier approach. The initial analyst verifies all of his work, a secondary review of 100% of the initial is conducted by a an independent qualified analyst. A Customer Services representative reviews data for project and method performance requirements where applicable. A QA representative reviews data for project and method performance requirements when requested by a Customer. Final report review is performed by an authorized company signatory.

For drinking water suppliers, every effort is made to notify the Customer within 24-hours of obtaining valid data of any results that exceed any established maximum contaminant level or reportable concentration. Analyst or Department Supervisor notifies the Customer Services Department of the sample number(s), Customer name, analysis and sample results (preliminary or confirmed). The Customer Services Department notifies the customer.

The laboratory Report Generation and Approval SOP describes the practices to ensure that the reported data is free of transcription errors and calculation errors. Manually entered data into the LIMS is dual entered and checked by the LIMS to minimize transcription errors. The laboratory test method SOP describes the quality control measures used to assure method performance before reporting data.

10.8 Labeling of Standards and Reagents

The purchase, receipt and storage of consumable materials used for the technical operations of the laboratory include the following:

a) The laboratory retains records of manufacturer’s statement of purity, of the origin, purity and traceability of all chemical and physical standards.
b) Original reagent containers are labeled with the date opened and the expiration date.
c) Detailed records are maintained on reagent and standards preparation. These records indicate traceability to purchased stocks or neat compounds and include the date of preparation and preparer's initials.

d) Where calibrations do not include the generation of a calibration curve, records show the calibration date and type of calibration standard used.
e) All prepared reagents and standards are uniquely identified and the contents are clearly identified with preparation date, concentration and preparer's initials. These procedures are outlined in Westboro SOP/1745 and Mansfield SOP/1816.

10.9 Computers and Electronic Data Related Requirements

Computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data. The laboratory ensures that computer software and firmware is documented and adequate. The goals of the software development methodology, existing system validations and the change control system are to ensure that:

the software systems perform the required functions accurately,

the users understand how to use the system, and

auditors can assure themselves of the validity of the analytical data.

The computer systems used at Alpha Analytical are purchased. A coordinated effort is made with the supplier to assure the computer operations meet the laboratory requirements for data integrity. Alpha Analytical has a formal validation program of its computer systems. The validation program is a comprehensive program to ensure data transmitted, reported or manipulated by electronic means is correct and free of errors. The validation and verification approach is separated into three areas.

1. New software is developed and validated using test data. Records of validation include the test data report, date and initials. Where formulas are part of the program, documentation includes manual verification of the final calculated values. New software includes the development of macros for spreadsheets and other tools using commercial software packages.

2. Reasons for changes to software are identified through flaws in existing documentation or the need to improve system processes and are documented on the Nonconformance Report. Final implementation of the change is documented on the nonconformance action form. The tracking and timelines of making the change is readily available. This process also provides the complete documentation of all software and electronic data reporting problems. All nonconformance identified with electronic data process result in corrective action that are reported to management before or at the bi-weekly executive meeting. Customers will be notified prior to any changes to software or hardware that will adversely affect customer electronic data. This information is provided by IT department to QA and Project Managers to be communicated to appropriate customers.

Verification of system integrity is through routine maintenance, protection from unauthorized access and electronic verification programs. Routine maintenance including system backups are performed on a scheduled basis. The backup process and password and access protections are defined in the Computer System Backup Control SOP/1562 and Computer Security SOP/1563. Electronic verification may be used to assure the commercially purchased software is performing at its original specifications. This includes virus checking of all network operation at least once per week. Documentation of all verification and maintenance operations is retained.

11 Sample Handling, Sample Acceptance Policy and Sample Receipt

The Sample Login and Custody procedures define the process for sample management from sample receipt through analysis and to disposal. These procedures detail the process for sample receipt, records and storage pending analysis.

Customers or Alpha's Couriers deliver samples to the laboratory during normal business hours. Sample receiving occurs in the sample management area.

Customer service personnel place bottle orders. The orders are filled following the bottle order instruction form. Blanks are prepared as needed with minimal storage. All glass containers are packed to minimize or prevent breakage. The containers are placed in plastic coolers or shipping packages and Chain-of Custody forms, seals (if requested) and labels enclosed. The bottle order is shipped by third party, picked up by the customer or customer representative or delivered by Alpha courier to the customer.

11.1 Sampling Supplies

11.1.1 Sample Containers

Sample containers provided by Alpha Analytical include labels, preservatives and a blank chain of custody form. Preservatives and containers are lot controlled and verified as appropriate for the indicated type of analysis.

Each lot of containers used for the collection of samples for microbiological analysis is checked for sterility prior to distribution. Sterility checks are performed by Microbiology staff and results recorded in Microbiology Sample Container Sterility Log.

Sample Containers for collecting Air samples (TO-15) are cleaned and prepared according to SOP 2190 "Cleaning and Preparation Procedures for Equipment used to collect Air sample for analytis of Volatile Organic Compounds".

11.1.2 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

11.1.3 Reagent Water

Alpha Analytical supplies laboratory pure water for field QC blanks. Water used for volatile organics must be free of volatile compounds below the method detection limit. The quality of the laboratory water is monitored for conductivity once per day. Additional water quality criteria may be monitored based on customer specific requests. The water quality in the laboratory is monitored for chemical parameters as required by the EPA certification manual for drinking water (Water Quality Monitoring SOP/1738).

11.2 Sample Tracking

Alpha Analytical uses an internal chain-of-custody in LIMs for sample tracking control purposes. When requested or required by regulation a legal custody program is used in addition to the routine laboratory practices. Legal custody practices must be arranged at the time of contractual commitment.

For legal custody the process must include complete and continuous records of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For legal custody a sample is in someone's custody if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked up so that no one can tamper with it;
4. It is kept in a secured area, restricted to authorized personnel only.

The routine sample handling and tracking process includes unique identification of all sample containers, initials of the person removing the sample from the sample management area and documentation of the date of sample removal for disposal.

Samples are assigned a unique identification number from the LIMS program. Each sample container label includes a unique identifier for the container. The person handling the sample is recorded along with the unique identifier in the container tracking records in LIMS.

ALPHA ANALYTICAL utilizes a custom designed Laboratory Information Management System (LIMS) to uniquely identify and track samples and analytical data throughout the facility. The LIMS log-in, is initiated by the Sample Custodian when the following information is entered into the computer:

- Quote number (unique to the project if requested)
- Project name or description
- Analyses requested (per matrices received)
- Sample number (unique to this sample)
- Sample descriptions (customer ID, including number of received containers)
- Date received
- Date(s) and time(s) collected
- Date analytical results are due

11.2.1 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

- Customer's name and address
- Notation of special handling instructions
- Additional comments or instruction for the laboratory
- Purchase order number(s), if applicable

Alpha Job Numbers (Process for assigning numbers)

Alpha Job Numbers are unique #'s automatically designated by our LIMS computer system for every individual customer project.

There are 3 parts to this number:

- All numbers start with the letter "L"
- The next two numbers are the last two numbers of the current year.
- The last five numbers are pulled sequentially by the LIMS as each Login personnel requests a new number for a job.

For example…. L0904165 ---- Year 2009 and 4,165th job to be logged in this year.

The Alpha Job Number then may contain as many extensions as there are individual samples in a job. L0904165-01 is the first sample, L0904165-02 is the second and so on. Each sample may contain as many as 26 containers as the containers are designated with the letters of the Alphabet, and each container receives its own bar-coded label. For example, L0904165-09A is the first container of the 9th sample listed on a customer's Chain of Custody.

Each container is labeled with a unique identifier, a label with a unique identifier number is placed on each sample container. Once labeled, the sample containers are placed in the appropriate storage area.

11.3 Sample Acceptance Policy

The sample management personnel check for proper sample labeling, preservation and handling at the time of arrival at the laboratory. The customer and customer services manager specifies the proper sample preservation, containers, cooling and other criteria on the project review form and in the LIMS. Sample management staff record all observations and immediate notify customer services of any discrepancies or questions arising during sample receipt.

It is possible for samples or sample containers to be lost, damaged, or determined to be unsuitable, for whatever reason, after initial receipt at Alpha Analytical. The problem is brought to the attention of a customer services manager who reports it to the customer. Plans for disposition of the affected samples or container are agreed upon with the customer, carried out, and recorded in the project records. Sample hold times and preservations are listed on the Alpha website (www.alphalab.com) under Support Services "Sampling Reference Guide".

11.4 Sample Receipt Protocols

The sample management staff receives all samples. A unique job number is assigned to each shipment of samples received from a customer. The in-house records for the incoming job, including the internal Chain-of -Custody, are initiated with a Sample Delivery Group (SDG) form. The customer, and Alpha courier and/or the sample management personnel sign the sample custody form at the time of receipt at the laboratory. Samples received via overnight courier are signed on the bill of lading. The bill of lading, SDG form and the sample custody form are completed for external courier delivered samples.

The sample management staff examines the shipping containers, their contents, and accompanying customer documentation. Information about the sample identification, the location, date and time of collection, collector's name, preservation type, sample type, presence and condition of custody seals, the state of preservation of the samples and other required information is noted on the SDG form. Any discrepancies in documentation or problems with sample condition such as appropriate sample containers, thermal preservation variation, holding times and adequate sample volumes are noted and brought to the attention of the customer via the nonconformance action form, The login staff or project manager contacts the client via email or or by phone. The Customer Services Manager provides clarification or further instruction to the sample management staff on the processing of the samples that are incomplete or missing required information.

The sample management staff logs the samples in the LIMs and a durable label for each container is printed. The custodian attaches each label to the appropriate sample container. The following information is recorded for tracking internal custody: laboratory sample ID, customer sample ID, sample matrix and storage location. Sample receipt and log-in specifically requires: date and time of laboratory receipt of sample(s); sample collection date; unique laboratory ID code; field ID code supplied by sample submitter; requested analyses; signature or initials of data logger; comments from inspection for sample acceptance or rejection and in some cases, sample bottle codes.

11.5 Storage Conditions

Alpha Analytical stores samples under proper environmental conditions to ensure their integrity and security. Samples are stored at temperatures that meet specifications of the methodology, regulatory agencies and customer directives. Refrigerators are monitored and controlled to be within 4 ± 2°C. Chemical, temperature, holding times and container storage requirements are listed in the LIMS project database.

Customer Quality Assurance Project Plans may list preservation requirements differing from the laboratory. The sample management staff reviews project information for projects specific handling. Addition of chemical preservative to sample containers normally is done in the field at the time of sampling. Chemical preservation and temperature preservation checks at the time of receipt are recorded except for volatile organic compounds, bacteria, sulfite, and dissolved oxygen preservation. Any differences from laboratory or customer specific requirements are recorded on nonconformance action forms and contact made with the customer by the Customer Services Manager or designee.

Sample storage facilities are located within the sample management area, walk-in custody refrigerator or in designated sample storage areas within the analytical departments. Internal chain-of-custody procedures and documentation pertaining to sample possession, removal from storage, and transfer are outlined in the sample custody procedure. Samples are returned to the sample storage area after the sample portion is removed for analysis. Extracts and digestates are tracked and follow the same internal custody operation. Extracts and digestates are removed to the waste disposal area after analysis for proper disposal.

Sample storage precautions are used to ensure that cross contamination does not occur during sample storage. Refrigerator storage blanks are monitored bi-weekly for volatile compounds.. The storage blank information allows the assessment of potential cross contamination in the sample storage refrigerator.

Temperatures of cold storage areas are recorded continuously in the data logger system. Corrective action is done as necessary when temperatures are not within the control criteria. In both the Westboro and Mansfield facilities, Automated Data loggers are linked to thermocouples in custody refrigerators and freezers in the Sample Storage areas as well as department standards/storage refrigerators and freezers. The Data logger is calibrated and certified by an outside vendor annually and on a quarterly basis for DOD standards/storage refrigerators and freezers. If there is a catastrophic failure of custody refrigerators, a record of all samples affected and customers associated with such samples are notified of any samples affected by the failure. Refrigerators and/or freezers not connected to the Data Logger system have temperatures measured with NIST traceable thermometers. Temperature records indicate the thermometer or sensor (Data logger) used for obtaining the measurement.

11.6 Sample Disposal

Samples are held for 21 calendar days after the report is released to the customer. Upon written customer request samples may be held longer in an uncontrolled area. Requests for controlled

sample storage must be arranged at the time of contractual commitment. Air canister samples are held for 3 days after the report is released to the customer.

An authorized waste carrier is contracted to pick up waste as needed and dispose of it, in accordance with all regulatory requirements. Post-analysis disposition of samples is dependent upon project specific requests. Remaining sample material may be returned to the customer, safely discarded, or archived for a specific time prior to disposal. The waste disposal SOP 1797 defines the specific requirements for sample disposal and other waste disposal operations.

The sample management staff are responsible for the archival and disposal of raw samples, extracts and digestates. Raw and prepared samples may not be archived or disposed until all of the designated analyses are complete and resultant analytical data is sent to customers. Samples in storage are retained a minimum of 21 calendar days after reporting the results to the customer. Any samples requiring more than 21 calendar days are archived. Air canister samples requiring storage more than 3 business days require prior approval.

When a customer has requested the return of samples, the sample management staff prepares and ships the samples according to the same custody procedures in which the samples were received and following any customer specified requirements. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the control of this laboratory. Customers are informed that a commercial carrier will deliver their samples if required**.**

12 Records

Alpha Analytical has a record system that produces accurate records, which document all laboratory activities. The laboratory retains records of all original observations, calculations and derived data, calibration records and a copy of the test for ten years minimum. The system retains records longer than the minimum upon the request of authorized customers, agencies or another regulator. Note: Ohio VAP requires notification before disposal of any VAP records.

12.1 Record Keeping System and Design

The record keeping system allows reconstruction of laboratory processes that produced the analytical data of the sample.

a) The records include the names of personnel involved in sampling, preparation, calibration or testing.

b) Information relating to laboratory facilities equipment, analytical methods, and activities such as sample receipt, preparation, or data verification are documented.

c) The record keeping system provides retrieval of working files and archived records for inspection and verification purposes.

d) Documentation entries are signed or initialed by responsible staff.

e) Generated data requiring operator logging on appropriate logsheets or logbooks are recorded directly and legibly in permanent ink

f) Entries in records are not obliterated by any method. Corrections to errors are made by one line marked through the error. The person making the correction signs and dates the correction.

g) Data entry is minimized by electronic data transfer and ensuring the number of manual data transcriptions is reduced.

12.2 Records Management and Storage

1. Records including calibration and test equipment, certificates and reports are safely stored, held secure and in confidence to the customer.
2. The laboratory maintains hardware and software necessary for reconstruction of data.
3. Records that are stored or generated by computers have hard copy or write-protected backup copies.
4. Alpha Analytical has established a record management system, for control of hard copy laboratory notebooks.
5. Access to archived information is carefully controlled. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources. Any access to the archive is documented in the Data Archive Access Logbook which is used strictly by the QA Department.
6. In the event that Alpha Analytical transfers ownership or goes out of business, there is a plan to ensure that the records are maintained or

transferred according to the customer's instructions. A plan will be developed to maintain continuity of our record keeping systems as requested and/or required by both state and federal laws.

Alpha Analytical retains all original hard copy or electronic raw data for calibrations, samples, and quality control measures for ten years, including:

1. Analysts work sheets and data output records,
2. Reference to the specific method,
3. Calculation steps including definition of symbols to reduce observations to a reportable value,
4. Copies of all final reports
5. Archived SOPs,
6. Correspondence relating to laboratory activities for a specific project,
7. All nonconformance action reports, audits and audit responses,
8. Proficiency test results and raw data,
9. Data review and cross checking.

The basic information to tie together analysis and peripherals such as strip charts, printouts, computer files, analytical notebooks and run logs for Alpha Analytical includes:

1. Unique ID code for each Laboratory sample or QC sample;
2. Date of analysis;
3. Instrument identification and operating conditions;
4. SOP reference and version;
5. Calculations;
6. Analyst or operator's initials/signature.

In addition, Alpha Analytical maintains records of:

1. Personnel qualifications, experience and training
2. Initial and continuing demonstration of proficiency for each analyst
3. A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory records. Use of electronic signatures has been approved by regulatory agencies.

12.3 Laboratory Sample Tracking

A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include but are not limited to records pertaining to:

a) Sample preservation including appropriate sample container and compliance with holding time requirement; If the time of the sample collection is not provided, the laboratory must assume the most conservative time of day (i.e., earliest).

b) Sample identification, receipt, acceptance or rejection and log-in;

c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records; this includes inter-laboratory transfers of samples, extracts and digestates.

d) Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

e) Sample analysis;

f) Standard and reagent origin, receipt, preparation, and use;

g) Equipment receipt, use, specification, operating conditions and preventative maintenance;

h) Calibration criteria, frequency and acceptance criteria;

i) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

j) Method performance criteria including expected quality control requirements;

k) Quality control protocols and assessment;

l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

m) Automated sample handling systems;

n) Records storage and retention; and

o) Disposal of hazardous samples including the date of sample or sub-sample disposal and the name of the responsible person.

p) The COC records account for all time periods associated with the samples.

q) The COC records include signatures of all individuals who had access to individual samples. Signatures (written or electronic) of all personnel who physically handle the samples. Time of day and calendar date of each transfer or handling procedure.

r) Common carrier documents.

13 Laboratory Report Format and Contents

The Process Planning and Control Procedure details the recording and reporting of data as required by the customer and in accordance with relevant environmental regulations.

Customers specify the report delivery and deliverables required for the work submitted. Report delivery includes standard turnaround and rush turnaround. Customers specify the delivery address or multiple addresses and method of delivery such as U.S. Mail, facsimile or electronic at the start of the project. Alpha Analytical provides data deliverables in hardcopy or electronic format. At the start of any project, the electronic deliverable formats required must be received before sample arrival. Affidavits are required with each report or series of reports generated for a particular project for Ohio VAP reports.

Reporting packages are available for routine regulatory reporting requirements. Regulatory reporting packages include only the information requested by the regulatory agency. In addition to regulatory report packages, Alpha Analytical prepares a standard report format. The standard report format includes:

1. Title: “Certification of Analysis”
2. Name and address of the laboratory
3. Laboratory Job Number, page number and total number of pages included in the report.
4. Name and address of the customer
5. Alpha sample number, Customer identification, Sample location
6. Samples identified that do not meet the sample acceptance requirements for project.
7. Date of sample receipt, sample collection, preparation or extraction date and time (if applicable), analysis date and time, report date and analyst
8. Identification of data reported by subcontractors
9. Test name and reference method number
10. Delivery method and sampling procedures when collected by lab personnel
11. Deviations or modifications that affect data quality and/or data integrity. These deviations or modifications are included in narrative statements and/or data merger files.
12. Statement that results relate only to the sample tested
13. Statement that report must be copied in full unless the laboratory provides written permission for partial copies
14. Glossary, References and limits of liability
15. Units of measure and reporting detection limit
16. Quality control data for: % Recovery surrogates, % Recovery of LCS, % RPD of LCSD, Blank analysis, % Recovery Matrix Spike, %RPD of Laboratory Duplicates, as applicable
17. Signature, title and date of report

18. A “Certificate/Approval Program Summary” page is included at the end of the report that identifies analytes for which Alpha Analytical holds certification and for those analytes reported that it does not. This summary also includes the certification numbers for either NELAP certified states, State certifications (e.g. Massachusetts laboratory certification identification number)..
19. Alpha Analytical does not accept samples from private residents for drinking water analysis and therefore maximum contaminant levels are not necessary. If Alpha were to change its policy and report drinking water samples, MCLs would be included with the report.

Results transmitted by facsimile or other electronic means include a statement of confidentiality and return of the materials at the laboratory’s expense.

The laboratory notifies the customer in writing of any circumstance that causes doubt on the validity of the results. The amended or modified report lists the change, reason for the change, affected page numbers, date of the amendment and authorized signature. The customer will be notified prior to changes in LIMs software or hardware configurations that will adversely affect customer electronic data.

13.1 Data Qualifiers

The following data qualifiers are used in conjunction with analytical results depending on the definition, state or regulatory program and report type.

Note: ”J” Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Method Detection Limit (DL) with a “J” qualifier as long as there is a LOD study on file. (See section 5.11)

Data Qualifier	Qualifier Information	Regulatory Requirement
A	Spectra identified as "Aldol Condensation Product".	CT RCP, NC
B	The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at <5x the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than 10x the concentration found in the blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone) For DOD related projects, flag applies to detectable concentration of target analyte in the blank that exceeds ½ the LOG or is greater than 1/10 the concentration in the field sample	EPA Functional Guidelines 'MassDEP MCP, CT RCP, NJ-TO15/LL-TO15; NJ Tech Guidance 2014, DOD QSM 5.4
C	Co-elution: target analyte co-elutes with a known lab standard (i.e. surrogates, internal standards, etc.) for co-extracted analyses.	
D	Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.	NJ-TO15/LL-TO15 - Air only EPA Functional Guidelines; EPA Region 2,5
DL	Same was re-analyzed at a dilution. Qualifier applied to sample number.	

E		Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.	EPA Region 2,5 CT RCP, NJ-TO15/LL-TO15
G		The concentration may be biased high due to matrix interferences (i.e. co-elution) with non-target compound(s). The result should be considered estimated.	In-house/Forensics.
H		The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.	THE NELAC INSTITUTE (TNI) STANDARDS
I		The lower value for the two columns has been reported due to obvious interference.	In-house.
J		Estimated value. This represents an estimated concentration for Tentatively Identified Compounds (TICs).	CT RCP (for TICs),
JN (NJ)		Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search.	EPA Functional Guidelines 'NJ-TO15-LL
ND	DU-J	Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for same-related analysis	In-house
P	All DU	The RPD between the results for the two columns exceeds the method-specified criteria.	MassDEP MCP, CT RCP
Q	All DU	The quality control sample exceeds the associated acceptance criteria. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)	
R	All DU	Analytical results are from sample re-analysis	Customer-specific

RE	All DU	Analytical results are from sample re-extraction.	Customer-specific
S		Analytical results are from modified screening analysis	

13.2 Compound Summation for Organic Analyses

In order to be compliant with regulations from certain states, Alpha Analytical has created the following Summation Rules to cover reporting “Total Analytes”. The following are an example of several compounds that can be reported as “Totals”:

Volatiles:	
1,3-Dichloropropene, Total	cis + trans isomers
Xylenes, Total	m/p + o isomers
1,2-Dichloroethene, Total	cis + trans isomers
Trihalomethanes, Total	Chloroform + Bromoform +
	Dibromochloromethane +
	Dichlorobromomethane
PCBs:	
PCBs, Total	Sum of reportable Aroclors
	(all Aroclors reported for the project)

The following are the summation rules that the LIMs uses to calculate the Total values:

Summation Rules:		
H + H = H		Key:
H + J = J		H = Hit (above RL)
J + J = J		J = J-flagged value
H + ND = H		ND = U-flagged value
J + ND = J		
ND + ND = ND		

The ND values are considered “0” during the calculations.
The “E” flagged values (over the calibration) are ignored and not utilized during the calculations.
Any “N” flagged values (do not report) are ignored and not utilized during the calculations.
For dual-column analysis, the Total is reported as part of column “A” data, unless all individuals are reported from “B” column.

For analytical group summations, the Total is reported based on the associated "Reporting List".
For example, if only 7 Aroclors are requested, then the Total is based on 7 Aroclors, not 9.

The RL and MDL for Totals will always be the lowest of the individual compounds used in the summation.

For each Total summation, two values are calculated: TOTALH (calculated from all associated hits above the R L– used in DU reporting formats) and TOTALJ (calculated from all associated hits and J flagged values – used in DJQL reporting formats). Total concentrations are calculated for all samples and QC samples (however, recoveries are not calculated since they are only calculated for the compounds spiked)

If a Total summation is requested, the individual compounds must also be reported.

14 Outside Support Services and Supplies

When Alpha Analytical purchases outside services and supplies in support of tests, the laboratory uses only those outside services and supplies that are of adequate quality to maintain confidence in the tests. Differences between Request/Tender and Contracts must be resolved before work commences.

The Purchasing SOP/1726 describes approval and monitoring of all suppliers and subcontractors used by the laboratory. Where no independent assurance of the quality of outside support services or supplies is available, the laboratory ensures that purchased equipment, materials, and services comply with specifications by evaluating method performance before routine use.

The laboratory checks shipments upon receipt as complying with purchase specifications. The use of purchased equipment and consumables is only after the evaluation and compliance to the specifications is complete. The Purchasing SOP/1726 describes the details for receipt and inspection of purchased product.

The Purchasing SOP describes the process for raising, review and placement of purchase orders. It is company policy to purchase from third party certified suppliers and subcontractors wherever possible. Purchases must be from suppliers approved by the Laboratory. Laboratory or sampling subcontractors specified by the customer are noted as “Trial” on the purchase order. This identifies the subcontractor as a non-approved subcontractor. All DoD work that is subcontracted must comply with Alpha’s management system and must comply with the QSM standard and is subject to DoD customer approval.

The laboratory maintains list of approved vendors (Form 18302) and subcontractors from whom it obtains support services or supplies required for tests.

14.1 Subcontracting Analytical Samples

Customers are advised, verbally and/or in writing, if any analyses will be subcontracted to another laboratory. Any testing covered under the NELAC Institute (TNI) Standards that requires subcontracting, will be subcontracted to another THE NELAC Institute (TNI) Standard accredited laboratory for the tests to be performed. The laboratory approves testing and sampling subcontractors by review of current state, national or other external parties’ certifications or approvals. This document must indicate current approval for the subcontracted work. Any sample(s) needing special reports (*i.e.*, MCL exceedance) will be identified on the chain of custody when the laboratory subcontracts with another laboratory. Subcontractor Laboratory Certifications are located in Qualtrax under Customer Services folder

The Sample Receipt and Login Procedure describes the process for sample handling when subcontracting samples. Customer notification of subcontracted work is in writing or verbally before releasing samples to the subcontractor.

The review of subcontractor documents for completeness and meeting the specifications defined for the project follows the laboratory process for reporting and verification of process data. The Reporting Deparment Designee is responsible for receiving the order reviews the information supplied by the subcontractor instead of the Department Supervisor.

15 Customer Relations

15.1 Customer Service

The majority of the customer services occur from personnel in the administration, sample receiving and sampling areas. Customer service involves inquiries into services offered, technical consulting, placing orders, and receiving orders, providing updates on the status of orders and completing orders. Personnel interacting with customers must document and review customer specific project requirements. Call Tracker is used to document communications with customers (SOP/1723). Personnel must document customer interactions following the appropriate laboratory procedures. Each person must communicate deviations, modifications and customer requests following the laboratory defined procedures.

15.2 Project Management

During staff meetings the laboratory management reviews requests for new work. The Operations Director and/or Laboratory Technical Manager address all capacity and capability issues. Where conflicts in workload arise, customer notification is immediate. The Project Communication Form (PCF) contains the documentation of all project information. Cooperation between laboratory and customer services staff allows direct communication and scheduling. Management arranges complex scheduling and coordination between departmental areas. Documentation of approval for waivers from the DoD QSM requirements must be documented on a project specific waiver. This documentation needs to be in writing and readily available for review.

15.3 Complaint Processing

The laboratory staff documents all customers or other parties' complaints or concerns regarding the data quality or laboratory operations. The Nonconformance Report records complaints, correcting the concern, and resolving the concern with the customer or other party. The process uses the same form and process as the nonconformance action process. Where repetitive corrective actions indicate a problem, an audit of the area, Customer Inquiry and Complaint SOP/1722 is immediate to ensure the corrective action has effectively solved the concern.

16 Appendix A – Definitions/References

The following definitions are from Section 3.0 of the 2016 TNI Standard unless otherwise cited. The laboratory adopts these definitions for all work performed in the laboratory.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (EPA QAD glossary)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analyte:

A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. (TNI)

The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation).

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, which 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 (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample

in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.

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.

Blanks include:

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.

Field Blank: blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

Instrument Blank: a clean sample (e.g. distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Reagent Blank: (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Calibration: set of operations which establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

1) In calibration of support equipment the values realized by standards are established through the use of Reference Standards that are traceable to the International System of Units (SI).

2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the Laboratory with a certificate of analysis or purity, or prepared by the Laboratory using support equipment that has been calibrated verified to meet specifications.

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM): Reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.

Chain of Custody Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. See also Legal Chain of Custody Protocols.

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq*., Public Law 91-604, 84 Stat. 1676 Pub.L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation, Alternate wavelength, Derivatization, Mass spectral interpretation, Alternative detectors, or Additional cleanup procedures

Customer: Any individual or organization for which items or services are furnished or work performed in response to defined requirements and expectations. (ANSI/ASQ E4-2004)

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites.

Conformance: an affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Consensus Standard: A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof. (ANSI/ASQ ANSI/ASQ E4-2004)

Continuing calibration verification: The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. (IDQTF)

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.

Data Quality Objectives (DQO):

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

Definitive Data: Analytical data of known quality, concentration, and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making. (UFP-QAPP)

Demonstration of Capability: a procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Detection Limit: (previously referred to as Method Detection Limit –MDL) the lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit.

Detection Limit (DL) (Clarification): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Environmental Data: Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology. (ANSI/ASQ E4-2004)

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): the enabling legislation under 7 U.S.C. 135 *et seq*., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides.

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C 1251 et seq., Public Law 92-50086 Stat. 8.16, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance.

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Field of Proficiency Testing (FoPT): Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges, and acceptance criteria have been established by the PTPEC.

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.

Finding (Clarification): An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition (ANSI/ASQ E4-2004).

Holding Times: The maximum time that can elapse between two (2) specified activities. (TNI)

The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR part 136)

In-depth Data Monitoring: When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.

Inspection: An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard: 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.

Isomer: One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.

Laboratory: Body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (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. 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.

Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the customer. These protocols 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): The minimum result, which can be reliably discriminated from a blank with a predetermined confidence level. Also used is Detection Limit.

Limits 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.

For DOD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.

Lot: A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.

Management: Those individuals directly responsible and accountable for planning, implementing, and assessing work. (ANSI/ASQ E4-2004)

Management System: System to establish policy and objectives and to achieve those objectives (ISO 9000).

Matrix: The substrate of a test sample.

Matrix Duplicate: A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.

Matrix Spike (spiked sample, 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 (spiked sample or fortified sample duplicate): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Measurement System: A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

Method Detection Limit: One way to establish a Limit of Detection.

Method of Standard Additions: A set of procedures adding one or more increments

of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Mobile Laboratory: A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans and skid-mounted structures configured to house testing equipment and personnel.

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute. (NMI).

Physical Parameter: A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical or biological components.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis.

Primary Accreditation Body (Primary AB): The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.

Procedure: A specified way to carry out an activity or a process. Procedures can be documented or not.

Proficiency Testing: A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.

Proficiency Testing Provider (PT Provider): A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT program.

Proficiency Testing Provider Accreditor (PTPA): An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.

Proficiency Testing Reporting Limit (PTRL): A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.

Proficiency Testing Sample (PT): 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.

PT Study Closing Date:
a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider.
b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.

PT Study Opening Date:
a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT provider.
b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which 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 the type and quality needed and expected by the customer.

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control: 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 or 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: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking intended to demonstrate that a measurement system or activity is in control.

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, the ensure the quality of its product and the utility of its product to the users.

Quality Manual Clarification: Alpha Analytical refers to Quality Manual as Corporate Quality Systems Manual (CQSM). (Alpha)

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 quality assurance (QA) and quality control (QC) activities.

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 sorbent tube, impinger solution, filter, or other device.

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.

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, print outs of chromatograms, instrument outputs, and handwritten records.

Reference Material: Material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: (To be used to determine the extent of method validation in Modules 3-7.) A reference method is a published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a “standard method”, that term is equivalent to “reference method”). When a laboratory is required to analyze an analyte by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is not a regulatory requirement for the

analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location. (TNI)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage and disposal.

Revocation: The total or partial withdrawal of a laboratory's accreditation by an accreditation body

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Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent.

Sensitivity: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Signal to Noise Ratio: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Signatures, Electronic: A technology that allows a person to electronically affix a signature or its equivalent to an electronic document. The electronic signature links the signature to the signer's identity and to the time the document was signed. Alpha approves the use of electronic signatures for signing and initializing any laboratory record including, by not limited to: analytical reports, controlled documents, workflows and purchasing requests.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.

Standard Operating Procedures (SOPs): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Standard Method: a test method issued by an organization generally recognized as competent to do so.

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

Study (or PT Study): This term refers to a Scheduled PT Study or a Supplemental PT Study.

a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants.
b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard, but that does not have a pre-determined opening date and closing date.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Suspension: The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed six (6) months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.

Technology: a specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Tentatively Identified Compound (TIC): A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations. Tentatively Identified Compounds, if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported.

Test Method: An adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 et seq. (1976), the provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture.

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 constants 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.

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e. the air, water and land) upon which human life depends. (US-EPA)

Validation: the confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

NOTE - In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring

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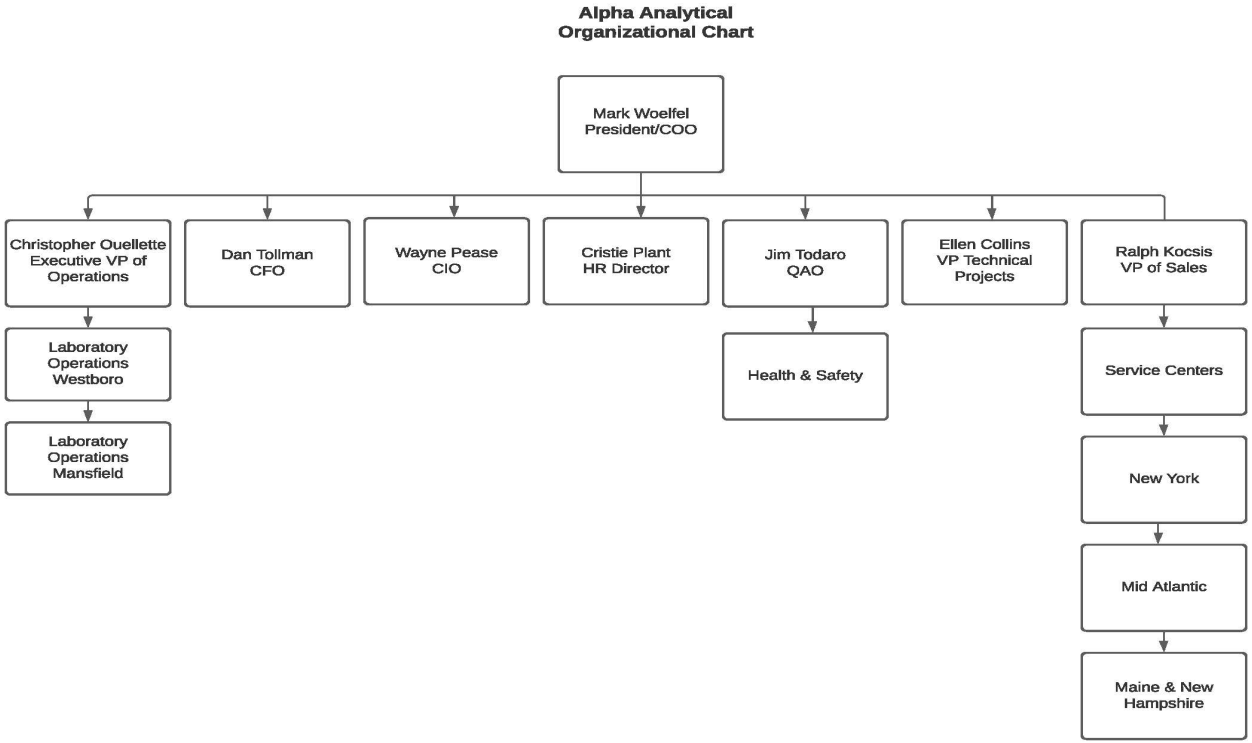
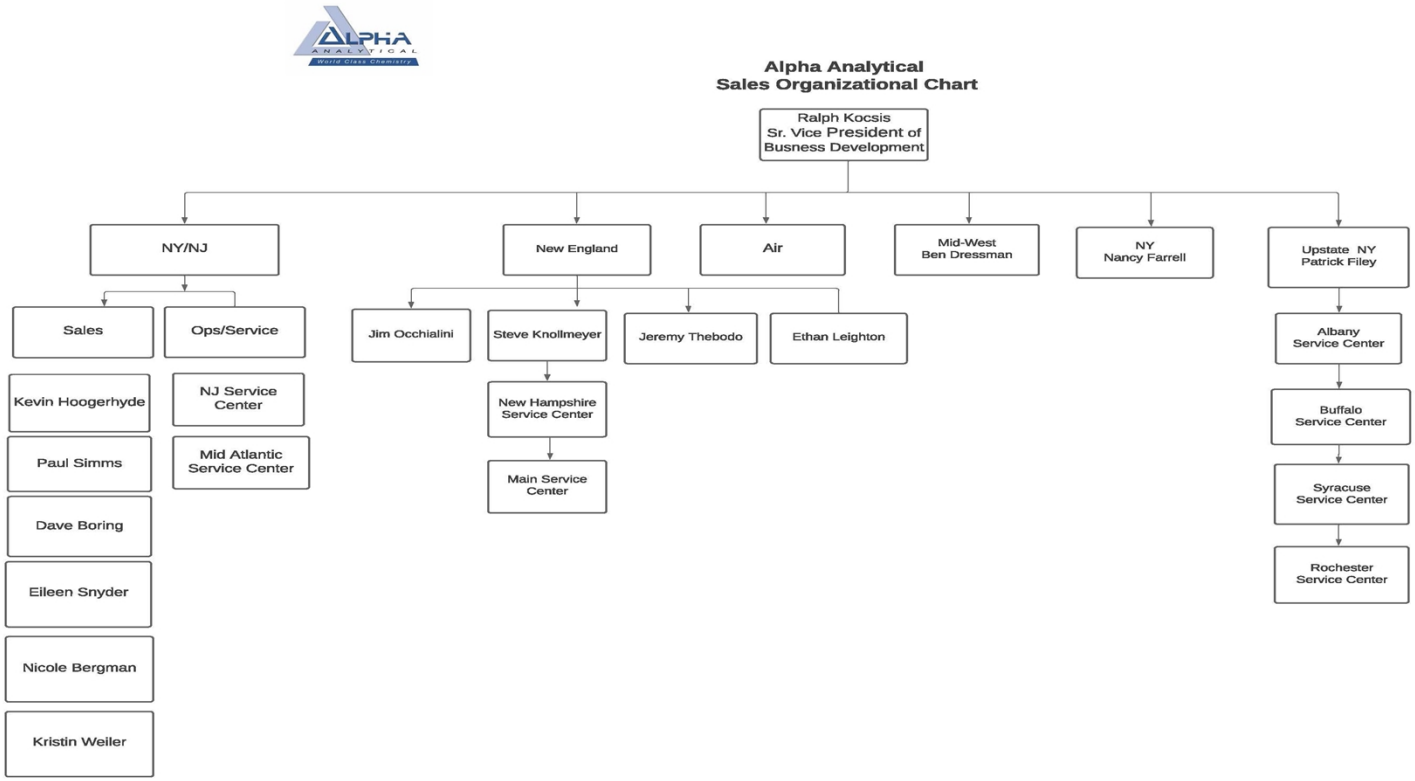
17 Appendix B – Organization Charts

The following charts provide an overview of the organizational structure of Alpha Analytical. The chart also identifies the key personnel responsible for the listed positions. For the various laboratory areas, the individual departmental supervisors are noted. For a listing of all current key personnel, please refer to Section 18, Appendix C.

Updated 8/3/2022

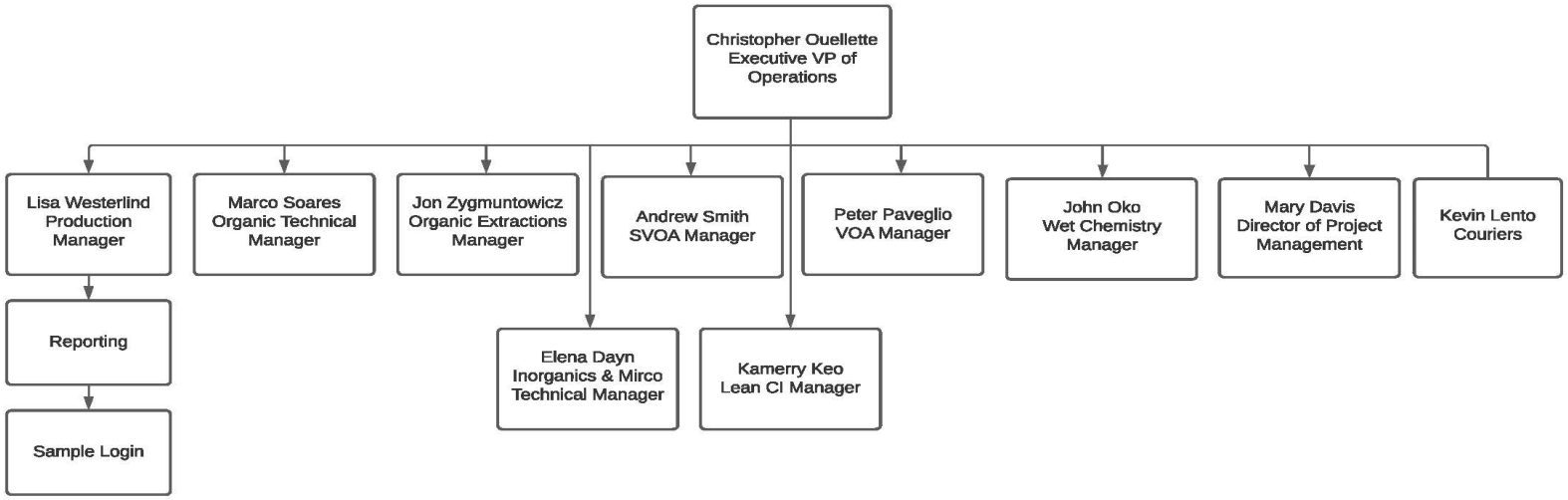


Updated 11/17/2022
Alpha Analytical
Sales Organizational Chart

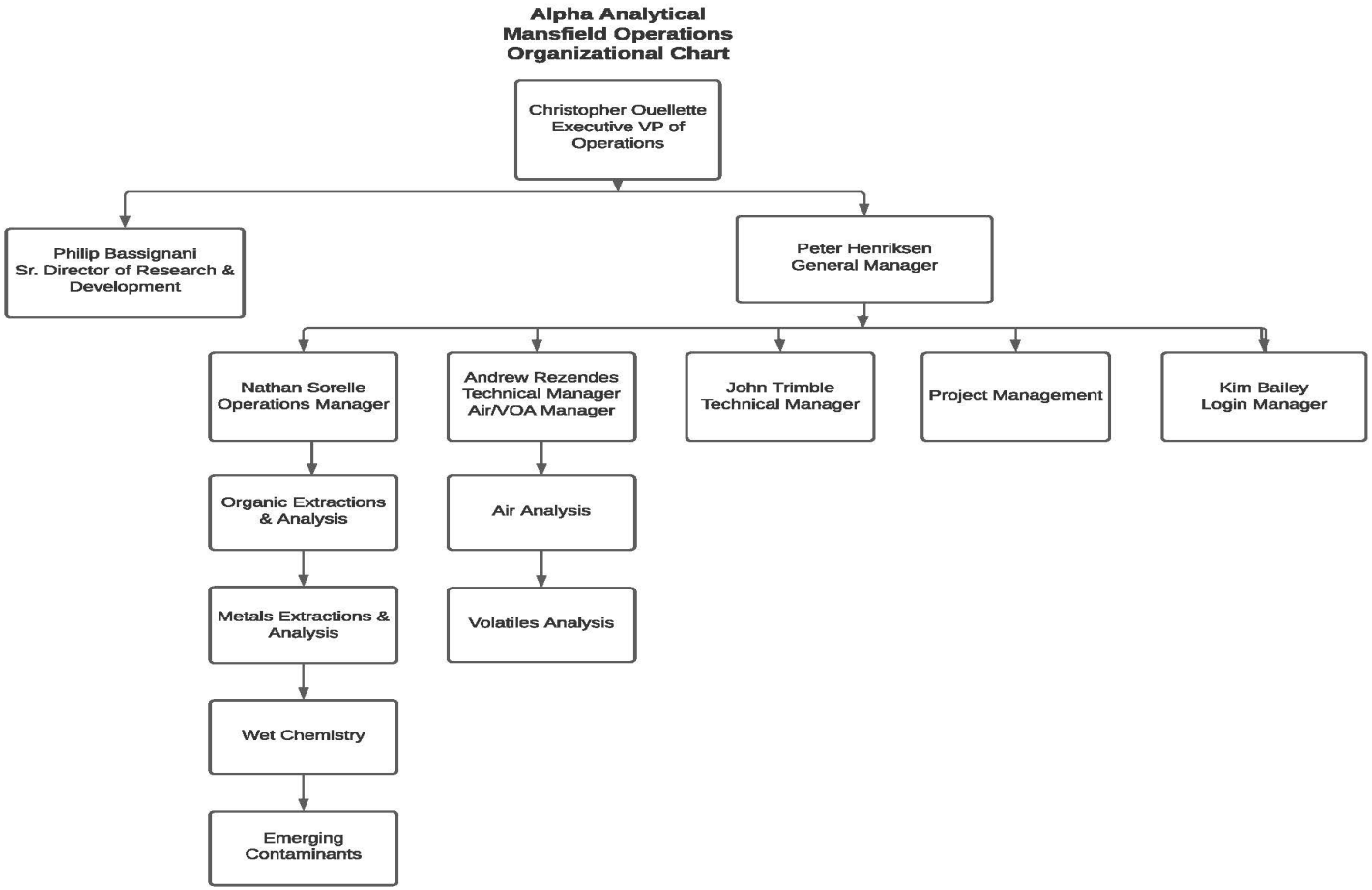


Updated 8/3/2022

Alpha Analytical
Westboro Operations
Organizational Chart



Updated 8/3/2022



18 Appendix C – List of Key Personnel

The following is a listing of all current key personnel. If role is specific to a facility it is denoted by either Westboro or Mansfield following the position title. **Updated 11/2022.**

President / COO: Mark Woelfel

Executive VP of Operations: Christopher Ouellette

CFO: Dan Tollman

CIO: Wayne Pease

Laboratory Technical Manager – Organics Westboro: Marco Soares

Laboratory Technical Manager – Inorganics Westboro: Elena Dayn

Laboratory Technical Manager - Mansfield: John Trimble

Laboratory Technical Manager- Air, Volatiles Manager - Mansfield: Andy Rezendes

Quality Assurance Officer/Health & Safety Manager: James C. Todaro

Senior Director of Research & Development: Philip Bassignani

VP, Technical Projects: Ellen Collins

VP of Sales: Ralph Kocsis

VP, Technical Sales: James Occhialini, Patrick Filey, Kevin Hoogerhyde, Stephen Knollmeyer, Nancy Struzenski

Technical Sales Reps: Paul Simms, David Boring, Jeremy Thebodo, Ben Dressman, Ethan Leighton, Kristin Weiler, Nicole Berman

Reginal Technical Coordinator: Eileen Snyder

General Manager, Mansfield: Peter Henriksen

Director of Project Management: Mary Davis

National Air Account Manager: Andy Rezendes

Information Technology Manager: Glenn Fitzgibbons

Service Delivery Manager: Tammy Winter

Human Resources Director: Cristie Plant

Health & Safety Officer: James Todaro

Operations Manager, Mansfield: Nathan Sorelle

SVOA Manager, Westboro: Andrew Smith

Extractions Manager, Westboro: Jon Zygmuntowicz

VOA Department Manager, Westboro: Peter Paveglio

Wet Chemistry Department Manager, Westboro: John Oko

Metals A2 Manager: Cassandra Daley

Metals Department Manager, Mansfield: Grace Deloughery

Metals Prep Manager: Raldi Cabral

Extractions Manager, Mansfield: Cynthia Pimental

Emerging Containments Prep Manager: Ross Lapenta

Login Manager/ Reporting Manager, Westboro Lisa Westerlind

Quality Systems Specialists: Amy Rice, Rene Bennett, Jason Hebert, Michael Selling, Michael Plante, Joseph Fullen

Purchasing: David Peak

Logistics Manager: Kevin Lento
Equipment Technical Specialists: Patrick Sullivan, Szymon Sus, Kimberly Rivera
Continuous Improvement Leader: Kamerry Keo

19 Appendix D – Preventive Maintenance Procedures

Optimized Service-Calibration Intervals		
Equipment	**Frequency**	**Type of Calibration or Maintenance**
Balances	semiannually daily	cleaning & operations check by service technician (external) calibration verification using Class S-1 certified weights
COD Reactor	annually	reaction temperature verification
Conductivity Bridge	annually annually each use	verification of cell constant complete operations check by service technician (external) calibration verification
DI Water System	as needed monthly annually daily	complete operations check by service technician (external) Residual Chlorine check Biosuitability testing (external) pH and Conductivity check
DO Meter	annually each use	complete operations check by service technician (external) calibration against air as specified by manufacturer
Emergency/Safety Equipment	annually monthly	fire extinguishers and emergency exit lighting check eye washes, showers, fire blanket and first aid kits checked
Freezers	daily	temperature verification
Gas Chromatographs	as needed as needed beginning and end of batch and 10 to 20 samples as per method	injection port preparation; cleaning of detectors initial multi-point calibration continuing calibration verification (CCV) against initial calibration
ICP	Every other day Daily Annually Annually As needed	Change pump tubing Calibration, profile Complete operations check by service technician (external), Linear Dynamic Range determination Clean torch, clean nebulizer, clean spray chamber
Lachat analyzer	Daily As needed	Calibration, clean lines Change tubing, change O-rings
Mass Spectrometers (GC & ICP)	bi-annually as needed 12 hour or daily	change of mechanical pump oil by service technician (external) cleaning of source BFB, DFTPP or ICP-MS tune analysis followed by ICAL or CCV
Mercury Analyzer	monthly each use	clean cell and change pump windings calibration using multi-point curve
Auto-pipettes	Quarterly Daily	verification of accuracy and precision verification of precision for DOD method auto-pipettes
Microwave	Quarterly Annually	power and temperature verification RPM verification
Ovens	Annually	temperature verification
pH Meters	Annually each use	complete operations check by service technician (external) calibration using certified buffers
Refrigerators (General Use)	daily	temperature verification
Refrigerators (Sample Management)	daily	temperature verification
Spectrophotometer	Semi-annually Semi-annually daily	cleaning & operations check by service technician (external) wavelength verification (external) continuing calibration verification (CCV) against initial calibration
TCLP/ZHE Rotator	Quarterly	RPM verification
Thermometers (Mercury/Alcohol) Thermometers (Bimetal/mechanical)	Annually Quarterly	calibration against NIST traceable thermometer (internal) calibration against NIST traceable thermometer (internal)
Thermometers (digital/IR)	Quarterly	calibration against NIST traceable thermometer (external)
Thermometer (NIST Traceable)	Annually	calibration and certification of conformance (external)
Turbidity meter	Annually each use	cleaning & operations check by service technician (external) calibration using formazin
Weights (Class S-1)	Annually Triennially	Working weights verified against reference weights Reference weights calibrated for conformance (external)

20 Appendix E – Alpha Code of Ethics Agreement

Alpha Analytical, Inc.
Ethical Conduct and Data Integrity Agreement

A. ***Personal Pledge:*** I understand that I am charged with meeting the highest degree of ethical standards in performing all of my duties and responsibilities and pledge to only report and/or communicate accurate and precise data and/or information of the highest quality as applicable to my position at Alpha.

B. ***Protocol Pledges:*** I agree to adhere to the following protocols and principles of ethical conduct in fulfilling my work assignments at Alpha:

1. I will perform all tasks for which I am responsible according to Alpha's Quality System Program and/or the applicable approved documentation.

2. I will not intentionally nor improperly manipulate or falsify data in any manner. I will not modify data values unless the modification can be technically justified through a measurable analytical process or method acceptable to Alpha. All such modifications will be clearly and thoroughly documented per Alpha's Quality System Program.

3. I will not intentionally alter dates and times associated with the collection, custody transfer, analysis and/or reporting of sample data. (Specific to Lab Operations).

4. I will not intentionally represent another individual’s work as my own or represent my work as someone else’s.

5. I will be honest and not make false statements to, or seek to otherwise deceive Alpha staff, leaders or clients. I will not improperly report and/or communicate measurements, results, data, test results or conclusions.

C. ***Guardian Pledge:***

1. I will not condone any accidental or intentional reporting of unauthentic data by other Alpha staff and will immediately report such occurrences to my supervisor, the QA Officer, the Laboratory Technical Manager or corporate leadership. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.

2. If a supervisor or other member of the Alpha leadership group requests me to engage in, or perform an activity that I feel is compromising data validity or quality, I have the right to not comply with the request and appeal this action through Alpha’s QA Officer, senior leadership or corporate officers, including the President of the company.

3. I understand that, if my job includes supervisory responsibilities, then I will not instruct, request or direct any subordinate to perform any laboratory practice that is unethical or improper. Also, I will not discourage, intimidate or inhibit a staff member who may choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.

D. ***Agreement Signature:*** I have read and fully understand all provisions of the *Alpha Analytical Ethical Conduct and Data Integrity Agreement*. I further realize and acknowledge my responsibility as an Alpha staff member to follow these standards. I clearly understand that adherence to these standards is a requirement of continued employment at Alpha.

_____
Employee Signature

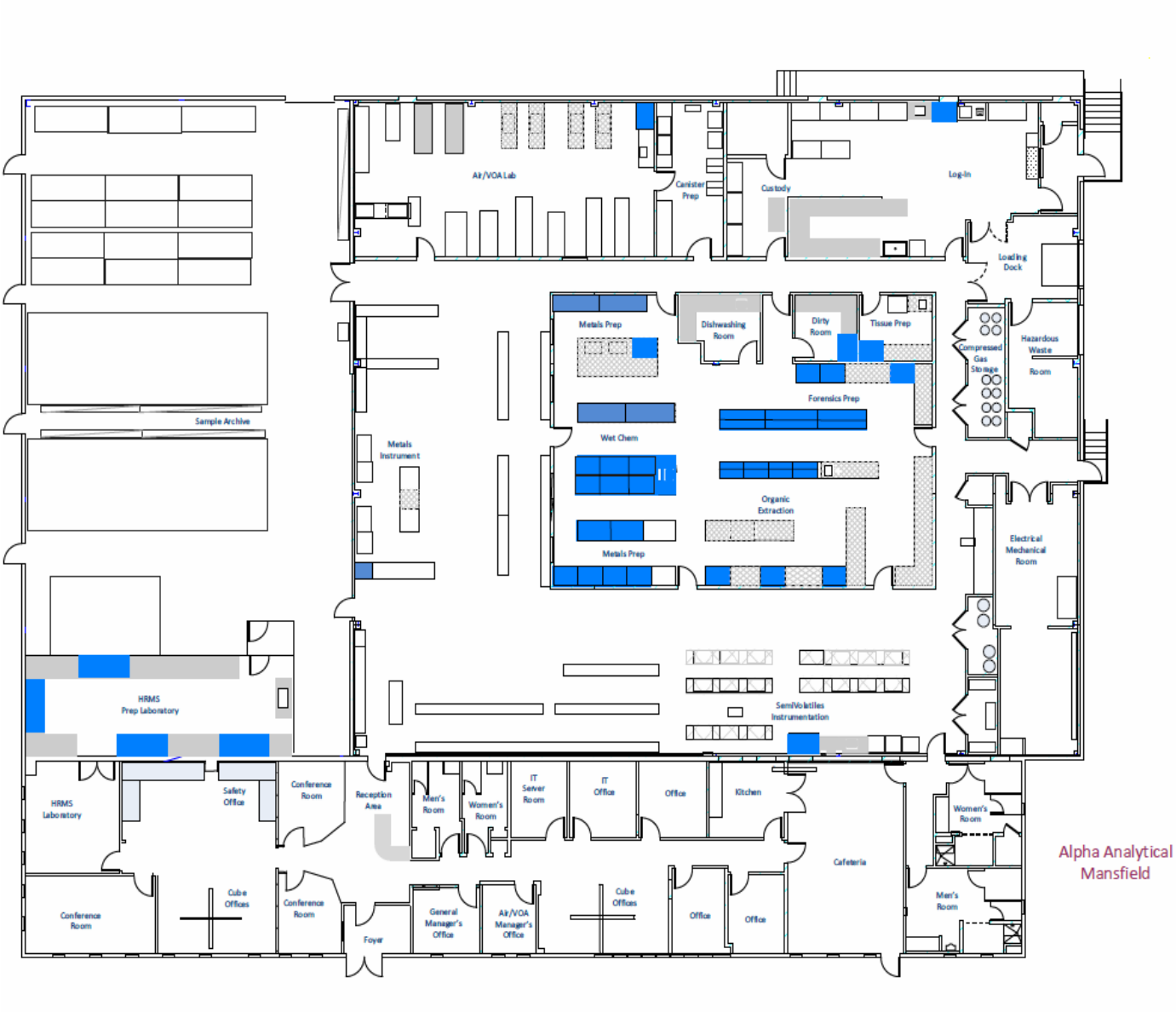
_____
Printed Name

_____
Date

Review Requirements
The *Ethical Conduct and Data Integrity Agreement* must be signed at the time of hire (or within 2 weeks of a staff member's receipt of this policy). Such signature is a condition of continued employment at Alpha. Failure to comply with these requirements will result in immediate discharge from Alpha employment. This agreement is not an employment contract and does not modify in any manner the company's *Employment-at-Will* Agreement.

21 Appendix F– Floor Plan Westboro Facility

22 Appendix G– Floor Plan Mansfield Facility



23 Appendix H – Job Titles and Requirements

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Technical Manager (Director) Organic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 24 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of organic analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Inorganic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of inorganic analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Microbiology Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in the Biological Sciences, including at least one course having microbiology as a major component. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of microbiological analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Quality Assurance Officer	BS/BA in Chemistry, Biology, Environmental or related Science	Two (2) years Environmental Laboratory Experience	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Knowledgeable in Federal, State Programs (THE NELAC INSTITUTE (TNI) STANDARDS, etc.) 3. Able to develop QA/QC policies and certification requirements 4. Able to develop training programs for quality procedures 5. Documented training and/or experience in QA and QA procedures 6. Knowledge of safe laboratory practices and emergency protocols

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Laboratory Coordinator	High School Diploma; Associates or BS/BA in Chemistry, Biology or Environmental or related Science preferred	1 year +	1. Knowledge of safe laboratory practices and emergency protocols 2. Proficient in all methods and SOP's within their department 3. Experience with and understanding of LIMS 4. Proven ability to meet TAT (turnaround times)
Quality Systems Specialist	BS/BA Chemistry, Biology, Environmental or related Science	2 years +	1. General knowledge of laboratory methods 2. Experience with and understanding of LIMS 3. Strong attention to detail 4. Strong oral/written communication and organizational skills 5. Knowledge of QA/QC policies and certification requirements
EH&S Coordinator	High School or Equivalent	2 years +	1. General knowledge of lab operations 2. Detailed knowledge of safe lab practices and emergency protocols 3. Hazardous Waste Management and RCRA Regulation Training 4. DOT Hazardous Materials Regulations Training 5. OSHA Compliance Training 6. Able to develop and deliver new hire and ongoing safety training programs
Lab Technician I	HS or Equivalent	0-1 years. 1+ years preferred.	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician II	HS or Equivalent	2-4 years	1. All skills of Lab Technician I 2. Trained in majority of technician skills relative to department
Lab Technician III	HS or Equivalent	5 years +	1. All skills of Lab Technician II 2. Experienced in training staff
Lab Technician/Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician/Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Chemist I 2. Trained in majority of department methods

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Lab Technician/Chemist III	BS/BA in Chemistry, Biology, Environmental or related Science	5 years +	1. All skills of Chemist II 2. Experienced in training staff
Analyst I	HS or Equivalent	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analyst II	HS or Equivalent	2-4 years	1. All skills of Analyst I 2. Experienced in machine operation, maintenance and troubleshooting
Analyst III	HS or Equivalent	5 years +	1. All skills of Analyst II 2. Experienced in data review and reporting 3. Experienced in training staff
Analytical Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	6 mos-1 year	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analytical Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Analytical Chemist I 2. Experienced in machine operation, maintenance and troubleshooting
Analytical Chemist III	BS/BA in Chemistry, Biology, or Environmental or related Science	5 years +	1. All skills of Analytical Chemist II 2. Experienced in data review and reporting 3. Experienced in training staff
Data Deliverable Specialist I	HS Diploma, BS/BA or Associates preferred	0-1 years	1. Introductory knowledge of laboratory methods 2.Able to follow direction and Standard Operating Procedures (SOP's) 3. Working knowledge of Adobe Acrobat, Microsoft Word, Excel 4. Good writing and typing skills
Data Deliverable Specialist II	HS Diploma, BS/BA or Associates preferred	2-4 years	1. All skills of Data Deliverable Specialist I 2. General knowledge of laboratory methods 3. Understanding of data review/ data reporting process 4. Experience with and understanding of LIMS and electronic data deliverables

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Data Deliverable Specialist III	HS Diploma, BS/BA or Associates preferred	5 years +	1. All skills of Data Deliverable Specialist II 2. Intermediate/advanced knowledge of laboratory methods 3. Able to perform report review 4. Experience with and understanding of LIMS and electronic data deliverables 5. Able to initiate re-work where necessary
Laboratory Intern	2 Semesters of Chemistry, Biology or Environmental Science	None; Lab work study experience preferred	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures

KEY

* Internal terms only. Full title would have "Environmental Laboratory" and specific department preceding it.

** Substitutions: Equivalent knowledge may be substituted for a degree in some instances.

*** Not meant to be an exhaustive list of skill requirements. For full list of skills consult the "Laboratory Skills" list. Actual Job Duties and Responsibilities can be found within job descriptions for each position.

24 Appendix I – Standard Operating Procedures

WESTBORO SOP #	Title
1728	Waste Management and Disposal - Westborough
1730	Balance Calibration Check
1733	Thermometer Calibration
1737	Inorganics Glassware Cleaning and Handling
1738	Water Quality Monitoring
1745	Reagent, Solvent and Standard Control
1948	Separatory Funnel Liquid-Liquid Extraction – EPA 3510C
1953	Organic Extraction Glassware Cleaning & Handling
1954	Soxhlet Extraction – EPA 3540C
1955	Sulfur Cleanup – EPA 3660A
1956	Oil and Waste Dilution – EPA 3580A
1959	Microwave Extraction – EPA 3546
1960	Sulfuric Acid Cleanup – EPA 3665A
1962	Florisil Cleanup
1963	Fractionation Cleanup
1964	Preparation of Samples for Chlorinated Herbicides
2107	Volatile Organic Compounds – EPA 524.2
2108	Volatile Organic Compounds – EPA 8260C
2109	Polynuclear Aromatic Hydrocarbons (PAHs) by SIM – EPA 8270D (modified)
2111	Semivolatile Organics by GC/MS – EPA 8270D
2112	TCLP/SPLP Extraction - Volatile Organics SW-846 Method 1311/1312
2113	EDB, DBCP & TCP in Water by Microextraction & Gas Chromatography – EPA 504.1, 8011
2116	Organochlorine Pesticides by Capillary Column GC – EPA 8081B
2119	Extractable Petroleum Hydrocarbons – MADEP
2120	Volatile Petroleum Hydrocarbons – MADEP
2123	Polychlorinated Biphenyls in Oil – EPA 600/4-81-045
2125	TPH-Diesel Range Organics, Maine 4.1.25, EPA 8015C (Modified)
2127	CT-ETPH
2128	Herbicides by 8151A
2129	PCBs by Capillary Column Gas Chromatography - EPA 8082A
2131	New Jersey EPH Method
2133	TCLP Extraction Metals and Semi-Volatile Organics – SW-846 Method 1311
2135	SPLP Extraction Inorganics and Semivolatile Organics, EPA 1312

WESTBORO SOP #	Title
2161	Fecal Coliform by Membrane Filtration – SM 9222D
2163	Fecal Coliform by Multiple Tube Fermentation – SM 9221E
2191	Heterotrophic Plate Count – SM 9215B
2192	Total Coliform/E.Coli – Presence/Absence (Colilert) – SM 9223B
2194	Total Coliform by Multiple Tube Fermentation – SM 9221B
2195	Chlorophyll A – SM 10200H
2196	E. Coli – Membrane Filtration
2197	Chlorophyll A – EPA 446
2198	Air Density Monitoring
2199	Inhibitory Residue Test
2200	Enterococcus – MF
2201	Total Coliform, E.Coli & Enterococcus by Quantification Methods (Quanti Tray)
2202	pH, Liquid Samples
2203	pH, Soil & Waste Samples
2204	Hexavalent Chromium
2205	Biological Oxygen Demand
2206	Ammonia Nitrogen
2207	Total Kjeldahl Nitrogen
2208	Chemical Oxygen Demand
2209	Oil & Grease by n-Hexane Extraction Method & Gravimetry
2210	Cyanide, Total
2211	Phenol, Total
2212	Sulfate, Turbidimetric Method
2213	Alkalinity, Titration Method –SM 2320B
2214	Determination of Inorganic Anions by Ion Chromatography – EPA 300.0
2215	Total Organic Carbon/Dissolved Organic Carbon
2216	Chloride – SM 4500Cl-E, EPA 9251
2217	Nitrate, Nitrite and Nitrate/Nitrite Nitrogen – EPA 353.2, SM 4500NO_3-F
2218	Total Solids (Dried @ 103-105^o) and TVS – SM 2540B, SM 2540E
2219	Total Dissolved Solids – SM 2540C
2220	Total Suspended Solids – SM 2540D
2221	Total Sulfide – SM 4500S2-AD, EPA 9030B
2222	MBAS, Anionic Surfactants – SM 5540C
2223	Fluoride, Electrode Method – SM 4500F-BC
2224	Turbidity, Nephelometric Method – EPA 180.1, SM 2130B
2225	Orthophosphate, Colorimetric Single Reagent Method – SM 4500P-E
2226	Total Phosphorous, Colorimetric Combined Reagent Method – SM 4500P-E
2227	Flashpoint – EPA 1010

WESTBORO SOP #	Title
2228	Reactivity – EPA Chapter 7.3
2229	Total Solids (Dried @ 103-105°) – SM 2540G
2230	Specific Conductance and Salinity
2231	True and Apparent Color, Visual Comparison Method
2232	Acidity, Titration Method
2233	Determination of Formaldehyde by HPLC, EPA 8315A
2234	Sulfite, Iodometric
2235	Ferrous Iron
2236	Residual Chlorine
2237	ORP
2238	Ignitability of Solids EPA 1030
2239	Physiologically Available Cyanide (PAC)
2240	Total Settleable Solids SM 2540 F
2241	Fixed and Volatile Solids in Solid and Semisolid Samples – SM 2540G
2242	Tannin & Lignin
2243	Nitrite - Manual Colorimetric Method
2244	Paint Filter Liquids Test
2245	Odor, Threshold Odor Test
2249	Dissolved Oxygen
2251	Perchlorate by IC/MS/MS
3743	Free Cyanide
9177	Total Phenol - SEAL Method
9733	Oil & Grease and TPH in Soil
10807	Percent Organic Matter in Soil
14751	Determination of UV-Absorbing Organic Constituents at 254nm
17972	Extractable Organic Halides (EOX)
18236	Chloropicrin and Carbon Tetrachloride by EPA 8011
19332	DI Water Extraction ASTM D3987
21994	Nonfractionated EPH
23148	Gilson EPH Fractionation
25691	Semivolatile Organic Compounds by GC/MS EPA 625.1
25693	Volatile Organic Compounds by EPA 624.1
26801	TPH - Gasoline Range Organics Maine 4.2.17, EPA 8015D
27634	True and Apparent Color, Single Wavelength Method
28200	PCBs by EPA 608.3
28201	Pesticides by EPA 608.3
32637	Polynuclear Aromatic Hydrocarbons (PAHs) by SIM EPA 8270E (M)
32639	Volatile Organic Compounds EPA 8260D
33262	Extractable Petroleum Hydrocarbons (MA-EPH) 2.1

MANSFIELD SOP #	Title
1753	Glassware Cleaning
1754	Balance Calibration
1755	Pipette Checks
1797	Waste Management and Disposal - Mansfield
1816	Reagent Solvent Standard Control
2134	Hot Block Digestion for Aqueous Samples EPA 3005A
2138	Mercury Aqueous 7470A
2139	Mercury Soil 7471B
2140	AVS SEM
2141	Hydride Generation
2142	Mercury Aqueous 7474
2143	Mercury Soil 7474
2148	Metals Soil Digestion 3050
2150	Metals Microwave 3015
2155	EPA 8270D
2157	PAH by SIM
2158	EPA 8081B
2160	EPA 8082A Aroclors/Congeners by GC and TO-10A
2162	Pesticides/PCB Aroclors/Congeners by GC/MS SIM
2164	1,4-Dioxane GC/MS SIM
2165	Separatory Funnel Extraction EPA 3510C
2166	Tissue Prep
2167	GPC
2168	Sulfur Cleanup 3660
2169	Sulfuric Acid Cleanup 3665
2170	Silica Gel Cleanup
2171	% Lipids
2172	Microscale Solvent Extraction EPA 3570
2173	Soxhlet Extraction EPA 3540C
2174	Soxhlet Extraction of PUFs
2175	% Total Solids
2182	TOC by Lloyd Kahn
2183	Particle Size Determination
2184	Particulates in Air PM-10
2186	EPA TO-15
2187	APH
2188	Air PIANO
2189	Dissolved Gases RSK-175

MANSFIELD SOP #	Title
2190	Cleaning & Preparation of Air Sampling Equipment
2246	TPH and SHC
2247	Alkylated PAH
2248	Organic Lead
2252	Fixed Gases
2255	PIANO Volatiles
2256	Ethanol in Oil
2257	Whole Oil Analysis
2259	Density Determination of Oils
2260	Alumina Cleanup
2261	Shaker Table
2263	Gravimetric Determination
2264	Tissue Extraction
2265	Organic Waste Dilution
2267	Client SOP: SGC - Manual Method
2268	Client SOP: DCM Extractable Method
4246	PAHs by SPME
6438	Mercury in Sorbent Tubes by CVAA
7900	Mercury 1631E Using Cetac-M-8000 Analyzer
9077	Porewater Generation
9480	EPA-TO-12
12863	EPA 8270D GC/MS Full Scan TO-13A
13091	HPAH
13406	Particulate Organic Carbon
14500	Lead in Particulate Matter
17452	TOC by EPA 9060A
17456	Moisture, Ash and Organic Matter
17829	Specific Gravity of Soil
17830	Liquid Limit, Plastic Limit and Plasticity Index of Soils
17940	1,4-Dioxane in Drinking Water by EPA 522
18086	Total Suspended Solids (TSS) SM 2540D
18705	PCB Congeners by GC/MS-SIM EPA 8270D
18710	Trace Elements in Waters and Wastes by ICP-MS EPA 200.8
18711	Metals by ICP EPA 200.7
18715	Mercury in Water (CVAA) EPA 245.1
18716	Hot Block Digestion for Aqueous Samples EPA 3005A
18717	Microwave Assisted Acid Digestion of TCLP Extracts EPA 3015
18718	Microwave Assisted Acid Digestion for Metals EPA 3015A/3051A
18817	Alcohols by FID- Aqueous Direct Injection EPA 8015D

MANSFIELD SOP #	Title
19625	Glycols by GC-FID EPA 8015D
19971	Air Drying Samples for PCBs and Metals Analysis
19978	Density of Soil
22132	Data Review – Ohio VAP
23511	PFAS by LC/MS/MS by EPA 537
23528	PFAS by LC/MS/MS Isotope Dilution by EPA 537(M)
24454	Acetonitrile Extraction for Unknown Compounds via GCFID
25896	EPA 8290A Dioxins and Furans by Hi-Res MS
25900	EPA 1613B Dioxins and Furans by Hi-Res MS
25923	Mercury in Liquid Waste (Automated Cold-Vapor Technique) EPA 7470A
25924	Mercury in Solid/Semisolid Waste (Manual Cold-Vapor Technique) EPA 7471B
26796	Metals by ICP EPA 6010D
26797	Metals by ICP-MS EPA 6020B
27056	HiRes Laboratory Glassware Cleaning
27322	In Vitro Accessibility Assay for Lead in Soil EPA 1340
27360	PFAS in Cranberry Matrix by EPA 537 (M) LC/MS/MS Isotope Dilution
27485	Total Petroleum Hydrocarbons Screen by GC/FID 8015D
27897	PCB Congeners by High Resolution GC/MS
29033	PFAS by LC/MS/MS in Non-Potable Water
29139	Biomimetic Extraction Using SPME
32082	MADEP PFAS by SPE & LC/MS/MS Isotope
31164	Semivolatile Organic Compounds By Gas Chromatography / Mass Spectrometry (GC/MS) 8270E
32324	PAH and PCB Congeners by GCMS with SIM 8270E TO-13A
32200	EPA 533 PFAS LC/MS/MS Isotope Dilution
36216	PFAS LC/MS/MS Isotope Dilution Nonpotable Water
36957	PFAS by EPA 537.1 in Drinking water by LC/MS/MS
45852	Method 1633 Draft PFAS in Aqueous, Solid, Biosolids and Tissue by LCMSMS
40380	Resin Extraction

CORPORATE SOP #	Title
1559	Sample Receipt and Login
1560	Sample Custody and Tracking
1561	Bottle Order Preparation
1562	Computer System Backup/Control
1563	Computer and Network Security
1564	Software Validation and Control

CORPORATE SOP #	Title
1565	Training Program
1566	Report Generation and Approval
1567	Organics Data Deliverable Package Review
1722	Customer Inquiry and Complaint Procedures
1723	Project Management
1724	Quote/Contract Procedure
1725	Project Communication Form Generation
1726	Purchasing Procedure
1727	Accounts Payable Invoice Processing
1729	Document Control
1731	Manual Integration and Compound Rejection
1732	DL LOD LOQ Generation
1734	Control Limit Generation
1735	Analytical Guidelines for Method Validation
1736	Corrective and Preventative Actions
1738	Water Quality Monitoring
1739	Demonstration of Capability (DOC) Generation
1740	Internal Audit Procedure
1741	Data Review – Organics
1742	Calculating Measurement Uncertainty
1743	Annual Management Review
1744	Sample Compositing Procedure
1746	Nonconformance Planning/Procedures
1747	Temperature Datalogger Operation
2274	Data Validation Package
17553	Lab Supply Transfer Procedure
18821	Weights Verification
18909	PT Corrective and Preventive Action Process

25 Appendix J– Report Signing - List of Authorized Personnel

All final reports are reviewed and signed by authorized personnel, who have been designated to perform such review.

The following is the listing of all authorized representatives of the company who have been authorized by the Laboratory Technical Manager to perform final report review as of 05/25/2022. Refer to Qualtrax Form ID 17878 for the most current listing.

Name	Title
Peter Henriksen	General Manager
Susan O'Neil	Project Manager
Christopher Anderson	Project Manager
Elizabeth Porta	Project Manager
Andrew Rezendes	Volatiles Manager and Air Technical Manager
Lisa S. Westerlind	Technical Representative
Ellen M. Collins	Technical Representative
Michelle M. Morris	Technical Representative
Kelly Stenstrom	Technical Representative
Cristin Walker	Technical Representative
Cynthia McQueen	Technical Representative
Melissa Sturgis	Technical Representative
Caitlin Walukevich	Technical Representative
Tiffani Morrissey	Technical Representative
Jennifer Clements	Technical Representative
Kelly O'Neill	Technical Representative
Alycia Mogayzel	Technical Representative
Sebastian Corbin	Technical Representative
Jennifer Jerome	Technical Representative
Steven Gniadek	Technical Representative
Michael Chang	Technical Representative
James C. Todaro	Quality Assurance Officer

Analysis of Polynuclear Aromatic Hydrocarbons and PCB Congeners by Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring

References: Method EPA 8270E, SW-846,Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision VI (Phase II), June 2018.

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-13A – Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using GC/MS-SIM, January 1999.

USEPA, Superfund Analytical Services/Contract Laboratory Program (CLP), Multi-Media, Multi-Concentration Organics Analysis, SOM01.1, Exhibit D - Analytical Methods, "Analytical Method for the Analysis of Semivolatile Organic Compounds," May, 2005.

1. Scope and Application

Matrices: Solid, waste, soil, sediment, tissue, oil, water and Air PUF.

Definitions: Refer to Alpha Analytical Quality Manual.

This standard operating procedure (SOP) is applicable to the quantification of polynuclear aromatic hydrocarbons (PAHs) and PCB Congeners by gas chromatography/mass spectrometry with selected ion monitoring (GC/MS-SIM). Method is modified from USEPA method 8270D for the application of SIM following the SW846 sited CLP SOW reference.

The sample preparation methods described herein are: 3510C (water), 3540 (Air PUF), 3580 (oil) and 3570 (solid/waste, soil, sediment and tissue) from the above reference.

The individual target compounds are found in Table B.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS instrumentation and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Aqueous samples are serially extracted with methylene chloride in a 2 liter separatory funnel at the required pH. Soil, sediment, solid/waste and tissue samples are extracted by micro scale solvent extraction (MSE) 3570 modified. Oil samples are diluted with methylene chloride (1mL sample to 9mL of solvent) following method 3580. Air PUF samples are extracted via soxhlet method 3540 in hexane/ether (90/10). The extracts are concentrated to the required final volume and spiked with internal standards in preparation for analysis. All extraction procedures are documented in the associated sample preparation SOPs. The extracts are analyzed by GC/MS-SIM. The target analytes are resolved on the GC column and detected using a mass selective detector (MSD). Concentrations are determined using mean relative response factors from a multi-level calibration

curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

2.1 Method Modifications from Reference

Section 7.5.5 of EPA SW846 8270C is referred to for the application of SIM, along with EPA SOM01.1, Exhibit D for Semivolatiles. This method has been modified to include a sub list of PCB Congeners that are co-analyzed along with the PAHs. These PCB Congeners are commonly referred to the NOAA 18, or NOAA 22 Congener List. If analyzing for the PCB Congeners in conjunction with the PAHs, the Mass Spectrometer DFTPP tuning criteria for Maximum Sensitivity described in Section 10.1.4.4 must be utilized.

3. Reporting Limits

Reporting limits for individual PAH compounds can range from 10ng/L to 10,000ng/L for water samples, and a range of 8μg/Kg to 8000μg/Kg for soil/sediment, solid/waste and tissue samples. Oil samples can range from 100ug/Kg to 100,000ug/Kg. Air PUF samples can range from 200ng/PUF to 40,000ng/PUF. PCB Congener compounds can range from 1.0ng/L to 500ng/L for water samples and can range from 0.133ug/Kg to 66.7ug/Kg for soil/sediment, solid/waste and tissue samples. Oil samples can range from 5ug/Kg to 5000ug/Kg. Air PUF samples are not analyzed for PCB Congeners following this SOP. Specific reporting limits for each matrix per analyte can be viewed within the LIMS under Applications/Product Catalog.

Project-specific reporting limits may be determined based on the sample size extracted, the extract final volume and the lowest concentration standard included in the initial calibration.

4. Interferences

Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction or analysis.

When emulsions are formed during the separatory funnel extraction process, centrifuging the extract may be necessary to break down the emulsion before continuing the serial extractions. If water does filter into the collected extract, the extract should be re-filtered through sodium sulfate before the extract is concentrated to its final volume.

Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

The following cleanup methods can be used to help minimize co-extracted matrix/interferences: Method 3610 Alumina Cleanup, Method 3630 Silica Cleanup, Method 3660 Sulfur Removal using Copper and Method 3640 GPC Cleanup.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Water samples are collected in pre-cleaned 1-L amber glass bottle with a Teflon lined screw cap. Approximately 1 L of sample is required for the analysis. Soil/Sediment/Solid/Waste/Oil samples are collected in a pre-cleaned 250 mL glass jar with Teflon lined screw cap. Approximately 100g of soil/sediment is required for the analysis although more may be collected to provide a representative sample. Solid/Waste and Oil samples require half this amount, approximately 50g. Tissue samples are typically received from the field wrapped in foil and inside Ziploc baggies. As much tissue sample as possible should be provided. Air PUF samples are collected per the sited TO-13A reference method.

6.2 Sample Preservation

Water, soil/sediment, solid, waste, oil, Air PUF and tissue samples are preserved by cooling to 4°C $\pm$ 2°C and should remain at this temperature until extraction. No chemical preservative is required, and may interfere with the analysis. Sediment and Tissue samples may also be frozen to -20°C $\pm$ 2°C per client specific instructions.

6.3 Sample Shipping

No special sample shipping requirements.

6.4 Sample Handling

Water and Air PUF samples are extracted within seven days of collection. Soil, sediment, solid/waste, oil and tissue samples are extracted within 14 days of collection. Sediment and tissue samples may be frozen to extend holding times per client request. All sample extracts are analyzed within 40 days of sample preparation.

7. Equipment and Supplies

7.1 Gas chromatograph/Mass Spectrometer and Autosampler: Programmable; heating range from 40°C to 350°C; split/splitless-type inlet system; (Agilent 6890 or similar); mass selective detector (Agilent 5973, or similar); automatic injector (Agilent 7683B, or similar)..

7.2 Chromatography Column: Fused silica, capillary column, 0.25 mm ID x 30m length, 0.25µm film thickness (ZB-5, Phenomenex, 5% Phenyl-95% dimethypolysiloxane, Restek RTX5 30m x 0.25mm ID x 0.25um film thickness, or equivalent).

7.3 Data Acquisition System: Computerized system for collecting, storing, and processing detector output (Agilent Enviroquant target software or equivalent

7.4 Gases: BIP Ultra high purity helium (99.9995%), Compressed Nitrogen for N-Evap.

7.5 Hamilton Gas tight Syringes – 10uL to 1.0mL.

7.6 Vials including 2mL, 4mL, 10mL, and 40mL.

8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

8.1 Reagents: All solvent expirations determined as indicated by manufacturer guidelines.

8.1.1 Methylene Chloride: ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Standard Control* (G-008) for additional details regarding solvent purity. Used to extract samples and prepare instrument/analytical standards

8.1.2 Acetone: ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Standard Control* (G-008) for additional details regarding solvent purity. This water soluble solvent is used for surrogate and LCS/MS preparation.

8.1.3 Methanol: ACS approved, Pesticide grade, *Reagent, Solvent and Standard Control SOP* (G-008) for additional details regarding solvent purity.

8.1.4 Hexane: ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Solvent Control* (1816) for additional details regarding solvent purity. This solvent is used in the extraction of Air PUF samples.

8.1.5 Ether: see SOP *Reagent, Solvent and Solvent Control* (1816) for additional details regarding solvent purity. This solvent is used in the extraction of Air PUF samples.

8.2 Analytical Standards

Standards should be stored at -10 or less, away from light when not in use. They should be discarded after 1 year unless the vendor expiration date states otherwise or if degradation is observed. Stock standards are given a 1 year expiration from the preparation date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given a 6 month expiration from the preparation date or the expiration of the primary solution whichever occurs first. All analytical standards are made up in Methylene Chloride. All prep standards are made up in Acetone.

8.3 Primary Standards

8.3.1 PAH Custom Mix – Supelco 21353385 (100ug/mL)

8.3.2 Custom PCB Standard – Accustandard S-7911-2X (NOAA22 Mix 100ug/mL)

8.3.3 3,3',4,4'-Tertachlorobiphenyl – Accustandard C-077S-TP (BZ#77 100ug/mL)

8.3.4 2,3,3',4,6-Pentachlorobiphenyl – Accustandard C-110S-TP (BZ#110 100ug/mL)

8.3.5 3,3',4,4',5-Pentachlorobiphenyl – Accustandard C-126S-TP (BZ#126 100ug/mL)

8.3.6 4,4'-Dibromooctafluorobiphenyl – Ultra PPS-172 (DBOB 5000ug/mL)

8.3.7 2,2',3,3',4,5,5',6-Octachlorobiphenyl – Ultra RPC-075S (BZ#198 100ug/mL)

8.3.8 Custom Deuterated PAH Standard – Restek 563160 (2000ug/mL)

8.3.9 PAH Two Component Mixture – Cambridge Isotope Laboratory ES-5498 (2000ug/mL)

8.3.10 EPA PCB Congener Calibration Check solution – Ultra Scientific RPC-EPA2 (100ug/mL)

8.3.11 **Custom PAH Standard** - Restek 567608 (1000ug/mL)

8.3.12 **MA EPH Aromatic Hydrocarbon Standard** - Restek 31458 (1000ug/mL)

8.3.13 2**,2',3,4,4',5,5'-Heptachlorobiphenyl** – Cambridge Isotope Laboratory EC-1407-3 (BZ#180 13C12 40ug/mL)

8.3.14 **4,4'-Dichlorobiphenyl** – Cambridge Isotope Laboratory EC-1407-3 (BZ#15 13C12 40ug/mL)

8.3.15 **Semi-Volatile GCMS Tuning Standard** – Ultra GCM-150 (1000ug/mL)

8.3.16 **SV Internal Standard Mix** – Restek 31206 (2mg/mL)

8.4 Stock Solutions

8.4.1 **NOAA22 + BZ#198 Stock** – Prepared by Diluting 1.25mL of **Custom PCB Standard** (Accustandard S-7911-2X 100ug/mL), 1.25ml of **2,2',3,3',4,5,5',6-Octachlorobiphenyl** (Ultra RPC-075S BZ#198 100ug/mL), and 25ul of **4,4'-Dibromooctafluorobiphenyl** (Ultra PPS-172 DBOB 5000ug/mL) to 25mL in Hexane. Final concentration 5000ng/mL.

8.4.2 **BZ77/110/126 Stock** – Prepared by diluting 0.5ml of each **3,3',4,4'-Tertachlorobiphenyl** (Accustandard C-077S-TP BZ#77 100ug/mL), **2,3,3',4,6-Pentachlorobiphenyl** (Accustandard C-110S-TP BZ#110 100ug/mL), and **3,3',4,4',5-Pentachlorobiphenyl** (Accustandard C-126S-TP BZ#126 100ug/mL) to 10ml in hexane. Final concentration 5000ng/mL.

8.4.3 **BZ#198 and DBOB Stock** - Prepared by diluting 1.25ml of **2,2',3,3',4,5,5',6-Octachlorobiphenyl** (Ultra RPC-075S BZ#198 100ug/mL), and 25ul of **4,4'-Dibromooctafluorobiphenyl** (Ultra PPS-172 DBOB 5000ug/mL) to 25mL in Hexane. Final concentration 5000ng/mL.

8.4.4 **PCB Congener ICV Stock –** Prepared by diluting 0.020mL of **EPA PCB Congener Calibration Check solution** (Ultra Scientific RPC-EPA2 100ug/mL) to a final volume of 10mL in methylene chloride. Final concentration 200ng/mL.

8.4.5 **PAH ICV Stock –** Prepared by diluting 0.1 mL of **Custom PAH Standard** (Restek 567608 1000ug/mL), and 0.1mL of **MA EPH Aromatic Hydrocarbon Standard** (Restek 31458 1000ug/mL), to a final volume of 10mL in Methylene chloride. Final concentration 10ug/mL.

8.5 Spiking Solutions

8.5.1 **PAH Extraction Surrogate** – Prepared by diluting 1.25mL of **Custom Deuterated PAH Standard** (Restek 563160 2000ug/mL) to 500ml in Methylene Chloride. Final Concentration 5.0ug/ml.

8.5.2 **PAH Field Surrogate** – Prepared by diluting 1.0mL of **PAH Two Component Mixture** (C.I.L. ES-5498 2000ug/ml) to 10ml in Methylene Chloride. Final concentration 200ug/ml. 20ul of this resulting solution is spiked into all PUF samples prior to shipment to the client.

8.5.3 **PAH Laboratory Control Standard** – Prepared by diluting 0.5mL of **Custom PAH Standard** (Restek 567608 1000ug/mL), and 0.5ml of **MA EPH Aromatic Hydrocarbon Standard** (Restek 31458 100ug/mL) to 100mL in Acetone. Final concentration 100ug/mL.

8.5.4 Pest/Cong Surrogate Spike – Prepared by diluting 2.0mL of **BZ#198 and DBOB Stock** to 100ml in Acetone. Final concentration 100ug/mL.

8.5.5 PCB Laboratory Control Standard – Prepared by diluting 0.1mL of **Custom PCB Standard** (NOAA22 Mix, Accustandard S-7911-2X 100ug/mL), and 10mL of **Pesticide LCS Solution** (1000ng/mL) to a final volume of 100mL in Acetone. Final concentration 100ng/mL). Information for the formulation of the Pesticide LCS Solution can be found in SOP (2158_ Determination of Organochlorine Pesticides by Gas Chromatography/Electron Capture Detection. Pesticides are not reported by the method outlined in this SOP but combined extraction is sometimes performed.

8.5.6 PAH SIM Internal Standard - Prepared by diluting 0.125mL of **SV Internal Standard Mix** (Restek 31206 2000ug/ml) to 10ml in Methylene Chloride. Final Concentration 25ug/ml. 20ul of this solution is spiked into every 1ml of sample extract prior to instrument analysis. This solution can also be prepared from a serial dilution of the **8270 Internal Standard Mixture** (250ug/mL). Information for the preparation of the 8270 Internal Standard Mixture can be found in SOP (2155) Semivolatile Organic Compounds by Gas Chromatography/Mass Spectroscopy.

8.5.7 Internal Standard Primary Standard for PCB Congeners – Prepared by diluting 2.5ml of 2**,2',3,4,4',5,5'-Heptachlorobiphenyl** (Cambridge Isotope Laboratory EC-1407-3 BZ#180 13C12 40ug/mL), and **4,4'-Dichlorobiphenyl** (Cambridge Isotope Laboratory EC-1407-3 BZ#15 13C12 40ug/mL) to 10ml in Hexane. Final concentration 10ug/mL. 20ul of this solution is spiked into every 1ml of sample extract prior to instrument analysis.

8.6 Calibration Solutions

8.6.1 PAH/PCB Combo ICAL /Stock Solution – Prepared by following the recipe outlined in the following table, bringing the components to either a final volume of 1mL or 10mL in methylene chloride. The final concentration is 5,000ng/ml for PAHs and 500ng/mL for PCB congeners.

Standard/Solution	Volume Added
PAH Custom Mix (Supelco 21383385 100ug/ml	1.0mL
Custom Deuterated PAH Standard (Restek 563160 2000ug/mL)	50uL
PAH Two Component Mixture (C.I.L. ES-5498 2000ug/ml	50uL
NOAA 22 + Surrogates Stock	1.0mL
BZ77/110/126 Stock	1.0mL

The following calibration levels are prepared by diluting the indicated volume of the above stock solution to 10mL in methylene chloride.

8.6.1.1 Level 1 (10/0.5ng/mL): 0.010mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.2 Level 2 (20/1.0ng/mL): 0.020mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.3 Level 3 (100/5ng/mL): 0.100mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.4 Level 4 (200/10ng/mL): 0.200mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.5 Level 5 (500/25ng/mL): 0.500mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.6 Level 6 (1000/50ng/mL): 1.0mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.7 Level 7 (2000/100ng/mL): 0.200mL of Stock diluted to a final volume of 1.0mL in methylene chloride.

8.6.1.8 Level 8 (5000/250ng/mL): 5.0mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2 Initial Calibration Verification Solutions (ICV)

8.6.2.1 Initial Calibration Verification PAH SIM Working Solution (500ng/ml): Created by diluting 0.5ml of **PAH ICV Stock** solution (10,000ng/ml) to 10ml in methylene chloride

8.6.2.2 PCB Congener ICV Working (25ng/mL): 1.25mL of **PCB Congener ICV Stock** solution (200ng/mL) diluted to a final volume of 10mL in methylene chloride.

8.6.3 DFTPP Tuning Solution – Prepared by diluting 1.25mL of **Semi-Volatiles GCMS Tuning Standard (**Ultra GCM-150 1000ug/mL) diluted to a final volume of 25mL in methylene chloride.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A method blank (must be extracted with every batch of samples extracted or every 20 samples, whichever is more frequent. Organic compounds of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: Extraction of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. The results are qualified with a “B” flag for any associated sample concentrations that are less then 10x the blank concentration for the analyte. Exceptions may be made with if the samples associated with the out of control method blank are non-detect for the compound of interest, or if sample concentrations are greater than 10x the blank levels. In such cases when the sample results are accepted, the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

Laboratory control samples (LCS/LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent, and spiked with PAH SIM/PCB Congener spike solution, or TO-13 spike solution.

Acceptable Recovery limits are 40% - 140% for all matrices, except for TO-13 Air PUF - see Table D for TO13 LCS recovery criteria. The acceptable relative percent difference (RPD) between the LCS/LCSD is ≤30%. ≤10% of target compounds are allowed out provided recovery is >10%.

PAH and PCB portions need to be evaluated separately

Limits are adapted from MADEP-MCP protocol and/or TO-13A.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the LCS/LCSD recoveries and/or %RPD are still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be reported until an acceptable LCS is obtained.

9.3 Initial Calibration Verification (ICV)

The initial calibration for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing a second source calibration standard (Section 8.2.10).

Calculate the % D for each analyte. If the % D for each analyte is ≤30% then the calibration is assumed to be valid. If this criterion is not met, the standard is reanalyzed once. If the standard failure continues, corrective action must be taken prior to sample analysis.

A new initial calibration must be performed and acceptable ICV results obtained prior to any sample analysis.

9.4 Continuing Calibration Verification (CCV)

On a daily basis after the DFTPP has passed, a mid-level (500/25ng/mL) continuing calibration verification standard which contains all of the analytes of interest is analyzed.

9.5 Matrix Spike/ Matrix Spike Duplicate (MS/MSD)

Matrix spike / matrix spike duplicate (MS/MSD) samples will be performed upon client and/or work plan request. MS/MSD is not performed for Method TO-13A.

See Section 12 for MS/MSD recovery limits and %RPD limits.

Calculate the %RPD as described in Section 9.6.

Corrective Action: If the % recovery and/or %RPD exceeds the control limits and the LCS is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by MS/MSD recoveries and/or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses are performed upon client and/or work plan request. Acceptable relative percent differences (RPD) of duplicates are listed in Section 12. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit.

Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:
R1 = sample Replicate #1
R2 = sample Replicate #2

Corrective Action: If the % RPD exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported organic results as evidenced by the matrix duplicate % RPD exceedance.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate Spikes: Surrogate spikes must be added to QC and field samples to evaluate the extraction method performance.

See Section 12 for surrogate recovery acceptance criteria.

Corrective Action: Up to one surrogate can be out in the PAH portion and in the PCB portion, but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated if an analytical is suspected. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm the sample matrix. If *obvious* matrix interferences are noted, consultation with the Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-extraction. If no re-extraction occurs, the surrogate results and reasons for the no re-extract decision must be discussed in the project narrative to the client.

9.7.2 Standard Reference Materials (SRMs): Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project-specific basis. These are not used as controls, but rather to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

See Section 12 for SRM recovery acceptance criteria.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the % recovery and/or %D still exceeds the control limits, and the LCS and MS/MSD pair are compliant, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by SRM % recoveries and/or %D values outside of QC limits. These cases are normally isolated to the SRM, if all other controls are within limits.

9.7.3 Internal Standards: Internal standards must be added to all sample extracts, QC samples and standards for quantitation purposes.

The internal standards in the samples should remain at constant area counts with respect to the continuing calibration analyzed at the beginning of the run. Sample IS areas must be 50% to 200% of the Internal Standards in the Continuing Calibration.

Additionally, the IS retention times should not differ more than +0.06 RRT from the Continuing Calibration.

Corrective Action: Analysis must be repeated once unless there are obvious samples matrix interferences, e.g. If the sample extract was very colored and viscous, or there are obvious chromatographic interferences. If *obvious* matrix interferences are noted, consultation with the SemiVolatiles Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-analysis.

9.8 Method Sequence

- Tune
- CCV
- Method Blank
- LCS
- LCSD
- Samples

10. Procedure

10.1 Equipment Set-up

10.1.1 The **basic GC parameters** are as follows:

Injection Port Temp: 300°C
Oven Equib Time: 0.50 min
Oven Max: 350°C
Oven: On
Cryo: Off
Ambient: 45°C
Cryo Blast: Off
Initial Temp.: 45°C
Initial Time: 3.00 min

Level	Rate (°C /min)	Final Temp. (°C)	Final Time (min)
1	25.00	250	0
2	10.00	310	5.0
3	0.0off		

Final Time: 22.2min

10.1.2 The **basic injection port parameters** are as follows:

"Split" mode
Equilibrium Time: 0.50 minute
Temp: 300°C
Pressure: 8.28 psi (10.5 psi) Total Flow: 69.3mL/min)
Split: 50:1
Split Flow: 65 mL/min

Injection volume: 3uL

Please note that this may differ slightly from one instrument to another.

10.1.3 Column conditions parameters are as follows:

Column flow: 1.3mL/min
Linear Velocity: 42 cm/sec

10.1.4 Tuning:

10.1.4.1 Tune acceptance should be verified at the beginning of every 12 hour analytical shift. Before the analytical standards are analyzed the mass spectrometer must be adjusted to meet the proper ion criteria for DFTPP. This is demonstrated by injecting into the GC/MS system 1uL of a 50ug/mL DFTPP solution.

10.1.4.2 Within the Enviroquant software, "Autofind" is used first to evaluate Tune. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP.

10.1.4.3 If the "AutoFind" tune evaluation does not meet the criteria below, manual evaluation of the tune can be performed by attempting either of the options below:

- Blow up the DFTPP peak on the screen and select either one single scan at the apex of the peak, or a scan immediately preceding or following the apex.
- Take the average of the scans across the entire peak.

The following DFTPP mass intensity criteria should be used:

- **for PAH SIM analysis:**

The response of Benzidine and Pentachlorophenol, when monitored on a daily basis, will indicate the efficiency of the chromatographic system. Benzidine and Pentachlorophenol should be present at their normal responses and no peak tailing should be visible. The tailing factor of Benzidine and Pentachlorophenol must not exceed 2. DDT breakdown must be <20%. Although moderate tailing may indicate maintenance on the instrument will be required soon, these additional compounds in the DFTPP standard are included as an aid in instrument performance evaluation

DFTPP KEY MASSES AND ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
68	Less than 2 percent of mass 69.
69	confirmed to be present
70	Less than 2 percent of mass 69.
197	Less than 2 percent of mass 198.
198	Base peak, or >50 percent of Mass 442.
199	5-9 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than 24 percent of mass 442.
442	Base Peak, or > 50 percent of mass 198.
443	15-24 percent of mass 442.

- For PAH/PCB combined analysis:

DFTPP KEY MASSES AND ABUNDANCE CRITERIA
(Maximum Sensitivity)

Mass	m/e Abundance criteria
51	N/A
68	N/A
70	N/A
127	30-80 percent of mass 198.
197	Less than 3 percent of mass 198.
198	Greater than 40 percent of mass 442.
199	5-15 percent of mass 198.
275	15-50 percent of mass 198.
365	Greater than 3 percent of mass 198.
441	Present but less than mass 443.
442	Base peak, 100 percent relative abundance.
443	18-30 percent of mass 442.

10.2 Initial Calibration

10.2.1 After the DFTPP passes criteria, a set of multi-level calibration standards listed in Section 8.2.9 are analyzed, from low concentration to high. A minimum of five calibration levels are analyzed. The calibration standards are stored in the standards freezer. When the Initial Calibration is typed into a sequence, the standard ID for each of the levels must be noted in the “comments” of the sequence. (Refer to the Reagent Solvent and Standard Control SOP (SOP 1816) for further information regarding standard labeling convention.

10.2.2 Once the calibration curve is accepted and prior to processing any samples, the analyst must demonstrate through the analysis of a MB or instrument blank that equipment and reagents are free from contaminants and interferences. If a peak is found in the blank that would prevent the identification or bias the measurement of an analyte, the analyst should determine the source of the contaminant peak and eliminate it, if possible. Blanks

are generally considered to be acceptable if target analyte concentrations are less than one half the LLOQ or are less than project specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected

10.2.3 Once the standards have been analyzed, they are reduced by the search software of the Enviroquant data system. Once all the components are identified, a linear curve is calculated for the components. The criteria for evaluation are as follows:

The RSD of each target analyte, for each level in the ICAL, must meet the minimum response factor criteria found in Table C. The %RSD for each target analyte must not exceed 20%. If the %RSD for any target exceeds 20%, then the linear calibration model must be employed. A linear fit is acceptable if the correlation coefficient is greater than 0.99. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model. When calculating calibration curves using the linear regression model, a minimum quantitation check must be performed on the lowest calibration point. The recalculated concentration of the lowest calibration point should be within 30% of the true value. If this criterion is not met then corrective action must take place and a new initial calibration must be performed prior to sample analysis.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Extraction and Cleanup

Samples for PAH SIM/ PCB Congeners are generally extracted using one of the following SOPs:

- *Separatory Funnel Extraction* 3510C (SOP 2165)
- *Microscale Solvent Extraction* 3570 (SOP 2172)
- *Soxhlet Extraction for PUF* 3540C (SOP 2174)
- *Waste Dilution and Oil Prep* 3580 (SOP 2265)

Samples for PAH SIM/ PCB Congeners may be cleaned up before transfer to the instrument laboratory using one of the following SOPs:

- *Gel Permeation Column Cleanup* SOP (SOP 2167)
- *Silica Gel Clean-up* SOP (SOP 2170)
- *Sulfur Cleanup* SOP (SOP 2168)

10.3.2 The prep lab staff will transfer the samples to the instrument laboratory. The samples are generally brought to a 1.0mL to 4.0mL final volume; 1.0mL is transferred and any remaining sample is put into archive. One aliquot of each sample is then logged into the Internal Chain of Custody book and placed in the sample extract holding refrigerator located in the instrument laboratory.

10.3.3 All samples and standards are spiked with Internal Standards (IS) before analysis. The IS is intended to be used for both quantification and the establishment of relative retention times.

10.3.4 The analyst may determine to screen any samples for needed dilution. A sample that will need a dilution either for target analytes that are over calibration, or samples may need to

be diluted solely for matrix issues when the chromatogram shows a large UCM (unresolved complex mixture) or other non-target interferences, or when the extract itself is very colored and viscous. All of the samples at 500uL (including the QC samples of a method blank and LCS) are spiked with 10uL internal standard (see section 10.3.2 for specifics regarding the internal standard) for a concentration of 5ug/mL. The samples are shaken briefly after the internal standard is added to ensure mixing.

10.3.5 After the samples have been analyzed, the data files from the instrument are transferred to the server. The samples are quantified versus the proper method. The QCPRN1.MAC macro creates a form with which to easily check internal standard and surrogate criteria. The ISTDRPT.MAC macro may also be utilized to check the internal standard and surrogate criteria. The following should be reviewed initially:

10.3.5.1 Are all the surrogates within QC criteria? See Section 12 and Table A for surrogate recovery ranges.

10.3.5.2 Are all the internal standards within 50-200% and within ±0.06 RRT of the daily CCAL? If not, the samples should be checked for matrix interferences that may be causing these issues. The IS peaks should also be evaluated for peak splitting or incorrect integration by the software. A sample may not need to be reanalyzed if it can be determined (with guidance from a supervisor) that the QC is exceeded due to matrix interference.

10.3.5.3 Are all target analytes within calibration range? If not, the sample must be diluted and re-analyzed. If a dilution is performed after the internal standard has already been added, it will be necessary to add additional IS in order to provide a concentration of 5ug/mL. Conversely, if a sample has been over-diluted, it may need to be analyzed at less of a dilution to detect target analytes that may have been diluted out.

10.3.5.4 Are all analyses within 12 hour tune time? If a sample is analyzed outside tune time, it must be re-analyzed within another tune clock.

10.3.6 The sequence should also be printed out from Chemstation, initialed and dated, placed in the working logbook.

10.3.7 If anything in the initial review of the data indicates that there should be a re-analysis or a re-extract, the reason for re-analysis or re-extract should be noted on the sequence.

10.3.8 If a re-extract is required, the “Request for Reanalysis/Repreparation” book should be filled out and a photocopy of the appropriate page should be given to the Preparation Group leader or the SemiVolatile Organics Department Manager.

10.3.9 A standard sequence (saved as S5073001;”S” for semivolatile;”5” for PAH5; 0730 for July 30, and 01 is the first sequence that day) would appear as follows:

Tune: WG#-1, -4, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the Tune.

CCV: WG#-2, -3, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the calibration verification.

10.4 Continuing Calibration

10.4.1 On a daily basis after the DFTPP has passed, a mid-level (500/25ng/mL) continuing calibration standard which contains all of the analytes of interest is analyzed. The criteria for acceptance are :

10.4.1.1 The %D for all targets must be ≤20%. Up to 20% of all targets are allowed to have %D > 20% with high bias as long as all associated samples are non-detect for those analytes. If any of the targets have %D > 20% with low bias, corrective action must take place and all associated samples must be reanalyzed.
PAH and PCB portions need to be evaluated separately.

10.4.1.2 The retention times of the internal standards must be within ±0.05 RRT of the previous daily standard.

10.4.1.2 The area counts of the internal standards within the continuing calibration must be within 50 to 200% of the mid-level in previous ICAL

10.5 Preventive Maintenance

All repair and non-routine maintenance records including outside service visits are maintained in the instrument maintenance logbooks.

Injection Port Maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed.

Septum Maintenance: The septum needs to be changed approximately every two-hundred injections. Unscrew the top septum nut, remove the pierced septum, and replace with a new 11mm Thermolite green septum (Restek) or equivalent. Screw the top septum nut back on.

Column Maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed. Generally maintenance is performed by trimming 6 cm off the front of the column. The column is then installed into the injection port liner, and the inlet nut is tightened.

11. Data Evaluation, Calculations and Reporting

11.1 Qualitative Analysis

11.1.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as three ions of greatest relative intensity, or any ions over 20% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

11.1.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. A peak selected by the data system, based on the presence of target specific ions at a target specific retention time will be accepted as meeting this criteria.

11.1.3 The relative retention time of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

11.1.4 The relative intensities of the characteristic ions agree within 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%). Outlying abundances maybe included due to the presence of non-target interference. The relative intensities are monitored daily. The relative intensities will be updated when they exceed established values from the reference spectrum.

11.1.5 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 50% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.1.6 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 spectra and background spectra is important.

11.1.7 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute, (i.e. only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum may contain extraneous ions contributed by the coeluting compounds.

11.2 Response factors and %RSD to evaluate initial calibration acceptability

Calculate RF by:

$$RF = \frac{area_{cmp}}{area_{is}} \times \frac{conc_{is}}{conc_{cmp}}$$

where:

area cmp = Area of the characteristic ion for the compound being measured.
area is = Area of the characteristic ion for the specific internal standard.
conc is = Concentration of the specific internal standard.
conc cmp = Concentration of the compound being measured.

Calculate %RSD by:

$$\%RSD = \frac{SD}{\bar{x}} \times 100 \qquad SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N-1}}$$

where:

%RSD = percent relative standard deviation
x = average of RF's
SD = standard deviation
xi = analytical results of each level in the final reporting units
N = number of results (levels)

11.3 % Difference to evaluate daily calibration standard

Calculate %D by:

$$\%D = \frac{\overline{RF}_i - RF_c}{\overline{RF}_i} \times 100$$

where:

RFi = initial calibration average RF
RFc = continuing calibration RF

11.4 Results of Water Analysis

Calculation as performed in worksheet:

$$\textit{concentration (ng/L)} = \frac{\textit{(Conc) (Vf) (DF) x 1000}}{\textit{(Vi)}}$$

where:

Conc = Raw result concentration obtained from the quantitation report in ng/mL (ppb)
Vf = Final volume of extract (mL)
Vi = Volume of sample extracted (mL)
DF = Dilution factor, for manually prepared dilutions, not instrumental “dilutions”

11.5 Results of Sediment/Soil, Sludge/Waste Tissue, Oil and Air PUF Analysis

Calculation as performed in Excel worksheet:

$$\textit{concentration (}\mu\textit{g/Kg)} = \frac{\textit{(Conc) (Vf) (DF)}}{\textit{(W) (\%S)}}$$

where:

Conc = Raw result concentration obtained from the quantitation report in ng/mL (ppb)
DF = Dilution factor, for manually prepared dilutions, not instrumental “dilutions”
Vf = Extract final volume (mL)
W = Aliquot of sample (wet), g (for Air PUF, sample amount is 1)
%S = Sample % solid

NOTE: Tissue samples are calculated in the same manner, however %S may, or may not be utilized depending upon the client request to report data as a wet or dry weight. Oil and Air PUF sample calculations do not use %S in the final concentration.

11.6 The calculation for percentage breakdown for DDT is:

$$\%\ \text{Breakdown DDT} = \frac{\text{(Area DDD + Area DDE)}}{\text{(Area DDD + Area DDT + Area DDE)}} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All results for the organic compounds of interests are reportable without qualification if extraction and analytical holding times are met, preservation (including cooler temperatures) is met, all QC criteria are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

If non-compliant organic compound results are to be reported, the SemiVolatile Organics Department Manager, the Laboratory Director, and/or the QA Officer must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	Initial Calibration Curve ≤20%RSD or ≥0.99 linear for all targets. If linear regression is used, recalculation of the lowest calibration standard is required to be within 30% of the true value.
Independent Check Verification	+/- 30% recovery of the true values,
Continuing Calibration Verification	Analyzed every 12hr or at the minimum of every 20 samples, < +20% D for all target analytes. .
Method Blank	No analyte detected at or above the reporting limit, "B" qualify analyte if detected concentration is less than 10X the concentration found in the associated sample(s)
Laboratory Control Sample and LCSD	40-140% for all matrices, except for TO-13 - see Table D; 30% RPD.
Matrix Spike / Matrix Spike Duplicate	40-140 recovery; 30% RPD between the duplicates.
Sample / Sample Duplicate	30% RPD between the duplicates.
Surrogates	PAHSIM 30-130% all matrices; PAH/PCB: PAH 30-150% and PCB 50-125% all matrices; USACOE RIM PAH/PCB: 30-150% all matrices; TO-13: see Table A One Surrogate is allowed out from each PAH or PCB portion
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
Standard Reference Material	40-140% recovery based on values provided by Vendor
Project specific work plans and DQOs supersede the above limits.	

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste and Sample Disposal SOP (1797) for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)

SOP 1739 Demonstration of Capability (DOC) Generation
SOP 1797 Hazardous Waste and Sample Disposal

SOP 1731 Manual Integration & Compound Rejection

16. Attachments

Table A: Surrogate Recovery Acceptance Criteria

Table B: Target Compounds and Quantitation Ions

Table C: Recommended Minimum Response Factor Initial and Continuing Calibration

Table D: Matrix Spike and LCS Recovery Limits

Table A
Surrogate Recovery Acceptance Criteria

PAH Surrogate (separate analysis)	30-130% all matrices
PCB Congener Surrogate (separate analysis)	50-125% all matrices
PAH/PCB Surrogate (combined analysis)	PAH: 30-150%; PCB: 50-125% all matrices
USACOE RIM PAH/PCB Surrogate	30-150% all matrices

Surrogate Recovery Acceptance Criteria for TO-13

Compound	Recovery Limits (%)
PYRENE-D10	60-120
BENZO(B)FLUORANTHENE-D12	60-120
FLUORANTHENE-D10	60-120
BENZO(A)PYRENE-D12	40-120

Table B
Target Compounds and Quantitation Ions

Compound	Quantitation Ions Primary	Secondary
Acenaphthene	153	154
Acenaphthene-d10 **(IS)**	164	162
Acenaphthylene	152	151
Anthracene	178	176
Benzo(a)Anthracene	228	229
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(g,h,i)perylene	276	138
Benzo(a)pyrene	252	253
Chrysene	228	226
Chrysene-d12 **(IS)**	240	120
Dibenz(a,h)anthracene	278	139
Fluoranthene	202	101
Fluorene	166	165
2-Methylnaphthalene-D10 **(SURR)**	152	150
Indeno(1,2,3-cd)pyrene	276	138
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
Naphthalene	128	129
Naphthalene-d8 **(IS)**	136	68
Fluoranthene-D10 **(SURR)**	178	179
Perylene-d12 **(IS)**	264	260
Phenanthrene	178	179
Phenanthrene-d10 **(IS)**	188	94
Pyrene	202	200
Pyrene-D10 **(SURR)**	212	211
1,4-Dichlorobenzene	146	148
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
Dibenzothiophene	184	139
Benzo(a)pyrene-D12 **(SURR)**	264	132
2-Chloronaphthalene	162	164
Benzo)b)fluoranthene-D12 **(SURR)**	264	260
Biphenyl	154	153
2,6-Dimethylnaphthalene	156	155
2,3,5-Trimethylnaphthalene	170	155
1-Methylphenanthere	192	191
Perylene	252	253
Benzo(e)pyrene	252	253
BZ#15-C13 (IS)	234	236
BZ#180-C13 (IS)	406	408
DBOB (Surr)	456	296
BZ198 (Surr)	428	429
BZ#8	222	224
BZ#18	256	258

Table B (continued)
Target Compounds and Quantitation Ions

Compound	Quantitation Ions	
	Primary	Secondary
BZ#28	256	258
BZ#52	292	290
BZ#49	292	290
BZ#44	292	290
BZ#66	292	290
BZ#101	326	324
BZ#87	326	324
BZ#77	292	290
BZ#110	326	324
BZ#118	326	324
BZ#153	360	362
BZ#184	394	396
BZ#105	326	324
BZ#138	360	362
BZ#126	326	324
BZ#187	394	396
BZ#183	394	396
BZ#128	360	362
BZ#180	394	396
BZ#170	394	396
BZ#195	428	429
BZ#206	464	466
BZ#209	498	500

Table C: Recommended Minimum Response Factor Initial and Continuing Calibration

Semivolatile Compounds	Minimum Response Factor (RF)
Naphthalene	0.7
2-Methylnaphthalene	0.4
1,1'-Biphenyl	0.01
Acenaphthylene	0.9
Acenaphthene	0.9
Fluorene	0.9
Phenanthrene	0.7
Anthracene	0.7
Fluoranthene	0.6
Pyrene	0.6
Benzo(a)anthracene	0.8
Chrysene	0.7
Benzo(b)fluoranthene	0.7
Benzo(k)fluoranthene	0.7
Benzo(a)pyrene	0.7
Indeno(1,2,3-cd)pyrene	0.5
Dibenz(a,h)anthracene	0.4
Benzo(g,h,i)perylene	0.5

Table D. Matrix Spike and LCS Recovery Limits

Compound	All Matrices Recovery Limits (%)
Acenaphthene	40-140
Acenaphthylene	40-140
Anthracene	40-140
Benzo(a)Anthracene	40-140
Benzo(b)fluoranthene	40-140
Benzo(k)fluoranthene	40-140
Benzo(g,h,i)perylene	40-140
Benzo(a)pyrene	40-140
Chrysene	40-140
Dibenz(a,h)anthracene	40-140
Fluoranthene	40-140
Fluorene	40-140
Indeno(1,2,3-cd)pyrene	40-140
Naphthalene	40-140
Phenanthrene	40-140
Pyrene	40-140
1,4-Dichlorobenzene	40-140
2-Methylnaphthalene	40-140
1-Methylnaphthalene	40-140
Dibenzothiophene	40-140
2-Chloronaphthalene	40-140
Biphenyl	40-140
2,6-Dimethylnaphthalene	40-140
2,3,5-Trimethylnaphthalene	40-140
1-Methylphenanthere	40-140
Perylene	40-140
Benzo(e)pyrene	40-140
BZ#8	40-140
BZ#28	40-140
BZ#52	40-140
BZ#49	40-140
BZ#44	40-140
BZ#66	40-140
BZ#101	40-140
BZ#87	40-140
BZ#77	40-140
BZ#110	40-140
BZ#118	40-140
BZ#153	40-140
BZ#184	40-140
BZ#105	40-140
BZ#138	40-140
BZ#126	40-140
BZ#187	40-140
BZ#183	40-140
BZ#128	40-140
BZ#180	40-140
BZ#170	40-140
BZ#195	40-140
BZ#206	40-140
BZ#209	40-140

Matrix spike and LCS recovery Limits for TO-13

Compound	Recovery Limits (%)
Acenaphthene	60-120
Acenaphthylene	30-114
Anthracene	40-98
Benzo(a)Anthracene	60-120
Benzo(b)fluoranthene	60-120
Benzo(k)fluoranthene	60-120
Benzo(g,h,i)perylene	60-120
Benzo(a)pyrene	60-120
Chrysene	60-120
Dibenz(a,h)anthracene	60-120
Fluoranthene	60-120
Fluorene	60-120
Indeno(1,2,3-cd)pyrene	60-120
Naphthalene	60-120
Phenanthrene	60-120
Pyrene	60-120
1-Methylnaphthalene	60-120
2-Methylnaphthalene	60-120

APPENDIX D

STANDARD OPERATING PROCEDURES

SOP D-11:
Organic Vapor Monitoring Utilizing a PID

PURPOSE

The purpose of this standard operating procedure (SOP) is to provide general guidance for conducting field measurement of organic vapors/gases utilizing a photoionization detector (PID).

This procedure covers the screening of environmental media (water, soil, sediment and air monitoring) utilizing the following instruments:

1. Thermo Environmental Instruments, Inc., (TEI) Organic Vapor Meter (OVM), Model 580B; and,
2. MiniRae 2000 Portable VOC Monitor (Model PGM-7600).

This SOP includes calibration/operation procedures for the aforementioned detectors as well as procedures for field screening of environmental media and air. In addition to this SOP, manufacturer specific instruction manuals should also be consulted prior to detector usage.

The selection of an appropriate detector, detector lamp, calibration gas standard, correction factor/response factor, screening mode, alarm limits, etc., shall be determined prior to site mobilization and defined in the site-specific Health and Safety Plan (HASP).

EQUIPMENT AND MATERIALS

- PID including probe assembly and hydrophobic filter (water trap) assembly;
- Spare batteries and/or battery charger;
- Detector lamp (selection based on the ionization potential (IP) of the site contaminants of concern (COCs);
- Calibration gas (100 parts per million [ppm] isobutylene-in-air and zero air); with regulator

  Note: The calibration standard must be valid within the expiration date;
- Tedlar® Sampling Bag (minimum 1 L capacity) Note: 1 bag per calibration standard is required;
- Teflon tubing (0.25”OD x 0.17”ID) for connections to the Tedlar® Sampling Bag;
- Masterflex® Silicon Tubing L/S 15 (0.39”OD x 0.19”ID) or equivalent for connections to the Tedlar® Sampling Bag;
- Field screening containers (i.e., 8-ounce or 16-ounce glass “driller” jars with screw caps, quart-size or smaller polyethylene Ziploc® bags); and
- Aluminum foil

I. CALIBRATION/OPERATION

Regardless of the make and model of the selected PID, the detector should be fully charged and calibrated on site, prior to the start of daily field activities. During precipitation events and/or extreme heat/cold, the detector may be calibrated at an off-site location or site vehicle as long as the exhaust of all running vehicles is directed away from the area where the detector is being calibrated. At the end of the field day, the detector should be checked against the calibration standard(s) to confirm that calibration has been maintained throughout the day. In the event that the detector readings appear to be irregular or drifting while in use, the instrument should be checked against the calibration standard and/or recalibrated prior to collection of additional field measurements.

For ease of calibration, refer to the below applicable calibration procedures for the selected field detector. The *Special Notes* section at the end of this document and the manufacturer specific instruction manual provide

additional details regarding lamp selection, instrument calibration, correction factors/response factors, ionization potentials, etc. Additional information such as setting alarm limits, maintenance, troubleshooting, etc., may also be found in the instruction manual. Manufacturer specific instruction manuals for the specific instruments used shall be onsite during each sampling event.

1. Thermo Environmental Instruments, Inc., Organic Vapor Meter, Model 580B

A. Preparation for Calibration and Use

1. Allow the temperature of the unit to equilibrate to its surroundings. This could take up to 15 minutes depending on the difference between the temperature of the environment where the detector was stored prior to use and the temperature on site. Note: The range of operating temperatures for this instrument is 32°F to 105°F.
2. Attach probe tip and hydrophobic (water trap) filter by screwing it to the detector inlet. Ensure that the probe tube, filter, and detector inlet fittings are tight. Note: Do not operate the 580-B without a water trap filter installed. Operating without it could cause the pump to strain and/or be damaged and could prevent the sample from reaching the unit. The filter should be replaced when clogged, visibly dirty, and after high field measurements.
3. Insert the three-pronged shorting (power) plug into the RUN/CHG port located on the back of the unit. Align the red marks on the plug and socket. The nub should be on the top of the plug. Warning: If the plug is inserted improperly, or twisted, the fuses in the unit could burn out, rendering the unit inoperable. With the power plug inserted, the LCD screen should indicate "Lamp Out."
4. Turn on the detector by depressing the ON/OFF button. Continue depressing the ON/OFF button until the pump is activated. This will also activate the UV lamp. Once the lamp is lit, the display will show the concentration of what is being drawn into the detector. Measurements will be displayed as parts per million (ppm).
5. Allow the instrument to "warm up" prior to calibrating, by running it for ~ 5 minutes. During this time, fill the Tedlar® Sampling Bag with the calibration reference standard.

B. Calibration

1. Press the MODE/STORE button.
2. The display will read "LOG THIS VALUE? MAX PPM =." Press -/CRSR.
3. The display will read "R/COM, -/PARAM, +/ACCESS, S/CLOCK." Press -/CRSR.
4. The display will read "CONC. METER, MAX HOLD." Press -/CRSR.
5. The display will read "FREE SPACE =." Press -/CRSR.
6. The display will read "RESET TO CALIBRATE." Press RESET.
7. The display will read "RESTORE BACKUP + = YES." Press -/CRSR.
8. The display will read "ZERO GAS RESET WHEN READY." Ensure that the unit is drawing clean ambient air or from a zero air source. Press RESET.
9. The unit will read, "MODEL 580 ZEROING." When it has finished zeroing it will read "SPAN PPM = 0100 '+' TO CONTINUE."
10. Frequently rental units will come from the vendor set for 100 ppm isobutylene-in- air standard. If you are utilizing this standard, skip the next two steps. If your calibration gas is not 100 ppm isobutylene-in-air standard, conduct the following:

a. Hold down the RESET button with one finger. Use another finger to move the cursor with the -/CRSR button. While still holding down the RESET button, use the +/INC button to increase each digit (Note that there is no decrease button. When you get to nine, the next push of the button will return the unit back to zero).

b. Match the number to the concentration on your gas cylinder.

11. Press the +/INC button.

12. The screen will read "SPAN GAS, RESET WHEN READY." Connect the probe tip to a FULL Tedlar® Sampling Bag of 100 ppm isobutylene-in-air standard. Press the RESET button. If the pump sounds like its restricted, the bag is not open enough.

13. The display will read, "MODEL 580 CALIBRATING," followed by "RESET TO CALIBRATE." Press the MODE/STORE button to return to the run mode.

14. While in the run mode, the instrument should read 100 ppm. Remove the gas source and the instrument should read 0 ppm. These measurements serve as "post-calibration" checks. In the event that the unit does not read within +/- 5% of the standard concentration or ambient air concentration of 0 ppm, recalibrate. If the detector cannot be recalibrated to measure within 5% of the calibration standard(s), the instrument should be taken out of service and replaced with a properly functioning unit. All calibration information should be documented on the attached *PID Daily Calibration Field Sheet.*

15. The unit is now ready for use.

C. Post Use Calibration Check and Shut Down

1. Complete an end-of-the-day calibration check. While the instrument is in the run mode, connect the probe tip to a FULL Tedlar® Sampling Bag of 100 ppm isobutylene-in-air standard. NOTE: Use a *fresh* bag of cal gas for the calibration check. A bag filled in the morning may not be accurate in the afternoon, especially if the bag was exposed to sunlight. Record this measurement on the attached *PID Daily Calibration Field Sheet*. If the measurement does not fall within 5% of the calibration standard, the field data will need to be qualified. For multiple days of field use, if the instrument fails the end-of-the-day check on two consecutive days, the unit should be replaced.

2. Turn off the detector by depressing the ON/OFF button. Continue depressing the ON/OFF button until the pump shuts off.

3. Remove the power plug from the RUN/CHG port.

4. Remove the probe tip and water trap filter by unscrewing it from the detector inlet.

5. If recharging is required, attach the battery charger plug into the RUN/CHG port. Plug the associated AC adapter into a wall outlet.

2. MiniRae 2000 Portable VOC Monitor (Model PGM-7600)

[Note that this instrument is not waterproof or water resistant. Do not use it during precipitation events without proper protection from the elements]

A. Preparation for Calibration and Use

1. Allow the temperature of the unit to equilibrate to its surroundings. This could take up to 15 minutes depending on the difference between the temperature of the environment where the detector was stored prior to use and the temperature on site. Note: The range of operating temperatures for this instrument is 14°F to 104°F.

2. Attach probe tip and hydrophobic (water trap) filter by screwing it to the detector inlet. Ensure that the probe tube, filter, and detector inlet fittings are tight. Note: Do not operate the MiniRae 2000 without a water trap filter installed. Operating without it could cause the pump to strain and/or be damaged and could prevent the sample from reaching the unit. The filter should be replaced when clogged, visibly dirty, and after high measurements.
3. Turn on the detector by depressing the MODE button.
4. Allow the instrument to "warm up" prior to calibrating, by running it for ~ 5-10 minutes. During this time the unit will display its setting during the warm up sequence. When it has finished its warm up, readings will be in parts per million (ppm).
5. Fill the Tedlar® Sampling Bag with the calibration reference standard.

B. Calibration

1. To enter the calibration mode, simultaneously press the MODE and N/- buttons until the screen displays "Calibrate/ select Gas?"
2. Press the Y/+ button.
3. Ensure that the unit is drawing clean ambient air or from a zero air source.
4. "Fresh air cal?" is displayed. Press Y/+.
5. The unit will display "zero in progress" followed by "wait" and a 15 second countdown.
6. When the unit is finished zeroing it will display "zeroed! reading 0.0 ppm."
7. Press the MODE button once.
8. Frequently rental units will come from the vendor set for 100 ppm isobutylene-in-air standard. If you are utilizing this standard, skip the next four steps. If your calibration gas is not 100 ppm isobutylene-in-air standard, change the span value by conducting the following:
    a. From the "Span cal" screen, press the N/- button twice or until the screen reads "Change span value." Press Y/+.
    b. The screen will read "Cal gas = isobutylene, Span value = 0100.0."Press the MODE button to move the cursor, and the Y/+ and N/- buttons to increase/ decrease the span value to match the concentration of the calibration gas standard.
    c. When finished changing the value, press and hold the MODE button.
    d. The screen will read "Save?" Press the Y/+ button to save. The screen will read "Saved."
9. Press the MODE button again until "Span cal" is displayed.
10. Press Y/+. The screen will read "Cal gas = Isobutylene, Span value = 0100.0, Apply gas now!"
11. Open and connect a FULL Tedlar® Sampling Bag of 100 ppm isobutylene-in-air standard to the probe tip. The unit will recognize the gas and start to span. The screen will read "Wait…." while it counts down from 30 seconds. Some newer units will display "Update data" after the countdown. If the pump sounds like its restricted, the bag is not open enough.
12. When the countdown is finished the screen will read "cal'ed reading = 100 ppm" It should read within a few ppm of the span value.
13. Press MODE once. The screen will read "cal done turn off gas". Press the MODE button twice to return to the run mode.
14. While in the run mode, the instrument should read 100 ppm. Remove the gas source and the

instrument should read 0 ppm. These measurements serve as "post-calibration" checks. In the event that the unit does not read within +/- 5% of the standard concentration or ambient air concentration of 0 ppm, recalibrate. If the detector cannot be recalibrated to measure within 5% of the calibration standards, the instrument should be taken out of service and replaced with a properly functioning unit. All calibration information should be documented on the attached *PID Daily Calibration Field Sheet*.

15. The unit is now ready for use.

C. Post Use Calibration Check and Shut Down

1. Complete an end-of-the-day calibration check. While the instrument is in the run mode, connect the probe tip to a FULL Tedlar® Sampling Bag of 100 ppm isobutylene-in-air standard. NOTE: Use a *fresh* bag of cal gas for the calibration check. A bag filled in the morning may not be accurate in the afternoon, especially if the bag was exposed to sunlight. Record this measurement on the attached *PID Daily Calibration Field Sheet*. If the measurement does not fall within 5% of the calibration standard, the field data will need to be qualified. For multiple days of field use, if the instrument fails the end-of-the-day check on two consecutive days, the unit should be replaced.

2. Turn off the detector by depressing the MODE button for 5 seconds. The unit will beep once per second during the power-down sequence with a countdown timer showing the number of seconds remaining. The message "Off!"... flashes on the LCD display and then the display will go blank indicating that the monitor is turned off.

3. Remove the probe tip and water trap filter by unscrewing it from the detector inlet.

4. If recharging is required, attach the battery charger plug into the DC jack on the instrument. Plug the associated AC adapter into a wall outlet. The unit will turn on and display the message "Deep discharge?". This message will be displayed three times. If a deep discharge is not applied, the unit will move directly on to the charge mode.

II. FIELD SCREENING

A. *Soil/Sediment Screening for VOCs*

1. Screen environmental media following sample collection. Ideally the samples should be screened immediately following collection but there may be circumstances which result in a delay. Note: If samples are to be collected for laboratory analysis, keep these samples separate from media that will be field screened.

2. Fill a glass drillers jar or Ziploc® bag.

   a. If using glass jars:

      1) Fill the jars half way.

      2) Cut two (2) aluminum foil squares (approximately 3-inch by 3-inch).

      3) Seal the top of the jar with aluminum foil and secure the lid.

   b. If using Ziploc® bag:

      1) Half fill the Ziploc® bag.

3. Secure the bag by zipping it closed or using a zip tie. Vigorously shake the sample jar or bag for ~ 30 seconds, 1-2 times during a 10-15-minute period to allow organic vapors to be transferred from the media to the air space above it (headspace).

4. Minimize the duration that the screening containers containing soil/sediment are exposed to

direct sunlight. Note: If ambient temperatures are below 40°F, the samples should be moved into heated space, either building or field vehicle and allowed to warm prior to screening.

5. Prior to screening environmental media, measure the ambient air or background concentrations. Record this information in on the boring log, field book, as or other appropriate field data collection sheet.

6. Use the PID probe to screen the media.

   a. If using glass jars, remove the cover and insert the probe tip through the aluminum foil.

   b. If using Ziploc® bags, unzip the corner of the bag (1-2") or insert the probe tip directly through the bag.

7. Record the maximum reading, which generally occurs within 2-5 seconds. Note: The probe should not make contact soil/sediment or liquid contained in the sample container. Record the maximum concentration measured by the detector onto a boring log, test pit log, or appropriate field data collection sheet as ppm above background.

8. If screening of media shall be performed directly on soil or sediment cores, record the measurements along the length of the core starting at the top of the sample run and progressing in 1-foot intervals the entire length of the core, as well as additional zones where staining or strong odors are observed. If the media is contained within plastic liners, use a knife or screw driver to poke a hole though the liner, to facilitate field screening. Note: The probe tip should not be used to puncture the core liner as it could damage the probe. In addition, soil/sediment could be forced into the probe tip, resulting in the unit being inoperable.

B. ***Air Quality Monitoring for VOCs***

1. Use the PID probe to measure the ambient air or background concentrations around the perimeter of the work area. Record this information on the boring log, field book, or other appropriate field data collection sheet.

2. Once background concentrations have been established, begin collecting measurements within the work area which may include the source area, and breathing zone. The detector should be operated as close to the area being monitored as technically feasible. Record this information in a field book, or other appropriate field data collection sheet.

3. If the air quality measurements are being collected in the "breathing zone" of the work area, compare field measurements to the range of concentrations included HASP to confirm that a hazardous atmosphere does not exist. If safe breathing zone concentrations are not being maintained, the use of additional personal protective equipment (PPE) or termination of work activities may be required.

III. RECORDS AND DOCUMENTATION

Calibration as well as the detector lamp energy, calibration standard, and correction factor/response factors, maintenance for each piece of equipment, etc. will be documented on the calibration logs and included in the reports. A calibration log is provided at the end of this SOP. Field screening measurements shall be recorded on applicable data collection sheets, boring logs and/or field book.

IV. SPECIAL NOTES

1. These instruments should not be used to ascertain whether further personal protective equipment may be necessary.

2. For site COCs, which include volatile organic compound (VOCs) and Semi-Volatile Compounds (SVOCs), knowing the ionization potential (IP) is critical in determining the appropriate detector and lamp for field screening. Note that a single detector and lamp combination does not exist for all potential site COCs. The manufacturer's instruction manual and/or additional outside references must be consulted in order to assist with proper instrument and lamp selection.

3. An appropriately selected detector will consist of a lamp with energy greater than the highest IP identified for the site COCs. As a general rule of thumb, if site COC have IPs less than 11.8 electron volts (eV), it is possible to use a PID for field screening. Confirm with the project manager and/or health and safety officer that the detector and lamp that has been selected is appropriate for the site COCs.

4. The detectors included in this SOP are capable of utilizing a range of lamp energies (i.e. 9.8eV, 10.0 eV, 10.6 eV, 11.7 eV and 11.8 eV). Note that lower energy lamps are more sensitive and "see" fewer compounds than high energy lamps. The higher energy lamps (11.7 eV and 11,8 eV) should be used only when COCs with IPs greater than 10.6 eV are anticipated. Refer to the attached *RAE Systems, A Guideline for PID Instrument Response, Technical Note TN-106* for the IPs of common organic solvents and gases.

5. The Correction Factor (CR) or Response Factor (RF) are synonymous and are utilized to adjust the sensitivity of a PID to directly measure a particular gas compared to the calibration gas. The lower the CF/RF is, the more sensitive a PID is to a gas or vapor. The greater the toxicity of the gas or vapor, the greater the sensitivity the meter needs to be. CFs/RFs permit the calibration of the instrument to one gas while directly reading the concentration of another. This eliminates the need for multiple calibration gases. A 100-ppm isobutylene-in-air standard is frequently used to calibrate PIDs since it is approximately the midpoint of the range of the instrument sensitivities. It is non- toxic, and non-flammable at a concentration of 100 ppm. Any ionizable gas may be used for calibration. Note that the CFs/RFs tend to be detector and/or manufacturer specific. Refer to the manufacturer's instruction manual when selecting the CF/RF and calibration gas. Refer to the attached *RAE Systems, A Guideline for PID Instrument Response, Technical Note TN-106* for Response Factors.

6. PID sensor and lamp cleaning is not required on a regular basis; however, it should be considered routine maintenance and conducted in accordance with the manufacturer's instruction manual. Indications that a sensor and lamp may need to be cleaned could include the inability to calibrate successfully or a detector which is very sensitive to moisture. If liquid of any sort has been drawn into the instrument, the lamp and sensor should be cleaned immediately. The use of the water trap will help prevent accidental drawing of liquid into the sensor.

V. REFERENCES

RAE Systems, A Guideline for PID Instrument Response, Technical Note TN-106

Note: The following references are the manufacture instruction manuals. The appropriate manual for the instrument(s) being used shall be on site during each sampling event.

Thermo Environmental Instruments, Inc., Organic Vapor Meter (OVM), Model 580B, instruction manual dated January 9, 1996.

MiniRae 2000 Portable VOC Monitor, Model PGM-7600, instruction manual (Revision E) dated May 2005.

VI. ATTACHMENTS

PID Daily Calibration Log

SOP D-19:
Calibration of Field Instruments

PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a framework for calibrating field instruments used to measure water quality parameters for ground water, surface water and pore water. Water quality parameters include temperature, pH, dissolved oxygen (DO), specific conductance, oxidation reduction potential (ORP) and turbidity.

This SOP is written for instruments where the probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature; such as the YSI Models 600XL and the In-Situ smarTROLL™ multiparameter meter (referred to as the Smartroll in this SOP). Meters measuring pH must be calibrated using three pH standards (4, 7 and 10). Turbidity must be taken with a separate meter (such as the Hach 2100P or 2100Q Turbidity meters).

For ground water monitoring, the instrument must be equipped with a clear flow-through cell with a maximum capacity of 250 milliliters and the display/logger or computer display screen needs to be large enough to simultaneously display the readouts of each probe in the instrument. Turbidity samples must be taken at a point before the flow-through cell and analyzed in a separate meter. A three-way stopcock is needed to divert sample flow prior to the flow-through cell so that an aliquot can be collected for the turbidity reading. Turbidity cannot be measured in or after a flow-through cell because the flow-through cell acts as a sediment trap. This procedure is applicable for use with the current SOP D-43: *Low Flow Groundwater Purging and Sampling*.

HEALTH AND SAFETY WARNINGS

Read all labels on the standards and note any warnings on the labels. Wear appropriate personal protection equipment (e.g., gloves, eye shields) when handling the standards. If necessary, consult the Safety Data Sheets for additional safety information on the chemicals in the standards.

CALIBRATION ACCEPTANCE CRITERIA

The instruments shall be calibrated at the beginning of each sampling day at the Site prior to sample collection. The calibration shall then be checked immediately following the calibration to ensure the instrument was calibrated properly. If the morning calibration check is not within the acceptable range for a parameter, the instrument shall be recalibrated using all the standards for that parameter and the calibration shall be checked again. See individual parameters for specific instructions. In general, if the calibration or calibration check is not successful, recalibrate using a new standard solution with a different lot number and recheck. Then, if the calibration or calibration check is still unsuccessful, replace the meter with a backup unit. Backup instruments shall be fully calibrated, checked and used in place of the inoperable unit.

The calibration shall be checked again at the end of the day of use to ensure that the instruments have remained in calibration throughout the day. In addition, should any erratic or illogical readings occur during the field day and between calibrations/calibration checks, the calibration shall be re-checked for those parameters and recalibrated, as necessary, in order to ensure that representative measurements are obtained. All calibration and check values shall be documented on the Calibration Log (see attached log).

If a calibration check at the end of the day is not within the acceptable range for that parameter, the data collected that day for that parameter shall be qualified in its use. This qualification shall be documented on the Calibration Log and the field sheets/logs for the appropriate sampling locations. For example: pH measurements are collected as part of the low flow sampling procedure. If the afternoon pH calibration check was not within the acceptable range that day, the pH data collected by that instrument on that day would be qualified as useful only for determining stabilization and not as representative pH measurements of the water being sampled. That qualification would then be documented on the Calibration Log and the sampling worksheets for each of the locations where the instrument was used.

WEATHER CONDITIONS

Normally, everyday calibration procedures are performed in the field. However, under adverse weather conditions, it is permissible to perform the beginning-of-day calibration and calibration check in the office or other facility just prior to going

into the field. It is also permissible under similar conditions to perform the end-of-day calibration check off-site. The calibration solutions must be brought into the field with the multiparameter meters and protected from extreme temperatures.

EQUIPMENT AND MATERIALS

The following is a list of equipment and materials required for calibration:

- Site-specific QAPP.
- Manufacturer's instruction manuals (including the instrument specifications) to accompany the instruments into the field.
- Multi-meter sonde and handheld meter.
- Calibration solutions:

  It is advisable to have a sufficient number of extra bottles of each standard on hand in case of complications (especially the conductivity standards). Be sure that the standards are not near their expiration dates (e.g., greater than one month from expiration).

  - Small wet sponge or paper towel for DO 100% saturation calibration.
  - "Zero" (0) milligrams per liter (mg/L) DO check standard (minimum of two bottles from two separate lots for the Smartroll).
  - pH buffers 4, 7 and 10.
  - Two standards for specific conductance: 718 microsiemens per centimeter (µS/cm) and 1,413 µS/cm. One to use for calibration and the other one to use for checking the calibration.
  - Zobell Solution for ORP.
- Separate Turbidimeter w/calibration standards: <0.1, 10, 20, 100, 800 Nephelometric Turbidity Units (NTUs) as appropriate for each meter.
- Calibration cup/Storage cup.
- Cooler (for storage of calibration solutions).
- Laboratory-grade deionized (DI) water.
- Paper towels.
- Kimwipes.
- Calibration log.
- If using the Smartroll, you MUST request the following specific equipment from the vendor as they may not automatically be provided with the Smartroll:

  - A Power Pack that has the firmware update to be used with Android or iOS. The Power Pack enables wireless communication between the device and the probe and supplies power to the probe.
- The Smartroll MP Storage and Calibration Cup

The Storage and Calibration cup (referred to as the storage cup when discussing the Smartroll in this SOP) is a rugged alternative to the standard calibration cup. This storage cup is not provided with the Smartroll multiparameter meters from In-Situ. The vendor orders them separately from In-Situ and you must specifically request the storage cup from the vendor.

Do not use the larger and more flexible calibration cup referred to in the manual (with or without the probe guard) the for the following reasons: 1) the probe guard makes it hard to see the probes making rinsing and drying the probes between each standard unnecessarily difficult; 2) the large cup requires more calibration solution; 3) the calibration cup is flimsy; and 4) the flexible collar makes it harder to connect to the rest of the calibration apparatus and instrument.

GENERAL INFORMATION

This SOP requires that the manufacturer's instruction manuals (including the instrument specifications) accompany the instruments into the field.

In general, all instrumentation necessary for field monitoring and health and safety purposes shall be maintained, tested and inspected according to the manufacturer's instructions. It is assumed that most of this equipment will be rented and is not owned by the contractor. Any reference made to a vendor applies to the owner/renter of the equipment.

All calibration solutions shall be stored at room temperature or at cool/stable temperatures in the field. Storage of calibration solutions in an insulated cooler kept in the shade will help to maintain calibration solution integrity.

All calibration solutions shall be placed into the calibration cup (or, in the case of the Smartroll, the storage cup) to calibrate the instrument and to check the calibration. The calibration cup shall be rinsed with DI water and dried with paper towels or Kimwipes between each standard. The probes shall not be put directly into the bottles of calibration solutions from the vendor. The volume of the calibration solutions must be sufficient to cover both the probe and temperature sensor. See manufacturer's instructions for additional information. Do not pour the used calibration solutions back into their original bottles.

While calibrating or measuring, make sure there are no air bubbles lodged on or between the probes.

Calibration standard values, check results, temperature and barometer checks, and maintenance for each piece of equipment shall be documented on the Calibration Logs and included in the reports. This information includes dates, personnel, calibration standards expiration dates, etc. A Calibration Log is provided at the end of this SOP.

GENERAL PRELIMINARY CALIBRATION PROCEDURES

Prior to calibration, all instrument probes must be cleaned in accordance with the manufacturer's instructions, preferably by the vendor if the unit is to be rented. Failure to perform this proper maintenance step can lead to erratic measurements. The vendor is required to provide written documentation (which will be included in sampling reports) that indicates the equipment was cleaned, who cleaned it, and the date of the cleaning.

Program the multi-probe instrument so that the following parameters to be measured will be displayed: temperature in ˚C; pH; DO in % for calibration and mg/L for measurements; DO charge in millivolts; specific conductance in µS/cm; and ORP in millivolt (mV). The DO charge is not applicable for the Smartroll.

Allow all calibration standards to equilibrate to the ambient temperature. Mark the "date opened" on each new bottle of calibration solution.

MULTIPARAMETER METER CALIBRATION PROCEDURES

TEMPERATURE (THIS SECTION APPLIES TO BOTH YSI AND IN-SITU MODELS)

This procedure is not to be done in the field.

For instrument probes that rely on the temperature sensor, each temperature sensor must be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST) prior to the sampling event. A temperature check is required once a year for each instrument, at a minimum.

The temperature check shall be performed prior to the field event, preferably via the vendor if the unit is rented. If the check is not performed by the vendor, then it must be performed by field personal prior to using the unit. Verification and documentation, including accuracy, dates and personnel, of this procedure are required. The documentation shall be recorded on the Calibration Log and included in any sampling reports.

TEMPERATURE SENSORY ACCURACY PROCEDURE

1. Allow a container filled with water to come to room temperature.

2. Place a NIST thermometer and the instrument's temperature sensor into the water and wait for both temperature readings to stabilize.

3. Compare the two measurements and record the results on the Calibration Log. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (typically ±0.15 °C or ±0.2 °C). Check the manual that came with the instrument. If the measurements do not agree, the instrument may not be working properly and the vendor/manufacturer may need to be consulted or the unit replaced.

A. YSI 600XL CALIBRATION/CALIBRATION CHECK PROCEDURES

Preliminary/general steps to set up the instrument for calibration:

1. Make sure that the cable is connected to the sonde and the handheld display.

    a. Align the pins on the cable with the pins on the sonde, and then twist the outer portion of the connector until the connection is secure.

    b. Align the pins on the cable with the pins on the handheld display and then twist the outer portion of the connector until the connection is secure.

2. Turn on the instrument and allow it to warm up according to the manufacturer's instructions.

3. Select Calibrate from the Main Menu.

YSI - DISSOLVED OXYGEN

The YSI instrument measures dissolved oxygen (DO) content in water using a membrane electrode. The instrument is calibrated to 100% DO saturation and then the calibration is checked with a 0% saturated DO solution.

The DO probe's membrane and electrolyte solution shall be replaced prior to the sampling event and replaced as needed thereafter. Failure to perform this step may lead to erratic measurements. If the vendor changes the membrane and electrolyte solution, they must send the appropriate documentation with each unit. If there is no documentation with the unit, the field personnel will have to replace the membrane and electrolyte solution before the sampling event begins. Documentation shall be noted on the Calibration Log.

YSI - DO CALIBRATION/CALIBRATION CHECK PROCEDURE

1. Record the DO charge on the worksheet. Note: According to manufacturer, the DO charge should be between 25-75 millivolts for the probe to be working correctly. If the DO charge is outside this range, replace the membrane and electrolyte solution prior to calibration.

2. Gently dry the temperature sensor and remove any droplets of water from the DO probe's sensor membrane according to manufacturer's instructions. Inspect the DO membrane for air bubbles and nicks. If any are found, replace the membrane and electrolyte solution.

3. Place a wet sponge or a wet paper towel on the bottom of the DO calibration container to create a 100 percent water-saturated air environment.

4. Place the DO probe loosely into the calibration container to prevent the escape of moisture evaporating from the sponge or paper towel while allowing pressure equilibration before calibration. Do not allow the probe to come into contact with the wet sponge or paper towel. **The storage cup must be vented to the atmosphere.** Do not screw the calibration cup tightly onto the sonde.

5. Allow the confined air to become saturated with water vapor (saturation occurs in approximately 10 to 15 minutes). Make sure that the instrument is turned on during this time to allow the DO probe to warm-up according the manufacturer's directions. Make sure that both the DO reading and the temperature have stabilized before starting the calibration sequence.

6. Select calibration mode; then select "DO %".

7. Enter the local barometric pressure (usually in mm of mercury) for the sampling location into the instrument using an on-site hand-held barometer, unless the instrument already has a temperature-compensated barometer.

8. Record the barometric pressure on the Calibration Log.

9. The instrument should indicate that the calibration is in progress. Observe the readings for percent dissolved oxygen and temperature. When they show no significant change for approximately 30 seconds, press enter. After calibration, the instrument should display dissolved oxygen in mg/L (% DO is only used for calibration).

10. Record the initial DO reading in mg/L and temperature reading in °C on the Calibration Log immediately after calibration.

11. To check the calibration, select monitoring/run mode (on a run/measurement screen), remove the probe from the container and place it into a 0.0 (zero) mg/L DO standard.

12. Wait until the "mg/L DO" and temperature readings have stabilized. Record the zero mg/L DO reading on the Calibration Log. The instrument must read 0 to 0.5 mg/L DO. If the instrument reads above 0.5 mg/L or reads a negative value, it will be necessary to clean the probe and change the membrane and electrolyte solution. If this is unsuccessful, use a new 0.0 mg/L DO standard. If these measures are still unsuccessful, consult the manufacturer/vendor or replace the unit.

    If the afternoon calibration check is not within the acceptable range then the data collected using the instrument that day must be qualified in its use as described above under Calibration Acceptance Criteria.

13. Remove probe from the zero DO standard, rinse with DI water, and gently blot dry. Rinse the calibration cup with DI water and dry it with paper towels or Kimwipes.

YSI - PH (ELECTROMETRIC)

The YSI instrument measures the PH of a sample electrometrically using a glass electrode. Three standards are needed for the calibration: pH 4, 7 and 10. Rinse the calibration cup with DI water and dry it with paper towels or Kimwipes between standards.

YSI - PH CALIBRATION/CALIBRATION CHECK PROCEDURE

1. Allow the buffered standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the buffered standards so each standard will cover the pH probe and temperature sensor.

3. Remove probe from its storage container, rinse with DI water, and gently blot dry with a Kimwipe. Use caution during drying, such that the dissolved oxygen probe membrane is not disturbed.

4. Select the calibration mode for a three-point pH calibration.

5. Immerse probe into the initial standard, pH 7.

6. Enter the buffered standard value (e.g., pH 7) into the instrument. Wait until temperature and pH readings stabilize. If the readings do not change within 30 seconds, press enter to accept the calibration.

7. Remove probe from the initial standard, rinse with DI water, and gently blot dry.

8. Immerse probe into the second standard (pH 4). Repeat step 6.

9. Remove probe from the second standard, rinse with DI water, and gently blot dry.

10. Immerse probe into the third standard (pH 10) and repeat step 6.

11. Remove probe from the third standard, rinse with DI water, and gently blot dry.

12. To check the calibration, select monitoring/run mode (on a run/measurement screen) and immerse the probe into the pH 7 buffer solution. Wait for the temperature and pH readings to stabilize. Record the pH value on the Calibration Log. The value must be pH 7 +/-5% (pH 6.65-7.35). If the calibration check failed, recalibrate the instrument using fresh standards for all three values and check it again. If re-calibration fails, clean the pH probe, recalibrate and check the calibration. If the calibration check fails again, consult the manufacturer/vendor or replace the unit.

    If the afternoon calibration check is not within the acceptable range then the data collected using the instrument that day must be qualified in its use as described above under Calibration Acceptance Criteria.

13. Remove probe from the pH 7 check standard, rinse with DI water, and gently blot dry. Rinse the calibration cup with DI water and dry it with paper towels or Kimwipes.

YSI - SPECIFIC CONDUCTANCE

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected to 25°C. When monitoring groundwater, surface water or pore water, use the specific conductance readings and record in µS/cm.

Most instruments are calibrated against a single standard which is near, (above or below) the specific conductance of the environmental samples. A second standard is used to check the linearity of the instrument in the range of measurements.

Specific conductivity standards concentrations are generally dependent on expected field conditions and availability. However, there have been some issues with the stability of some of the standards in the field.

Therefore, the HWRB recommends using the following standards as they have been field tested, are readily available from most vendors, and are acceptable for use by EPA: a 1,413 µS/cm standard and a 718 µS/cm standard. It is acceptable to use either one of the standards to calibrate and the other to check the calibration.

In general, the 718 µS/cm standard will be used to calibrate, and a 1413 µS/cm standard used to check the calibration. **It is advisable to have a sufficient number of extra bottles of each standard on hand in case of complications. Be sure that the standards are not near their expiration dates (e.g., greater than one month from expiration).** Rinse the calibration cup with DI water and dry it between standards.

Preliminary Cleaning Procedure

Before calibrating for specific conductance, clean the probe according to the manufacturer. In general, dip a small cleaning brush in DI water and insert it into each hole 15-20 times. Rinse with DI water and dry thoroughly.

YSI - Specific Conductance Calibration/Calibration Check Procedure

1. Allow the calibration standards to equilibrate to the ambient temperature.

2. Remove the probe from its storage container, rinse the probe with a small amount of the first (718 µS/cm) specific conductance standard, discard the rinsate and place the probe into the standard. Be sure that the temperature sensor and the probe's vent hole are fully immersed in the standard. Gently move the sonde up and down to dislodge any air bubbles from the conductivity cell.

3. Allow at least one minute for temperature equilibrium before proceeding.

4. Select the calibration mode for specific conductance. Enter the calibration value of the standard being used (718 µS/cm). Allow the temperature and specific conductance to stabilize. If the reading does not change within 30 seconds, press enter to accept the calibration.

5. Remove probe from the specific conductance check standard, rinse with DI water, and gently blot dry. Rinse the calibration cup with DI water and dry it with paper towels.

6. To check the calibration, select the monitoring/run mode (a run/measurement screen). Remove the probe from the first standard, rinse the probe with DI water and then a small amount of the second (1413 µS/cm) standard, discard the rinsate and place the probe into the second standard. The second standard will serve to verify the linearity of the instrument. Wait until the specific conductance and temperature readings have stabilized. Read the specific conductance value from the instrument and record the value on the Calibration Log. Compare the value to the standard. The value must be +/-5%.

   When the 718 µS/cm standard is used to calibrate, 5% of the 1413 µS/cm solution is 1342-1484 µS/cm. When the 1413 µS/cm standard is used to calibrate, 5% of the 718 µS/cm solution is 682-754 µS/cm.

   If the applicable range is not met, check the calibration using a fresh solution with a different lot number from that used in the initial calibration check. Then, if the range is not met, recalibrate using a fresh calibration solution with a different lot number and check again.

   If this range is still not met, calibrate using the second calibration solution and repeat the process above. If this is not successful, consult the manufacturer/vendor or replace the unit.

For example: if the 718 µS/cm standard is used to calibrate, then the 1413 µS/cm solution is used to check the calibration. If the 1413 µS/cm solution is not within the acceptable range of 1342-1484 µS/cm, then check the calibration with a fresh 1413 µS/cm solution with a different lot number. If this range is still not met, recalibrate the instrument using a new 718 µS/cm standard from a different lot number and check again.

If the acceptable range is still not met, calibrate the instrument using the 1413 µS/cm standard and check the calibration with a 718 µS/cm solution. If the 718 µS/cm solution is not within the acceptable range of 682-754 µS/cm, then check the calibration with another 718 µS/cm solution with a different lot number. If this range is still not met, recalibrate the instrument using a new 1413 µS/cm standard from a different lot number and check again.

If that does not solve the problem, consult the manufacturer/vendor or replace the unit.

If the afternoon calibration check is not within the acceptable range then the data collected using the instrument that day must be qualified in its use as described above under Calibration Acceptance Criteria.

Remove probe from the specific conductance check standard, rinse with DI water, and gently blot dry. Rinse the calibration cup with DI water and dry it with paper towels.

YSI - OXIDATION/REDUCTION POTENTIAL

The oxidation/reduction potential (ORP) is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts and is temperature dependent. A Zobell solution is required to calibrate ORP. Read the warning on the label before use.

A. YSI ORP Calibration/Calibration Check Procedure

1. Allow the Zobell solution calibration standard to equilibrate to ambient temperature.

2. Remove the probe from its storage container, rinse the probe with DI water, gently blot dry with a Kimwipe and place it into the standard.

3. Select monitoring/run mode.

4. Wait for the probe temperature to stabilize, and then read the temperature. Record the temperature reading on the Calibration Log.

5. Look up the mV value at this temperature from the temperature / mV chart found below and on the Calibration Log. These values have been rounded to the nearest whole number. Record this value on the Calibration Log.

6. Select the calibration mode for ORP. Enter the temperature-corrected ORP value into the instrument. Once the temperature and ORP values stabilize, press enter to accept the calibration.

7. To check the calibration, select monitoring/run mode (on a run/measurement screen). Immerse the probe in the Zobell solution. Wait until the ORP and temperature readings have stabilized. Read the temperature and the ORP on the instrument. Record the values on the Calibration Log. The instrument value must be within +/- 5% of the mV value for the current temperature. See the chart below for the check range. If it is not within +/- 5%, recalibrate using a new Zobell solution. If the re-calibration is not successful, consult the manufacturer/vendor or replace the unit. For the afternoon calibration check, the instrument must be within +/- 5% of the mV value for the current temperature or the data collected using the instrument that day must be qualified in its use as described above under Calibration Acceptance Criteria.

8. Remove the probe from the ORP check standard, rinse with DI water, and gently blot dry. Rinse the calibration cup with DI water and dry it with paper towels or Kimwipes.

Zobell Solution mV Values Based on Temperature for ORP Calibration
Calibration Check Range Values (+/- 5%)
(Round off temperature to whole number, e.g., 23.5 ºC rounds up to 24 ºC)

Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%	Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%	Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%
-3	**267**	254-280	10	**251**	238-264	23	**234**	222-246
-2	**266**	253-279	11	**249**	237-261	24	**232**	220-244
-1	**265**	252-278	12	**248**	236-260	25	**231**	219-243
0	**264**	251-277	13	**247**	235-259	26	**230**	219-242
1	**262**	249-275	14	**245**	233-257	27	**228**	217-239
2	**261**	248-274	15	**244**	232-256	28	**227**	216-238
3	**260**	247-273	16	**243**	231-255	29	**226**	215-237
4	**258**	245-271	17	**241**	229-253	30	**225**	214-236
5	**257**	244-270	18	**240**	228-252	31	**223**	212-234
6	**256**	243-269	19	**239**	227-251	32	**222**	211-233
7	**254**	241-267	20	**238**	226-250	33	**221**	210-232
8	**253**	240-266	21	**236**	224-248	34	**219**	208-230
9	**252**	239-265	22	**235**	223-247	35	**218**	207-229

B. IN-SITU SMARTROLL CALIBRATION PROCEDURES

When using the Smartroll, calibrate all parameters first, then select the "live readings" screen on the tablet display and check all the parameters after the calibration procedure has been completed.

Preliminary/general steps to set up the instrument for calibration:

1. Make sure that the cable is connected to the instrument and the Power Pack.
    a. Align the pins on the cable with the pins on the probe (sonde), and then twist the outer portion of the connector until the connection is secure.
    b. Align the pins on the cable with the pins on the Power Pack, and then twist the outer portion of the connector until the connection is secure.
2. Storage cup

    Use the storage cup that was specifically requested under the equipment section above to calibrate the instrument (the same type of storage/calibration cup as the YSI unit). **Do not use** the larger and more flexible calibration cup referred to in the manual. The storage cup is not referenced in the user manual because it is sold separately.

    If there is a metal sleeve in the storage/calibration cup, it must be removed before use so that the probes are visible.

    Unscrew the bottom of the calibration cup and pull the sleeve out prior to calibration. Remember to put the sleeve back in before you send the unit back to the vendor.

3. Press the power button on the Power Pack.
4. Turn on the tablet.
5. Tap on the VuSitu icon to open the application in the tablet. Connect to the instrument via Bluetooth. If using more than one Smartroll, be sure to connect to the Bluetooth device you are using and not others around you. The serial number of the Bluetooth Power Pack connected to the multiparameter meter will be displayed on your tablet.

6. Tap the **Calibration** icon (it looks like a laboratory beaker) on the bottom left of the tablet to access a list of sensors that are available for calibration.

7. Then tap the first parameter to be calibrated.

8. Calibration of each parameter should take no more than 1 or 2 minutes and is often quicker than 1 minute. If any parameter calibration reads "nominal", but is within the allowable range, you may accept the calibration. Often waiting another minute will allow the reading to stabilize. If any parameters indicate that they did not stabilize, perform the calibration procedure again.

IN-SITU - DISSOLVED OXYGEN

The In-Situ instrument measures DO content in water using an optical sensor.

The Smartroll performs a two-part calibration for DO: 100% saturation and 0% saturation. The meter should automatically detect all standard calibration solution values. If you are using a non-standard calibration solution, you have the option to manually enter the value.

In-Situ - DO Calibration Procedure

1. Inspect the rugged dissolved oxygen (RDO) cap for air bubbles and nicks. If any are found, replace the RDO cap.

2. Tap the **Calibration** icon on the tablet display to access a list of sensors that are available for calibration.

3. Tap **RDO Saturation** on the tablet display.

4. Select the two-point calibration method to calibrate the sensor: Tap **100% and 0% Saturation**.

5. Remove (unscrew) the bottom of the storage cup and place a water-saturated sponge or a small amount of water (cannot touch DO probe) in the end cap to create a 100 percent water-saturated air environment.

6. Place the probe **loosely** into the storage cup in order to vent the storage cup to barometric pressure. **The storage cup must be vented to the atmosphere.** Do not screw the storage cup tightly onto the meters.

7. Tap **Start** on the tablet display.

8. Allow the confined air to become saturated with water vapor.

9. The instrument will indicate that the calibration is in progress.

10. When the instrument indicates that the calibration is stable, record the initial DO reading in % and mg/l, and temperature reading in °C, on the Calibration Log.

11. Tap the **Accept** button on the tablet display.

12. Remove the sponge, dry the storage cup with paper towels or Kimwipes and add fresh sodium sulfite solution (zero DO solution) to the fill line.

13. Place the instrument into the storage cup, and tap **Start.**

14. When the instrument indicates that the calibration is stable, tap the Accept button.

15. Remove probe from the storage cup containing the zero DO standard, rinse well with DI water, and gently blot dry.

16. Rinse the storage cup well with DI water and dry it with paper towels or Kimwipes.

17. Tap on the left arrow "sensor calibration" in the top left corner of the tablet display to return to the calibration menu to select the next parameter.

RDO SALINITY SETTING

The Smartroll RDO does not include automatic salinity compensation, so you must set it manually.

1. From the main menu, select **Connected Instrument**.

2. Select **Instrument Settings**.

3. From the Instrument Settings menu select **Salinity Setting**.

4. Select the appropriate setting for your sampling environment (e.g., fresh, brackish, or salt water). The typical setting is fresh water.

IN SITU - PH (ELECTROMETRIC)

The pH of a sample is determined electrometrically using a glass electrode. Three standards are needed for the calibration: pH 4, 7 and 10. The meter should automatically detect all standard calibration solution values.

In-Situ - pH Calibration Procedure

1. Tap the **Calibration** icon on the tablet display to access a list of sensors that are available for calibration.

2. Tap **pH Sensor**.

3. Tap **3-Point Calibration**.

4. Fill the storage cup to the fill line with the first calibration buffer (pH 4).

5. Place the instrument into the storage cup, and tap **Start** on the tablet display.

6. When the instrument indicates that the calibration is stable, tap the **Accept** button on the tablet display.

7. Remove probe from the storage cup containing the standard, rinse with DI water, and gently blot dry.

8. Rinse the storage cup with DI water and dry it with paper towels or Kimwipes.

9. Fill the cup to the fill line with the second calibration buffer (pH 7).

10. Place the instrument into the storage cup, and tap **Start** on the tablet display.

11. When the instrument indicates that the calibration is stable, tap the **Accept** button on the tablet display.

12. Remove probe from the storage cup containing the standard, rinse with DI water, and gently blot dry.

13. Rinse the storage cup with DI water and dry it with paper towels or Kimwipes.

14. Fill the cup with the third calibration buffer (pH 10).

15. Place the instrument into the storage cup, and tap **Start** on the tablet display.

16. When the instrument indicates that the calibration is stable, tap the **Accept** button on the tablet display.

17. The tablet will automatically return to the initial pH Calibration screen (where the 3-point calibration was selected).

18. Remove probe from the storage cup containing the standard, rinse with DI water, and gently blot dry.

19. Rinse the storage cup with DI water and dry it with paper towels or Kimwipes.

20. Tap on the left arrow "sensor calibration" in the top left corner of the tablet display to return to the calibration menu to select the next parameter.

IN-SITU - SPECIFIC CONDUCTANCE

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected to 25°C. When monitoring groundwater, surface water, or pore water, use the specific conductance readings and record in µS/cm.

Most instruments are calibrated against a single standard that is near (above or below) the specific conductance of the environmental samples. A second standard is used to check the linearity of the instrument in the anticipated range of measurements. Specific conductivity standards concentrations are generally dependent on expected field conditions and availability. However, there have been some issues with the stability of some of the standards in the field.

Therefore, the HWRB recommends using a 1413 µS/cm standard and a 718 µS/cm standard as they have been field tested, are readily available from most vendors, and are acceptable for use by EPA. It is acceptable to use either one of the standards to calibrate and the other to check the calibration.

The use of only ONE standard to both calibrate and check the calibration is NOT ACCEPTABLE, as the second standard is used to check the linearity of the instrument in the range of measurements.

In general, the 718 µS/cm standard will be used to calibrate, and a 1413 µS/cm standard used to check the calibration. **It is advisable to have a sufficient number of extra bottles of each standard on hand in case of complications. Be sure that the standards are not near their expiration dates and are from different lots.** Rinse the storage cup with DI water and dry it between standards.

The meter should automatically detect all standard calibration solution values. If you are using a non-standard value calibration solution (e.g., 718 µS/cm), you have the option to manually enter the value.

Preliminary Cleaning Procedure

Before calibrating for specific conductance, clean the probe according to the manufacturer. Avoid damaging the plastic material of the conductivity cell. In general, gently wipe the conductivity cell with a soft polyurethane foam swab, or a thin cotton pipe cleaner, and DI water using a gentle back-and-forth motion. Rinse the probe with DI water and dry thoroughly.

In-Situ - Specific Conductance Calibration Procedure (718 µS/cm standard)

1. Tap the **Calibration** icon on the tablet display to access a list of sensors that are available for calibration.

2. Tap **Conductivity Sensor**.

3. Tap 1-Point Calibration.

4. Fill the storage cup to the fill line with 718 µS/cm calibration standard.

5. Place the instrument into the storage cup, and tap **Start** on the tablet display.

6. The tablet app does not automatically recognize the value of the 718 µS/cm standard. The value of the standard must be manually entered into the tablet app. To enter the value manually:

   a. At the top of the screen there is white blank box to the left of "µS/cm".

   b. Tap the blank box and a keyboard will appear.

   c. Enter 718 and tap the return button.

   d. The meter will start the calibration process.

   Note: If using the 1413 µS/cm calibration standard to calibrate, the tablet app will automatically recognize the value of the 1413 µS/cm calibration standard; therefore, the value does not have to be manually entered.

7. When the instrument indicates that the calibration is stable, tap the **Accept** button on the tablet display.

8. Remove probe from the storage cup containing the standard, rinse with DI water, and gently blot dry.

9. Rinse the storage cup with DI water and dry it with paper towels or Kimwipes.

10. Tap on the left arrow "sensor calibration" in the top left corner of the tablet display to return to the calibration menu to select the next parameter.

<u>IN-SITU - OXIDATION/REDUCTION POTENTIAL (ORP)</u>

The oxidation/reduction potential is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts and is temperature dependent. A Zobell solution is required to calibrate ORP. Read the warning on the label before use.

The meter should automatically detect all standard calibration solution values including the ORP standard corrected for temperature.

In-Situ - ORP Calibration Procedure

1. Tap the **Calibration** icon on the tablet display to access a list of sensors that are available for calibration.

2. Tap **ORP Sensor**.

3. Tap 1-Point Calibration

4. Fill the storage cup with Zobell calibration standard.

5. Place the instrument into the storage cup, and tap **Start** on the tablet display.

6. When the instrument indicates that the calibration is stable, record the ORP and temperature values on the Calibration Log.

7. Tap the **Accept** button on the tablet display.

8. Tap on the left arrow "sensor calibration" in the top left corner of the tablet display to return to the calibration menu to select the next parameter.

Once the last parameter has been calibrated, then tap on the left arrow on the tablet display and select "live readings" screen to perform the calibration check procedure.

In-Situ Smartroll Calibration Check Procedure

General procedure to check the parameter calibration:

1. Select the "live readings" screen on the tablet display.
2. Fill the storage cup to the fill line with the appropriate calibration check standard and insert the probe in the solution.
3. Wait until the temperature and parameter readings have stabilized (i.e., no changes in the temperature and parameter readings for minimum of 30 seconds).
4. Record the parameter reading on the Calibration Log.
5. Refer to each parameter below for instructions to follow if the check standard is outside the acceptable range.
6. Remove probe from the storage cup containing the check standard, rinse with DI water, and gently blot dry.
7. Rinse the storage cup with DI water and dry it with paper towels or Kimwipes.
8. Repeat these steps with each calibration check standard.

Acceptable Ranges for Check Standards:

1. In-Situ – DO Check (Zero DO standard)

   The instrument must read 0 to 0.5 mg/l DO. If the instrument reads above 0.5 mg/l or reads a negative value, it will be necessary to recalibrate the instrument for both 100% and 0% saturation and check again. If this is unsuccessful, use a new Zero DO standard with a different lot number. If these measures are still unsuccessful, consult the manufacturer/vendor. It may be necessary to replace the RDO cap on the Smartroll or replace the unit.

   If the afternoon calibration check is not within the acceptable range then the data collected using the instrument that day must be qualified in its use as described above under Calibration Acceptance Criteria.

2. In Situ - pH Calibration Check (pH 7 standard)

   The value must be pH 7 +/-5% (pH 6.65-7.35). If the calibration check failed, recalibrate the instrument using fresh standards, with different lot numbers from those used in the initial calibration, for all three values and check it again. If re-calibration fails, clean the pH probe, recalibrate and check the calibration again. If the calibration check fails again, consult the manufacturer/vendor or replace the unit.

   If the afternoon calibration check is not within the acceptable range then the data collected using the instrument that day must be qualified in its use as described above under Calibration Acceptance Criteria.

3. In-Situ - Specific Conductance Check (1413 µS/cm standard)

   When the 718 µS/cm standard is used to calibrate, 5% of the 1413 µS/cm solution is 1342-1484 µS/cm. When the 1413 µS/cm standard is used to calibrate, 5% of the 718 µS/cm solution is 682-754 µS/cm.

   If the applicable range is not met, check the calibration using a fresh solution with a different lot number from that used in the initial calibration check. Then, if the range is not met, recalibrate using a fresh calibration solution with a different lot number and check again.

If this range is still not met, calibrate using the second calibration solution and repeat the process above. If this is not successful, consult the manufacturer/vendor or replace the unit.

For example: if the 718 µS/cm standard is used to calibrate, then the 1413 µS/cm solution is used to check the calibration. If the 1413 µS/cm solution is not within the acceptable range of 1342-1484 µS/cm, then check the calibration with a fresh 1413 µS/cm solution with a different lot number. If this range is still not met, recalibrate the instrument using a new 718 µS/cm standard from a different lot number and check again.

If the acceptable range is still not met, calibrate the instrument using the 1413 µS/cm standard and check the calibration with a 718 µS/cm solution. If the 718 µS/cm solution is not within the acceptable range of 682-754 µS/cm, then check the calibration with another 718 µS/cm solution with a different lot number. If this range is still not met, recalibrate the instrument using a new 1413 µS/cm standard from a different lot number and check again.

If that does not solve the problem, consult the manufacturer/vendor or replace the unit.

If the afternoon calibration check is not within the acceptable range then the data collected using the instrument that day must be qualified in its use as described above under Calibration Acceptance Criteria.

4. <u>In-Situ ORP Calibration Check (Zobell solution)</u>

The Smartroll automatically corrects the ORP solution according to the temperature of the solution.

Wait until the ORP and temperature readings have stabilized. Read the ORP on the instrument. Record the check value on the Calibration Log**,** and compare the value to the ORP value of the standard at the time of calibration. The instrument must be +/- 5% of the mV value for the current temperature. See the chart below for the check range. If it is not within +/- 5%, recalibrate using a new Zobell solution. If the re-calibration is not successful, consult the manufacturer/vendor or replace the unit. For the afternoon calibration check, the instrument must be within +/- 5% of the mV value for the current temperature or the data collected using the instrument that day must be qualified in its use as described above under Calibration Acceptance Criteria.

Zobell Solution mV Values Based on Temperature for ORP Calibration
Calibration Check Range Values (+/- 5%)
(Round off temperature to whole number, e.g., 23.5 ºC rounds up to 24 ºC)

Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%	Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%	Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%
-3	**267**	254-280	10	**251**	238-264	23	**234**	222-246
-2	**266**	253-279	11	**249**	237-261	24	**232**	220-244
-1	**265**	252-278	12	**248**	236-260	25	**231**	219-243
0	**264**	251-277	13	**247**	235-259	26	**230**	219-242
1	**262**	249-275	14	**245**	233-257	27	**228**	217-239
2	**261**	248-274	15	**244**	232-256	28	**227**	216-238
3	**260**	247-273	16	**243**	231-255	29	**226**	215-237
4	**258**	245-271	17	**241**	229-253	30	**225**	214-236
5	**257**	244-270	18	**240**	228-252	31	**223**	212-234
6	**256**	243-269	19	**239**	227-251	32	**222**	211-233
7	**254**	241-267	20	**238**	226-250	33	**221**	210-232
8	**253**	240-266	21	**236**	224-248	34	**219**	208-230
9	**252**	239-265	22	**235**	223-247	35	**218**	207-229

TURBIDITY CALIBRATION PROCEDURES

The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference suspension. A turbidimeter is a nephelometer with a visible light source for illuminating the sample and one or more photo-electric detectors placed ninety degrees to the path of the light source. All turbidity vials must be free of scratches. If a vial is scratched, the vial must be replaced.

The GZA low flow procedure requires that the turbidity meter shall have a calibration range from 0.00 to 800NTUs.

Condensation (fogging) of Turbidity Vial:

Condensation may occur on the outside of the sample cell when measuring a cold sample in a warm, humid environment. Condensation interferes with turbidity measurement, so all moisture must be thoroughly wiped off the sample cell before measurement. If fogging recurs, let the sample warm slightly by standing at ambient temperature or immersing in a container of ambient temperature water for a short period. After warming, gently invert the sample cell to thoroughly mix the contents before measurement.

This procedure is based on the use of the Hach 2100P or the 2100Q turbidimeter and the commercially available StablCal® Formazin Primary Turbidity Standards appropriate for each meter.

A - Hach 2100Q Turbidity Meter Calibration/Calibration Check Procedures

1. Use the commercially available StablCal® Formazin Primary Turbidity Standards: 20, 100 and 800 NTUs and the 10 NTU Verification Standard.

2. Before performing the calibration procedure, make sure the cells containing the standards are not scratched. If a cell is scratched, the standard must be replaced.

3. Allow the calibration standards to equilibrate at the ambient temperature.

4. Turn on the meter.

5. Push the **CALIBRATION** key to enter the Calibration mode.

   The Calibration key is the graph symbol with 2 points in the lower left-hand side.  The screen shows the three standards (20, 100 & 800 NTUs). The 20 NTU standard is shown bolded with a box around it indicating that is the first standard to be calibrated.

6. Gently invert the standards to thoroughly mix the contents. (**DO NOT SHAKE**)

7. Wipe the standards with a soft, lint free cloth or Kimwipe to make sure the outside surfaces are dry and free from fingerprints and dust.

8. Insert the first standard, 20 NTU, into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment and close the lid firmly. Note: the standard to be inserted is highlighted on the display screen.

9. Press **READ** (right hand key)**.** The display shows Stabilizing and then shows the results accompanied by an audio beep. The display will automatically request the next standard by highlighting it and darkening the first standard. Remove the 20 NTU standard from the compartment.

10. Insert the second, 100 NTU, standard into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment and close the lid firmly.

11. Press **READ.** The display shows Stabilizing and then shows the results accompanied by an audio beep. The display will automatically request the next standard by highlighting it and darkening the previous standards. Remove the 100 NTU standard from the compartment.

12. Insert the third and last, 800 NTU, standard into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment and close the lid firmly.

13. Press **READ.** The display shows "Stabilizing" and then shows the results accompanied by an audio beep. Remove the 800 NTU standard from the compartment.

14. Push **DONE** to complete a 3-point calibration and review the calibration details (values of the three standards).

15. Push **STORE** to save the results.

16. After a calibration is complete, the meter automatically goes into the Verify Cal mode.

17. Insert the 10 NTU Verification Standard into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment and close the lid firmly.

18. Push **READ** (right hand key). The display shows Stabilizing and then shows the results and the tolerance range. The calibration check must be +/- 10% (9.0- 11.0 NTUs).

19. Push **DONE** to return to the reading display.

20. If the calibration verification (Cal Check) is not within the +/- 10% range, repeat the calibration verification. If that fails, recalibrate using all standards. If re-calibration is unsuccessful, use new standards, consult the manufacture/vendor or replace the unit.

OVERNIGHT STORAGE OF THE YSI AND IN-SITU INSTRUMENTS

Check with the vendor for the appropriate overnight storage of the probes. Some manufacturers/venders may recommend storing the multiparameter probes overnight in a storage cup filled with pH 4 solution. If so, fill the storage cup with pH 4 solution, place the probes into the storage cup and seal tightly.

RECORDS AND DOCUMENTATION

All calibration information must be documented on the attached Calibration Log, including the instrument manufacturer, model number and identification number; standards used to calibrate the instruments, including source, lot numbers and expiration dates; date; personnel; the instrument readings; barometer reading; DO membrane inspection (if applicable); changed DO membrane and solution/RDO cap, etc. Each daily Calibration Log shall be dated and signed by the user.

SPECIAL NOTES

None.

APPLICABLE STANDARDS AND REFERENCES

USEPA, Region 1, Standard Operating Procedure Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity), EPA SOP# EQASOP-FieldCalibrat3, Revision Number 3, March 23, 2017.

Instruction manuals for the YSI Models 600XL Multiparameter Meter & Sonde, In-Situ smarTROLL Multiparameter Meter and Hach Turbidity Meters.

ATTACHMENT

Calibration/Maintenance Log

INSTRUMENT CALIBRATION LOG

Date: **Time:** | Field Personnel (print):

Meter: (circle one) YSI: Models 600XL or XLM, 6820 **QED:** Model MP 20 **Multimeter Serial Number (**Sonde & Meter**):**

Beginning of Day Instrument Calibration

Multimeter Calibration	Value of Standard	Check as Completed	Lot #	Expiration Date	Comments
DO (% saturation)	100%				Allow time for stabilization per manufacture
DO mg/L reading					Record these values immediately after calibration
DO Temp. (˚C) reading					
pH 1st Standard	7				
2nd Standard	4				
3rd Standard	10				
Specific Conductance **(µS/cm)**	1,413				One standard is used to calibrate, second one to check (1,413 µS/cm and 718 µS/cm standards)
ORP using Zobell Solution					See Chart on Page 2 for ORP Zobell Solution mV Value Based on Current Temperature
Zobell Solution _____ ˚C					

Additional Information for Dissolved Oxygen Calibration

Barometric Pressure Check: NWS Barometer Location: _____ Personnel _____Date: _____

NWS Pressure: _____ mm Hg Original Hand-held Barometer Pressure _____ mm Hg Adjusted to NWS Pressure (circle one) YES or NO

Barometric Pressure of Meter or Hand Held Barometer : _____ mm Hg [BP inches _____ x 25.4 + BP _____ mm Hg]

Dissolved Oxygen Charge (YSI Meters): _____ (Acceptable Range: 25 to 75) You MUST change the membrane if charge is out of range.

Inspected DO membrane for nicks or bubbles (check as completed) _____ Personnel:_____

Changed Dissolved Oxygen Membrane and Electrolyte Solution (circle one) **YES or NO**

HACH 2100P or 2100Q * Turbidimeter Calibration	Value of Standard	Check as Completed	Lot #	Expiration Date	Comments
Turbidity 1st Standard (blank)	<0.1 NTU				Calibrate w/ StablCal® Formazin Primary Turbidity Standards
2nd Standard	20 NTU				
3rd Standard	100 NTU				
4th Standard	800 NTU				

HACH Serial Number: **Rental Company:**

*** Circle appropriate model. NOTE: the 2100Q does not have a <0.1 standard, record N/A (not applicable) in the <0.1 standard boxes as appropriate.**

Post Calibration Check

Date: **Time:** | **Personnel:**

Calibration Check	Value of Standard	Check Results	Acceptable Range	Within Range (yes/no)	Lot #	Expiration Date	Comments
Zero DO check (mg/l)	0		0 to 0.5 mg/L				
pH 7 check	7		+/- 5%				Range 6.7 - 7.3 pH
Specific Conductance **(µS/cm)** Second standard used for check	718		+/- 5%				Range 682 - 754 µS/cm (718) **or** Range 1,342 - 1,484 µS/cm (1413)
ORP check - Zobell (mV)			+/- 5%				See Chart on Page 2 for ORP Zobell Solution mV Value Based on Current Temperature
Zobell Solution _____ ˚C							
Turbidity Standard (NTU) **2100P**	20		+/- 5%				Range 19.0 - 21.0 NTU (2100P)
Turbidity Standard (NTU) **2100Q**	10		+/- 10%				Range 9.0 - 11.0 NTU (2100Q)

Notes: 1) NWS = National Weather Service
2) If the post calibration check is not within the acceptable range the meter must be recalibrated.
3) All calibration checks must be made in the run mode, not the calibration mode.
4) If the lot numbers and expiration dates are the same as the initial calibration place a check mark ✓ in the appropriate box.
5) Either standard (718 or 1,413 µS/cm) maybe used to calibrate specific conductance, the second standard is used to check it.
6) Record N/A (Not Applicable) in the boxes for the turbidity meter that was not used.

Calibration & Post Calibration Check Performed by: _____(Print)_____ (Sign)

END OF DAY INSTRUMENT CALIBRATION CHECK

Calibration Check	Value of Standard	Check Results	Acceptable Range	Within Range (yes/no)	Lot #	Expiration Date	Comments
Date: Time:		**Personnel:**					
Zero DO check (mg/l)	**0**		**0 to 0.5 mg/L**				
pH 7 check	**7**		**+/- 5%**				**Range 6.7 - 7.3 pH**
Specific Conductance **(µS/cm)** Second standard used for check	**718**		**+/- 5%**				**Range 682 - 754 µS/cm (718) or Range 1,342 - 1,484 µS/cm (1,413)**
ORP check - Zobell (mV)			**+/- 5%**				**See Chart below for ORP Zobell Solution mV Value Based on Current Temperature**
Zobell Solution _____ ˚C							
Turbidity Standard (NTU) **2100P**[5]	**20**		**+/- 5%**				**Range 19.0 - 21.0 NTU (2100P)**
Turbidity Standard (NTU) **2100Q**[5]	**10**		**+/- 10%**				**Range 9.0 - 11.0 NTU (2100Q)**

Notes:

1) If the end of the day calibration check is not within the acceptable range the data collected that day for that parameter shall be qualified in its use.
2) All calibration checks must be made in the run mode, not the calibration mode.
3) If the lot numbers and expiration dates are the same as the initial calibration place a check mark ✓ in the appropriate box.
4) Either standard (718 or 1,413 µS/cm) maybe used to calibrate specific conductance, the second standard is used to check it.
5) Record N/A (Not Applicable) in the boxes for the turbidity meter that was not used.

Weather Conditions:
If the calibration/calibration check was performed off-site (e.g., in the office, etc.) due to weather conditions, check (√) here:_____
Was prior approval given by the Project Manager? (Circle one) Yes or No

Where off-site was the calibration/calibration check performed? _____

Calibration Check by _____ _____
Print Name Signature

List wells sampled using this equipment on this day <u>if data needs to be qualified</u>. _____

_____ _____

_____ _____

_____ _____

Zobell Solution mV Value Based on Temperature for ORP Calibration
Calibration Check Range Values (+/- 5%)
Round off temperature to whole number (e.g. 23.5 ºC rounds up to 24 ºC)

Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%	Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%	Temp. ºC	ORP Zobell Solution mV Value	Calibration Check Range Values +/- 5%
-3	**267**	254-280	10	**251**	238-264	23	**234**	222-246
-2	**266**	253-279	11	**249**	237-261	24	**232**	220-244
-1	**265**	252-278	12	**248**	236-260	25	**231**	219-243
0	**264**	251-277	13	**247**	235-259	26	**230**	219-242
1	**262**	249-275	14	**245**	233-257	27	**228**	217-239
2	**261**	248-274	15	**244**	232-256	28	**227**	216-238
3	**260**	247-273	16	**243**	231-255	29	**226**	215-237
4	**258**	245-271	17	**241**	229-253	30	**225**	214-236
5	**257**	244-270	18	**240**	228-252	31	**223**	212-234
6	**256**	243-269	19	**239**	227-251	32	**222**	211-233
7	**254**	241-267	20	**238**	226-250	33	**221**	210-232
8	**253**	240-266	21	**236**	224-248	34	**219**	208-230
9	**252**	239-265	22	**235**	223-247	35	**218**	207-229

Equipment Rental Company:

Probe Pre-cleaned Certification Provided By: Name **Date:**

Temperature Calibration: Name: **Date:**

Manufactures Accuracy Range of Sensor (e.g., +/- 0.2˚C): **Temperature check results:**

SOP D-21:
Decontamination Procedure

PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a procedure for preventing, minimizing, or limiting cross-contamination of environmental samples. This procedure is intended to ensure that field equipment is properly and adequately decontaminated in order to preserve the integrity of data collected with that equipment in the field, as well as to protect staff working with the equipment from exposure to contaminants.

This SOP focuses on small equipment decontamination (e.g., pumps, water level meters, hand augers, stainless steel spoons and mixing bowls for sediments).

Decontamination consists of physically removing contaminants or changing their chemical nature to innocuous substances. How extensive decontamination must be depends on a number of factors, the most important being the type of contaminants involved. The more harmful the contaminant, the more extensive and thorough decontamination must be. Less harmful contaminants may require less decontamination.

Decontamination is an essential part of a successful field operation as it: prolongs the usable life of the equipment; lessens the potential for cross-contamination of samples; prevents the mixing of incompatible substances; and reduces the likelihood of contamination leaving the site and threatening other areas with contamination.

In addition to this SOP, personnel should also review the manufacturer's user manual for any equipment specific recommended decontamination procedures.

EQUIPMENT AND MATERIALS

The following is a list of equipment and material commonly used for decontamination:

- An approved site-specific Health and Safety Plan.
- Appropriate personal protective equipment (e.g., safety glasses, appropriate chemically resistant gloves, boots);
- Site-specific QAPP.
- Non-phosphate detergent.
- Selected solvent rinses (e.g., pesticide-grade isopropanol, hexane).
- Tap water.
- Laboratory-grade deionized (DI) water.
- Long and short handled brushes and bottle brushes.
- Drop cloth/plastic/polyethylene sheeting.
- Paper towels.
- Plastic, galvanized or stainless steel tubs or buckets, as appropriate.
- Spray bottles and/or pressurized sprayers.
- Aluminum foil and/or re-sealable plastic bags.

- Appropriate containers for decontamination waste products.

GUIDELINES

All field activities must be carried out in accordance with a site-specific Health and Safety Plan. All decontamination procedures should be completed with an appropriate level of personnel protection: at a minimum equal to the level the fieldwork was completed in; no less than level D which includes safety glasses, chemically resistant gloves, boots, etc. Some solvents used in decontamination may require more stringent protection levels than used for fieldwork.

Safety Data Sheets (SDS) are required to be on site when using decontamination acid and/or solvent solutions.

Each item used for the decontamination of equipment may also become contaminated and must be appropriately handled, stored, and either decontaminated itself or disposed of. Certain items that become grossly contaminated and cannot be practically decontaminated (e.g., small tools and tools with wooden handles) should be disposed of properly. In some instances it is more practical and sensible to dispose of these items properly than to attempt decontamination. Such decisions will be made by the project manager.

GENERAL PROCEDURE FOR SMALL EQUIPMENT DECONTAMINATION

The decontamination procedure is summarized as follows:

1. Disassemble any items that might trap contaminants internally before washing. Do not reassemble until decontamination is complete.

2. Remove gross contamination from the equipment by brushing and then rinsing with tap water.

3. Wash the equipment with a non-phosphate detergent* and tap water, as required.

4. Rinse with tap water.

5. Rinse with the appropriate solvents**, as required.

6. An additional step of wiping the equipment with appropriate solvent (e.g., hexane) saturated paper towels may be necessary if the equipment made contact with a light non-aqueous phase liquid (LNAPL) to assist in its removal.

7. Rinse the equipment with DI water between solvent rinses, with a final rinse of DI water.

8. Air dry and secure clean equipment.

***Non-Phosphate Detergent**

In some cases, it may not be necessary to wash the equipment with a non-phosphate detergent if it can be demonstrated that washing the equipment with tap water alone (then rinsing with DI water) is sufficient.

**** Solvents**

In some instances, an additional wash with a solvent, such as isopropyl alcohol or hexane, may be required depending upon the contaminant. In other cases, it also may be necessary to carefully wipe the equipment down (inside/outside, as appropriate) with a paper towel saturated with the solvent. A solvent wash/wipe may be necessary in the case of sampling in high levels of contamination, or when sampling particularly difficult to clean contamination such as LNAPL or coal tar. The need for a solvent wash/wipe will be determined on a site-by-site basis by project manager prior to the sampling event. Lab personnel can assist in determining an appropriate solvent.

SPECIAL NOTES

The decontamination procedure for water level meters and oil/interface probes shall include the probes and, at a minimum, the length of tape used in that well.

With instruments such as multiparameter meters and turbidity meters, the probes, flow through cells and turbidity vials shall be thoroughly rinsed with DI water. If appropriate, some of these items may be washed with non-phosphate detergent and tap water, rinsed with tap water and finally rinsed with DI water. Sensitive equipment that is not waterproof should be wiped down with a damp cloth. Great care shall be taken not to damage the instrument. Refer to the manufacture manuals for specific guidance.

ALTERNATIVES

Decontamination is, by its nature, an arduous and painstaking task which is often better to avoid. Avoid decontamination procedures whenever feasible by implementing alternative plans of action such as:

- Dedicating specific equipment to a well (e.g., bladder pumps or bailers) when economically feasible;
- Using disposable equipment when applicable; and
- Wrapping monitoring equipment in plastic bags (or other materials) to protect from contamination. It is important to keep monitoring equipment such as a photoionization detectors (PID) from contacting soil or liquids at hazardous substance sites. By eliminating contact with contamination and/or using disposable equipment, decontamination of equipment may be avoided. Develop a method of wrapping/bagging these instruments in polyethylene sheeting/bags so that contact with contamination is minimized but the performance of the instrument is not adversely affected (e.g., the knobs made inaccessible, the meter covered).

APPLICABLE STANDARDS AND REFERENCES

ASTM D 5088 – 15a. Standard Practice for Decontamination of Field Equipment Used at Waste Sites. American Society for Testing and Materials (ASTM), Pennsylvania
http://www.astm.org

US DHHS, 1985. Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities. U.S. Department of Health and Human Services, Washington, D.C.
https://www.osha.gov/Publications/complinks/OSHG-HazWaste/4agency.html

US EPA, 1984. Standard Operating Safety Guides. Office of Emergency and Remedial Response, Washington, D.C.

SOP D-26:
Sample Collection: PCB Wipe Samples

PURPOSE

This Standard Operating Procedure (SOP) provides a standardized method used by personnel to collect samples for analysis of wipe samples for polychlorinated biphenyls (PCBs).

EQUIPMENT AND MATERIALS

The following list is not exhaustive, but provides a listing of the minimum equipment required and supplies:

- Quality Assurance Project Plan (QAPP);
- Appropriate sample containers, pre-preserved as necessary;
- Loose ice and a sample cooler;
- Logbook, pencil/pen/sharpies;
- Field-data sheets, sample labels, chain-of-custody records and custody seals;
- Appropriate health and safety PPE and an approved site-specific Health and Safety Plan;
- Screw Top 4-oz jars with Teflon Lined Caps with hexane-soaked gauze pads (provided by laboratory);
- Metal Ruler/marker and/or disposable Sampling Template; and
- Forceps for removing the gauze pad from the vials.

PROCEDURES – WIPE SAMPLING FOR PCBS

If the surface being sampled is smooth and impervious (e.g., unpainted metal surfaces), a wipe sample can be collected to determine if the surface is contaminated with PCBs.

The following procedure shall be followed for the collection of wipe samples for PCBs.

- Identify and mark the exact location where the 100-square-centimeter (cm^2) sample will be taken. The sample location may be marked or framed by a template.

- With gloved hands, remove the cap from the sampling jar. With the forceps, remove the gauze from the sampling jar.

- Immediately begin applying the gauze using a gloved hand and, applying pressure, wipe the marked area completely twice, from left to right and then from top to bottom.

- Wiping proceeds from left to right in rows from the top to the bottom of the framed sampling area. Fold the wipe in half with the dirty side folded inward. The sampling area is wiped again with the same uniform pressure in columns from the top to the bottom from the left side to the right side of the entire framed area. It is not critical whether wiping starts at the top left or with rows first and then columns. The objective is to systematically, thoroughly, and consistently wipe the entire framed area twice, each time from a different direction and orientation.

- Fold the gauze (sampled side inward) and place it to the sample jar.

- Cap the sample jar.

RECORDS AND DOCUMENTATION

Information should be recorded in a bound field notebook, sample collection sheets, or field tablet.

A field note book/field log should be maintained during sampling activities with the following information, at a minimum:

- Site and location of the sample extraction;

- Date on each page;
- Exact times of sampling events or visual observations;
- Types of samples collected and sample identification numbers;
- Number of samples collected;
- Specific description of sample locations;
- Description of sampling methods;
- Field observations; and
- Names of all field personnel.

SPECIAL NOTES

None.

APPLICABLE STANDARDS AND REFERENCES

United States Environmental Protection Agency, Region 1, Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs), May 2011, Revision 4.

"Polychlorinated Biphenyls (PCBs) - How to Test for PCBs and Characterize Suspect Materials," United States Environmental Protection Agency official website, https://www.epa.gov/pcbs/how-test-pcbs-and-characterize-suspect-materials.

SOP D-27:
Soil Sampling Procedure

PURPOSE

This Standard Operating Procedure (SOP) applies to the collection of surface and subsurface soil samples for laboratory analysis.

All non-dedicated sampling equipment shall be decontaminated prior to use and between samples. Sample collection activities shall generally proceed progressively from the suspected least contaminated area to the suspected most contaminated area when possible. Stage all equipment and supplies on plastic sheeting, or equivalent, to prevent contact with potentially contaminated surfaces.

A variety of soil sampling tools, typically made of stainless steel, are available for collection of soil samples (e.g., hand augers, split spoons, coring devices, scoops, spoons, etc.). Boreholes for subsurface soil samples may be advanced by hand boring devices (hand augers), portable powered augers, drilling rig, or hammering equipment. This procedure primarily references hand augers but is applicable to other soil sampling equipment.

EQUIPMENT AND MATERIALS

The following equipment is typically used in collecting soil samples:

- Appropriate health and safety gear and an approved site-specific Health & Safety Plan.
- Site-Specific QAPP.
- EnCoreTM samplers or disposable syringes with the tips cut off for collecting VOC samples directly from the sampling device. These are to be used once and then disposed of. Be sure to have one for each VOC sample and duplicate, plus extras.
- Stainless steel scoops, bowls, spoons.
- Hand augers, as appropriate.
- Sample containers, preserved as necessary, cooler and loose ice.
- Re-sealable plastic bags to protect and store samples;
- Field worksheets, sample labels, chain of custody forms, or field tablet;
- Logbook, pencil/pen/sharpies.
- The manufacturer's instruction manuals for all equipment, if applicable;
- Decontamination supplies/equipment, including laboratory-grade deionized water.
- Paper towels.
- Flagging to mark locations.

GENERAL PROCEDURE FOR COLLECTION OF SOIL SAMPLES

Carefully clear away all surface debris (leaves, twigs, etc.) for a 1-foot radius around the sampling location. Collection of plant or foreign material that is not part of the sample should be avoided.

For surface soil samples (e.g., 0-6", 0-12", etc.): using a decontaminated stainless-steel hand auger or other soil sampling device, auger or core into the material which is being sampled to the specified depth, retrieve the sample, collect VOC samples directly from the sampling device, and place remaining sample in a stainless steel or glass pan. Continue to collect additional soil from areas adjacent to the original sample location to ensure staying within the required depth until the appropriate volume of soil is obtained. When collecting duplicates, collect enough sample material in the stainless steel mixing bowl for both the sample and the duplicate.

For subsurface soil samples: Using a decontaminated hand auger or other boring or drilling device, advance the borehole to the appropriate sampling depth. Prior to collecting the sample, remove and/or minimize cuttings/cavings from the borehole to avoid collection of material that is not from the target sampling interval. Then use a decontaminated hand auger or sampling device, such as a thin walled tube or split spoon sampler, to collect the sample. After retrieving the sampler, trim the upper portion of the sample to remove any cuttings or cavings that may be present with the sample, collect VOC samples directly from the sampling device, and place remaining sample in a stainless steel or glass pan. Continue to collect additional soil from areas adjacent to the original sample location to ensure staying within the required depth until the appropriate volume of soil is obtained. When collecting duplicates, collect enough sample material in the stainless steel mixing bowl for both the sample and the duplicate.

If using a backhoe, shovel or other equipment to remove soil from the excavation: use a stainless steel trowel to collect soil that has not come into contact with the tool used for excavation, collect VOC samples directly from the sampling device, and place remaining sample in a stainless steel or glass pan. Continue to collect additional soil from areas adjacent to the original sample location to ensure staying within the required depth until the appropriate volume of soil is obtained. When collecting duplicates, collect enough sample material in the stainless steel mixing bowl for both the sample and the duplicate.

Following VOC sample collection, mix and homogenize the remaining soil as described below. Fill and cap the remaining sample containers. Clean the exteriors of the containers to remove any potential residue. Place samples in re-sealable bags. VOC and other samples requiring cooling shall be placed within **loose** ice in a cooler.

With the exception of VOC samples, it is extremely important that the sample be mixed and homogenized as thoroughly as possible to ensure that the sample is representative of the sampled material. A common method of mixing is referred to as quartering. Using a decontaminated stainless steel trowel, the sample in the sample pan is divided into quarters. Each quarter is mixed, and then all quarters are mixed into the center of the pan. This procedure is followed several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion and occasionally turning the material. NOTE: If samples are predominantly moist and clayey, extra effort may be necessary to produce a homogenous mixture.

Record a general physical description of the sample such as color; basic makeup (sand, silt or clay); and whether or not there is a high degree of organic material, on the *Shallow Soil Sampling Log* or in the field note book. Note grain size unless a separate sample is collected for that purpose. Document the system of soil classification used.

If not previously done, use flagging to visually mark the location if possible.

Decontaminate equipment according to the Decontamination SOP. Disposable sampling equipment shall be discarded after completing the sampling task and not reused.

QUALITY ASSURANCE

Collect appropriate quality assurance samples as specified in the site-specific QAPP.

At least one duplicate sample should be collected. Collect enough sample material in the stainless steel mixing bowl for both the sample and the duplicate; thoroughly mix the soil to obtain a homogeneous sample; remove any leaves, twigs, rocks or other gross debris that may have been collected; fill a separate container for each analysis immediately following the actual field sample collection and cap the containers for both the sample and duplicate sample. Note that the VOC sample and duplicate shall be collected directly from the sampling device, prior to sample mixing.

Equipment blanks are collected to ensure that the equipment is clean and the decontamination procedure is adequate.

To collect an equipment blank after decontamination: Gently pour deionized water over all the equipment used to collect the soil sample. Collect the rinsate that flows off the equipment into the appropriate sample containers.

RECORDS AND DOCUMENTATION

In general, all data and sampling information will be documented. Specific reporting of these sampling events may include, but is not limited to, the following information as recorded on the *Shallow Soil Sampling Log* or field note book:

1. Samples collected.

2. Date and time of sample collection.

3. A general physical description of the sample using GZA's Modified Burmeister such as color; basic makeup (sand, silt or clay); grain size; and whether or not there is a high degree of organic material.

4. Tie-off measurements of the sampling location.

5. A general physical description of the sampling locations, as well as digital photographs of sampling locations including one or more of the larger surrounding area.

SPECIAL NOTES

None.

APPLICABLE STANDARDS AND REFERENCES

EPA Region I *Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls* (May 2011).

SOP D-28:
Concrete Sampling Procedure

PURPOSE

This Standard Operating Procedure (SOP) applies to the collection of concrete samples for contaminant analysis.

All non-dedicated sampling equipment shall be decontaminated prior to use and between samples. Sample collection activities shall generally proceed progressively from the suspected least contaminated area to the suspected most contaminated area when possible. Stage all equipment and supplies on plastic sheeting, or equivalent, to prevent contact with potentially contaminated surfaces.

NOTE: The collection of samples for polycyclic biphenyls (PCBs) analysis shall follow the guidelines written for concrete in the EPA Region I *Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls* (May 2011).

EQUIPMENT AND MATERIALS

The following equipment is typically used in collecting concrete samples:

- Appropriate health and safety gear and an approved site-specific Health & Safety Plan.
- Electric drill.
- ½-inch diameter drill bit.
- Stainless steel scoops, bowls, spoons.
- Sample containers, preserved as necessary, cooler and loose ice.
- Re-sealable plastic bags to protect and store samples;
- Field worksheets, sample labels, chain of custody forms, or field tablet;
- Logbook, pencil/pen/sharpies, calculator.
- The manufacturer's instruction manuals for all equipment, if applicable;
- Decontamination supplies/equipment, including laboratory-grade deionized water.
- Paper towels.

GENERAL PROCEDURE FOR COLLECTION OF CONCRETE SAMPLES

Carefully clear away all surface debris for a 1-foot radius around the sampling location. Collection of plant or foreign material that is not part of the sample should be avoided.

Concrete verification samples will be obtained utilizing an electric drill equipped with a ½-inch diameter drill bit and the resulting concrete dust will be collected via a stainless steel spatula. The drill bit and the spatula will be decontaminated between each core location consistent with 761.79(c)(2)(i) or (ii).

For surface concrete samples using an electric drill equipped with a decontaminated ½-inch diameter drill bit, drill into the concrete which is being sampled to the specified depth and retrieve the concrete dust via a stainless steel spatula. Place the sample in a stainless steel or glass pan. Continue to collect additional concrete dust from areas adjacent to the original sample location to ensure staying within the required depth until the appropriate volume of concrete dust is obtained. When collecting duplicates, collect enough sample material in the stainless steel mixing bowl for both the sample and the duplicate.

It is extremely important that the sample be mixed and homogenized as thoroughly as possible to ensure that the sample is representative of the sampled material. A common method of mixing is referred to as quartering. Using a decontaminated stainless steel trowel, the sample in the sample pan is divided into quarters. Each quarter is mixed, and then all quarters are mixed into the center of the pan. This procedure is followed several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion and occasionally turning the material.

Decontaminate equipment according to the Decontamination SOP. Disposable sampling equipment shall be discarded after completing the sampling task and not reused.

QUALITY ASSURANCE

Collect appropriate quality assurance samples as specified in the site-specific QAPP .

Equipment blanks are collected to ensure that the equipment is clean and the decontamination procedure is adequate.

To collect an equipment blank after decontamination: Gently pour deionized water over all the equipment used to collect the sample. Collect the rinsate that flows off the equipment into the appropriate sample containers.

RECORDS AND DOCUMENTATION

Specific reporting of these sampling events may include, but is not limited to, the following information as recorded on the Concrete Sampling Log or field note book:

1. Samples collected.

2. Date and time of sample collection.

3. Tie-off measurements of the sampling location.

4. A general physical description of the sampling locations, as well as digital photographs of sampling locations including one or more of the larger surrounding area.

SPECIAL NOTES

None.

APPLICABLE STANDARDS AND REFERENCES

EPA Region I *Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls* (May 2011).

SOP D-43:
Low Flow Groundwater Purging and Sampling

SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) provides a general framework for collecting groundwater samples that are indicative of total mobile organic and inorganic loads (dissolved and colloidal sized fractions) transported through the subsurface under ambient flow conditions with minimal physical and chemical alterations from sampling operations. This SOP is considered generally consistent with the United States Environmental Protection Agency (EPA), Region 1 SOP # EQASOP-GW-4, *Low Stress (low flow) Purging and Sampling Procedure For the Collection of Ground Water Samples From Monitoring Wells*.

The intent of this SOP is to enhance the quality of the samples being measured and to help ensure that the project-specific data quality objectives (DQOs) are met when low flow conditions are required. In this procedure, the user is required to monitor the rate at which the water level in the well drops, generally referred to herein as the drawdown rate. The procedure includes a reference table based on calculations that improve the potential that at least 90% of the water being pumped out of the well (i.e., the discharge water) was in contact with the formation and that no more than 10% of the discharge water is influenced by the stagnant water in the well riser. Total purge volume, stabilization of indicator field parameters [pH, turbidity, specific conductance, temperature, dissolved oxygen (DO), and oxidation-reduction potential (ORP)], and drawdown rate are used to indicate when conditions are suitable for sample collection.

In low permeability formations or poorly installed monitoring wells, it may not be possible to collect groundwater samples using the standard low flow procedure. Under such conditions a modified sampling procedure is employed. The conditions that will trigger switching from the standard low flow procedure to the modified procedure are:

1. The well is under artesian conditions.
2. The water level is within the well screen interval.
3. The pump flow rate needed to achieve an acceptable drawdown rate is below 50 milliliters (ml) per minute, in which case the well is considered to have insufficient recharge.
4. The water level falls below the top of the well screen interval while purging.

In long term monitoring programs, if the low flow procedure has been attempted unsuccessfully at a well for two consecutive rounds, it may be possible (with the project manager's approval) to proceed to the "Modified Sampling Procedure" without further attempt to use the standard low flow procedure.

In general, there is a two-hour time limit for each well.

PROCEDURE REQUIREMENTS

All measurements of field parameters must be obtained using a "flow-through cell", except for turbidity. Turbidity must be collected at a point before the flow-through cell and measured with an instrument that is separate from the flow-through cell apparatus. A three-way stop cock (or "T" connection) attached to the tubing before the flow-through cell will be used to collect the turbidity samples. Transparent flow-through cells with a cell capacity of 250 ml or less are required. The transparency allows field personnel to watch for air bubbles and particulate build-up within the flow-through cell: These conditions may affect indicator field parameter values measured within the cell. The flow-through cell must be designed and used in a way that prevents air bubble entrapment in the cell. Placing the flow-through cell at an approximate 45-degree (º) angle with the outlet port facing upward can help remove bubbles from the flow-through cell. The flow-through cell must remain free of any air bubbles at all times. Otherwise, the monitoring probes may act erratically. When the pump is turned off, water in the flow-through cell must not drain out. Monitoring probes must be submerged in water at all times during the collection of field parameter data.

A small volume flow-through cell (250 ml or less) facilitates rapid turnover of water in the cell between measurements of the indicator field parameters. The pump's flow rate must be able to "turn over" at least one flow-through cell volume between measurements. For example: The monitoring frequency for a 250 ml flow-through cell with a flow rate of 50 ml/minute would be every five minutes. **Note: The indicator field parameters shall be measured at a minimum frequency of five-minute intervals.**

HEALTH AND SAFETY

When working on-site, comply with all applicable OSHA requirements and the site's health/safety procedures. All appropriate personal protection clothing and equipment are to be worn. Some samples may contain biological and chemical hazards. These samples shall be handled with suitable protection for skin, eyes, etc.

PRECAUTIONS

Consider the following precautions when planning to collect groundwater samples if the conditions listed below are expected to occur.

If the groundwater degasses during purging of the monitoring well, dissolved gases and VOCs will be lost. When this happens, the groundwater data for dissolved gases (e.g., methane, ethane, ethene, dissolved oxygen) and VOCs will need to be qualified. Some conditions that can promote degassing are the use of a vacuum pump (e.g., peristaltic pumps), changes in aperture along the sampling tubing, and squeezing/pinching the pump's tubing (e.g., constricting the flow) which results in a pressure change. The observation of bubbles in the tubing is indicative of groundwater degassing.

When collecting the samples for dissolved gases and VOCs analyses, avoid aerating the groundwater in the pump's tubing. This can cause loss of the dissolved gases and VOCs in the groundwater. Having the pump's tubing completely filled prior to sampling will help avoid this problem.

Direct sunlight and hot ambient air temperatures may cause the groundwater in the tubing and flow-through cell to heat up. This may cause the groundwater to degas, which will result in loss of VOCs and dissolved gases. When sampling under these conditions, shade the equipment from the sunlight (e.g., umbrella, tent). If possible, sampling on hot days, or during the hottest time of the day, should be avoided. The tubing exiting the monitoring well should be kept as short as possible to avoid sunlight or ambient air heating up the groundwater in the tubing.

Condensation (fogging) of Turbidity Vial: Condensation may occur on the outside of the sample cell when measuring a cold sample in a warm, humid environment. Condensation interferes with turbidity measurement, so all moisture must be thoroughly wiped off the sample cell before measurement. If fogging recurs, let the sample warm slightly by standing at ambient temperature or immersing the sample cell in a container of ambient temperature water for a short period. After warming, gently invert the sample cell to thoroughly mix the contents before measurement.

EQUIPMENT AND MATERIALS

A. Appropriate health and safety gear.

B. Informational materials for sampling event
An approved site-specific Health and Safety Plan, site-specific QAPP, monitoring well construction data (e.g., well depth, inner casing diameter, screen interval), location maps, field data from prior sampling events, and the monitoring instrument's operation and maintenance manuals, shall be brought to the site.

C. PID instrument

Used if appropriate, to detect VOCs for health and safety purposes and provide qualitative field evaluations.

D. Well/site keys and spare locks.

E. Pumps:

Peristaltic pumps and adjustable rate submersible pumps (e.g., centrifugal, bladder) that are constructed of stainless steel are typically used with this procedure. Pumps capable of pumping at a low flow rate of 50 ml/minute may be required in wells with low recharge.

Peristaltic Pumps

An adjustable rate Geotech Peristaltic Pump Series II Variable Speed pump 300 + 600 RPM, or similar pump, with Easy Load Peristaltic Pump Heads (that allow a 50 ml/minute flow rate using thin walled tubing).

1. A thin walled tubing pump head is necessary in order to achieve a 50 ml/min flow rate during purging and the use of a thin walled tubing pump head is a requirement for sampling.
2. The use of piggybacked peristaltic pump heads is unacceptable as the arrangement may produce an uneven flow with potential to aerate the water and cause the loss of VOCs and dissolved gases.

Bladder Pumps:

Non-contact gas stainless steel bladder pump and stainless steel with polyethylene bladders or similar pump.

1. The size and capacity of the pump, and its placement in the well, shall be selected to maximize the filling of the pump's bladder. For proper operation, the bladder pump will need a minimum amount of water above the pump; consult the manufacturer for the recommended submergence. The pump's submergence requirements must be determined during the well drilling planning stage, because it may influence well construction and placement of dedicated pumps where water-level fluctuations are significant.
2. The control box shall have a manual control option.
3. For the collection of VOCs and dissolved gases:
   a. The bladder's capacity shall be large enough (greater than 70 milliliters) to fill a VOA vial in one pulse.
   b. The pump settings (refill and discharge rates) shall be set so that one pulse will deliver a water volume that is sufficient to fill a 40 ml VOA vial while allowing the discharge to flow gently down the inside of the container with minimal turbulence.

Centrifugal Submersible Pumps

Grundfos Redi-Flo2 submersible pump and control/converter box, or similar pump.

1. These pumps typically require a generator as a power source. **If a gasoline generator is used, it must be located downwind and at least 30 feet from the well** so that there is reduced potential for the exhaust fumes to contaminate the samples.
2. A stainless steel adapter may be necessary in order to use 1/4" inside diameter tubing.
3. If very low flow rates (i.e., 50 ml/minute) are required, it may be difficult to adjust the control boxes to maintain a laminar flow.

G. Power source

Portable power pack, twelve (12) volt, deep cycle marine battery, or other battery; nitrogen tank or air compressor; etc.; depending upon data quality objectives.

If a gasoline-powered source (e.g., generator) is used, it must be located downwind and at a safe distance from the well (at least 30 feet) so that there is reduced potential for the exhaust fumes to contaminate the samples.

H. Appropriate tubing:

Polyethylene tubing is typically used in the well. The use of one-quarter inch (1/4") inside diameter (ID) tubing is recommended. This will help ensure that the tubing remains liquid-filled when operating at very low pumping rates with centrifugal and peristaltic pumps. Three-eighths (3/8) inch ID tubing may also be used with project manager approval. Smaller diameter tubing (less than 1/4" ID) is not generally recommended. If sampling with a bladder pump, use the size tubing in this range that is recommended by the manufacturer.

Silicon tubing (pharmaceutical or surgical grade only) shall be used in the rotor head of peristaltic pumps (sized according to ID x outside diameter (OD) x wall thickness). It is preferable to use dedicated silicon tubing whenever possible. For sampling: Thin walled tubing #16 (1/8" x $^1/_4$" x 1/16") and/or thin walled tubing #14 (1/16" x 3/16" x 1/16") if necessary to reduce flow to 50 ml/min. For connections: thick walled tubing #15 (3/16" x 3/8" x 3/32") and possibly #17 (1/4 x 3/8 x 1/16). Either a tubing connector will be used to connect the pump rotor head tubing to the well tubing or silicon tubing shall be connected to the well tubing by placing the silicon tubing over the well tubing

(not inside the well tubing). Various sizes of silicon tubing shall be connected together by placing the end of the large tubing over the end of the smaller tubing. Make sure that tubing being connected together are sized appropriately so that the connections are snug and air bubbles do not form at the connections.

I. The water level measuring device

An electronic tape, pressure transducer, water level sounder/level indicator, etc., capable of measuring to one one-hundredth of a foot (0.01 ft.) accuracy shall be used. Data-recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include measurement checks with a water level tape at the start and end of each sampling event.

J. Interface probe

To be used to check on the presence of free phase liquids (LNAPL or DNAPL) before purging begins, as needed.

K. Multiparameter monitoring instruments

Multiparameter water quality meters (e.g., YSI 600XL) with a with built in barometer and a transparent 250 ml or less internal volume flow-through cell capable of measuring pH, ORP, DO/RDO, specific conductance and temperature are required. Record the equipment/instrument identification information (including manufacturer serial and model number) on the Calibration Log or appropriate form.

A barometer (used in the calibration of the DO probe) and the conversion formula (to convert the barometric pressure into the units of measure used by the DO probe) are needed unless the instrument already has a built-in temperature-compensated barometer.

The transparent cell allows easy detection of air bubbles and particulate buildup within the flow-through cell, which can interfere with the indicator field parameter values measured within the cell.

A small volume flow-through cell (250 ml or less) facilitates rapid turnover of water in the cell between measurements of the indicator field parameters. The pump's flow rate must be able to "turn over" at least one flow-through cell volume between measurements. For example: The monitoring frequency for a 250 ml flow-through cell with a flow rate of 50 ml/minute would be every five minutes. **Note: The indicator field parameters shall be measured at a minimum frequency of five-minute intervals.**

The flow-through cell must be designed and used in a way that prevents air bubble entrapment in the cell. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must be submerged in water at all times.

It is recommended to use a flow-through cell and monitoring probes from the same manufacturer and model to avoid incompatibility between the probes and flow-through cell.

The meter shall measure the following parameters to use with the low flow sampling technique:

- Temperature, degrees Celsius (°C)
- Specific Conductivity, micro Siemens per centimeter (µS/cm)
- DO/RDO, milligrams per liter (mg/l) with 100% saturation for calibration
- pH that calibrates using 3 standards (pH 4, 7 and 10)
- ORP, millivolts (mV)

A turbidity meter with a calibration range from 0.00 to 800 (or 1000) Nephelometric Turbidity Units (NTUs) is required. Turbidity samples are collected before the flow-through cell.

Appropriate calibration solutions are required for the multiparameter and turbidity meters, including: 100% water-saturated air chamber (small wet sponge or paper towel for DO 100% saturation calibration) and zero (0) mg/l solution for DO; Zobell solution for ORP; two different specific conductance standards, one high and one low, where

one standard is used to calibrate and the other standard is used to check the calibration (e.g., 1,413 µS/cm and 718 µS/cm); pH 4, 7, and 10 buffering solutions; and <0.1, 10, 20, 100, 800 NTUs standards, as appropriate for the selected turbidity meter. A minimum of two standards are needed to bracket the instrument measurement range for all parameters except ORP, which uses a Zobell solution as a standard.

A three-way stopcock (recommended) or a "T" connector coupled with a valve/clamp is connected between the pump's tubing and flow-through cell to divert sample flow to collect turbidity samples (e.g., Nalgene three-way stopcock with a plug bore of 4 mm [or 0.157 inch]: NNI No. 6470-0004, VWR catalog No. 59097-080). If using an in-line "T" connector, attach a short piece of tubing and a valve/clamp to the center branch of the "T" connector to serve as a sampling port for the turbidity samples. When a turbidity measurement is required, the valve/clamp is opened to allow the ground water flow to be diverted into a container. The valve/clamp is closed and the sample container is capped and then placed into the turbidity meter.

L. Flow measurement supplies

A graduated cylinder sized according to the flow rate (e.g., 100 ml or 250 ml graduated cylinder, measured in 10 ml increments) to accurately measure the flow in ml/minute.

A stopwatch to accurately measure the flow in ml/minute.

Large graduated bucket (e.g., five-gallon size) used to record the total volume of water purged from the well, marked in gallons and/or liters (milliliters where appropriate).

M. Record keeping supplies

Logbook(s) and other worksheets (e.g., field-data sheets, sample labels, chain-of-custody forms and seals, field worksheets, well purging forms, calibration logs), pens, sharpies, etc.

N. Sample containers, etc.

Sample containers preserved as necessary, provided by the laboratory. Sample labels, cooler and loose ice (not bagged). Re-sealable plastic bags and bubble wrap to protect and store samples. Clear tape to be placed over sample container labels before sampling in the event the labels are not water proof labels. Alternatively, use plastic water proof labels.

O. In-line filters

If filtered samples (e.g., dissolved metals) are required, use a one-time use in-line filter (transparent housing preferred). The filter size (0.45 micron is commonly used) is based on the sampling objective.

P. Decontamination supplies

Decontamination equipment and supplies in accordance with the Decontamination SOP in the site-specific QAPP, including: non-phosphate detergent, laboratory-grade deionized (DI) water, appropriate solvent such as isopropyl alcohol, plastic sheeting, appropriate size buckets and lids for containerization of liquids, if required, brushes and spray bottles. Note: Some non-phosphate detergents may contain 1,4-dioxane, which may be a concern.

Trash bags to containerize solid waste.

PRELIMINARY PROCEDURES INCLUDING WATER LEVEL MEASUREMENTS

1. Inspect the well for security (e.g., damage, evidence of tampering, missing lock) and record pertinent observations (include photographs as warranted). Note any physical changes to well condition, such as erosion or cracks in protective concrete pad, road box or standpipe. If a lock is found to be damaged, replace with a new lock. Wells shall be locked at all times when not being sampled; this ensures the integrity of the well, any samples collected and the chain of custody.

2. Remove well cap. Immediately measure VOCs at the rim of the well with a PID instrument if required in the site-specific QAPP or Health and Safety Plan; record the reading in the field logbook/worksheet.

3. Install sampling pump or tubing, if necessary. Lower equipment (e.g., pump, safety cable, tubing and electrical lines) slowly into the well so that the pump or tubing intake is located at the center of the saturated screened interval at a depth that will remain under water at all times based on the historical low groundwater, unless otherwise specified in the site-specific QAPP. The QAPP shall specify the sampling depth, or provide criteria for selection of intake depth for each well. Great care must be taken to minimize the disturbance of particulates that can greatly extend the purge time by increasing turbidity. If possible, install the pump the day before purging to allow particulates that were disturbed during pump insertion to settle.

   In general, keep the tubing intake at least 1 to 2 feet above the bottom of the well to minimize mobilization of particulates present in the bottom of the well. The exceptions to this include wells with 2-foot screen lengths and those wells that typically have less than 2 feet of saturated thickness and are not flowing under artesian conditions. For these wells, the intake needs to be at least 3⁄4-foot off the bottom of the well. If there is less than 3⁄4-foot of water, a sample is typically not collected.

   The suction-pump down-well tubing or the submersible pump's safety cable shall be secured to the well casing (or PVC stick-up) to minimize movement.

   Pump tubing lengths extending beyond the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating may cause the groundwater to degas, which is unacceptable for the collection of samples for VOC and dissolved gas analyses.

4. A synoptic water level measurement round is to be performed (in the shortest possible time, i.e., all in one day) before any purging and sampling activities begin. A synoptic water level measurement round will be necessary if potentiometric surface maps are to be constructed for the sampling event.

   If the well casing does not have an established reference point (usually a V-cut or indelible mark on the well casing), make one. Describe its location and record the date of the mark in the logbook (consider a photographic record as well). All water level measurements must be recorded relative to this reference point (and the elevation of this point should be determined using techniques that are appropriate to the site's DQOs).

5. Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent measurement checks with an interface probe may not be necessary, unless analytical data or field analyses signal a worsening situation. This SOP cannot be used in the presence of LNAPLs or DNAPLs. If NAPLs are present, the project team must decide upon an alternative sampling method. All project modifications must be approved and documented prior to implementation.

6. The depth to the bottom of the monitoring well shall be measured at least once every five years in the sampling event just prior to the Five Year Review at all Superfund Sites and as required. It may not be possible to measure the depth of the well if dedicated bladder pumps are used. If that is the case, the depth of the well should be measured when the pump is removed for maintenance, if there is evidence of high turbidity, or as otherwise required in the site-specific QAPP.

7. Lay out a sheet of clean polyethylene to place the monitoring and sampling equipment upon, unless equipment is elevated above the ground (e.g., on a table or a bucket).
   Note: When using peristaltic pumps, it is highly recommended that the pump be placed on a table, or other surface, as close to the height of the well casing as possible to eliminate or reduce the buildup of air bubbles in the sample line between the peristaltic pump and the top of the well casing.

8. Check to determine if the well is under artesian conditions, if the water level is at, or below, the top of the screen.

9. Set up equipment. Be sure to tilt the flow-through cell so that the outflow connection is facing upward, in order to eliminate and prevent air bubble entrapment in the flow-through cell.
   If using a peristaltic pump, be sure the pump head is set at 300 RPM, not 600 RPM. Either size #16 (i.e., 1/8" ID x $^1/_4$" OD x 1/16" Wall) or #14 (i.e., 1/16" ID x 3/16" OD x 1/16" Wall) silicon tubing shall be used through the pump head.

10. If available, check flow rate, drawdown and pump setting information from previous sampling events for each well. Duplicate, to the extent practicable, the final settings from previous events. For wells that are routinely sampled, refer to the prior ***Low Flow Sampling Worksheets*** to determine the initial settings to reach stabilization of the water level as quickly as possible. This is only a guide and the sampler will need to "fine tune" the operating conditions, because the recharge rate of groundwater in a given well may vary from event to event.

    If using a peristaltic pump and a 50 ml/minute flow could not be reached or maintained with the #16 tubing, a section of #14 silicon tubing shall be connected to the end of the #16 tubing and the size #14 tubing shall be positioned into the pump head to reach a 50 ml/minute flow rate. The #14 tubing shall only be used where a 50 ml/minute flow rate cannot be maintained with the #16 tubing.

11. Be sure all sampling equipment is properly protected from the weather.

12. Purge Volume Requirement

The procedure includes a purge volume requirement (PVR) to ensure that a minimum volume of water has been removed from the well before sampling begins. The PVR is calculated just before sampling begins.
The purge volume requirement is one tubing volume. In other words:

PVR = (Total Tubing Length [ft.] x Unit Tubing Capacity [gal/ft. or ml/ft.])

Note: Include the length of tubing that is outside the well in the Total Tubing Length.

For convenience, the table below can be used to determine the appropriate volumes in gallons or milliliters. Refer to the *Purge Volume Calculations* section in this SOP for the formula used for determining the table values, if necessary.

Unit Tubing Capacity Values

Tubing Diameter (Inches)	1/4 (0.25) OD **(0.17 ID)***	3/8 (0.375) OD **(0.25 ID)***	1/2 (0.50) OD **(0.375 ID)***	5/8 (0.625) OD **(0.50 ID)***
Volume (gal/foot)	0.0012	0.0026	0.0057	0.0102
Volume (ml/foot)	4.5	9.7	21.7	38.6

* Calculations are based on the ID, not the OD.

WELL PURGING AND SAMPLING PROCEDURE

Purging and sampling wells in order of increasing chemical concentrations (known or anticipated) is preferred.

1. Carefully lower a water level indicator to the top of groundwater. Measure and record the water level (to 0.01 feet) before any disturbance to the well. Care shall be taken to minimize suspension of any particulates attached to the sides.

2. If the well is under artesian conditions or the water level is within the screen, refer to the "Modified Sampling Procedure" section for instruction on sampling the well . If the well is not under either of these conditions, then proceed to the next step.

3. Start the pump and allow the flow-through cell to fill.
   a. Try to match the final pumping rate used during previous sampling events. If no previous information is available, start the pump at its lowest speed setting (using the #16 silicone tubing if using a peristaltic pump) and slowly increase the speed until discharge occurs.
   b. From the time the pump starts purging until the time the samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged.
   c. The water flow during purging and sampling needs to be a laminar flow without air bubbles. If air bubbles are observed, they can usually be removed by elevating the affected section of tubing to allow the air to continue rising until discharged with the purge water. When using a peristaltic pump, any air captured in the tubing can usually be removed by elevating the discharge tube and the pump.
      Prevent sample tubing from crimping and avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This may cause the groundwater to degas, which can result in a loss of VOCs and dissolved gasses in the groundwater samples. All tubing needs to be maintained in an open condition.
   d. When using the bladder pump for VOC or dissolved gas samples, the pump settings (refill and discharge rates) shall be set so that one pulse will deliver a water volume that is sufficient to fill a 40 ml VOA vial.
   e. If excessive turbidity or floc is anticipated or encountered with the pump startup, divert the water through the three-way stopcock, as if you were taking a turbidity sample, until it clears in order to minimize particulate buildup in the flow-through cell (this is a judgment call made by the sampler). Make sure that the discharge water is going into the graduated bucket so that it will be included in the determination of the final purge volume.

3. Measure and record the water level in the well after 5 minutes of pumping. Record the drawdown on the worksheet, if using the YSI.

4. Assess the water level drawdown rate.

6. Adjust pump rates until there is little or no water level drawdown.

Pumping rates shall, if needed, be reduced to the minimum capabilities of the pump and tubing size to avoid or minimize drawdown and to ensure stabilization of monitoring parameters. **However, pumping rates shall not be less than 50 ml/minute**.

Concentrate on the flow rate and drawdown rate stabilization for the first 15 minutes, or so, of the well purging effort (e.g., first 3 to 4 water level readings at 5-minute intervals). In general, the drawdown rate is expected to be stabilized within the first 15 to 20 minutes after the purge water exits the flow-through cell and enters the bucket.

The following drawdown rate limits are acceptable with a flow rate of no less than 50 ml/minute:

Maximum Allowable Drawdown Over One 5-Minute Period	
Inside Diameter of Inner Casing (Inches)	**Drawdown Rate (Feet per 5 Minutes)**
3 or less	0.02
3.5 - 5	0.01
6	No drawdown allowed

These maximum allowable drawdown rates enhance the potential for acquiring at least 90% (and greater for smaller diameter wells within the range) fresh aquifer water and no more than 10% stagnant well water in the flow-through cell and samples.

When using a peristaltic pump, if the pump flow rate is greater than 50 ml/minute, the maximum allowable drawdown rate cannot be achieved, and the pump cannot be adjusted to a slower rate using the #16 silicon tubing, then the #14 tubing must be used through the pump head. Use the following procedure to change the tubing:

a. Stop the pump. Do not release the tubing from the pump head at this time, in order to maintain the vacuum that retains the water in the tubing.
b. Add a length of #14 tubing to the discharge end of the #16 tubing. The #14 tubing should be about the same length as the #16 tubing, 12-18 inches.

c. Crimp the #16 silicon tubing on the influent side of the pump head with one hand so that the water does not flow back down the tubing and into the well.
d. Release the pump head, move the #14 tubing into place and close the pump head.
e. Release the crimp from the #16 tubing. The point here is to not let the water flow back into the well when moving the #14 tubing into the pump head housing, as this may increase the turbidity and prolong sample time. This procedure may require two people.
f. Once the tubing is changed, start the pump again, adjust the rate until a flow rate of 50 ml/minute is achieved and maintained, and continue to assess drawdown rate (e.g., by collecting 3 to 4 water level readings at 5-minute intervals).

   In general, recording of the indicator field parameters (i.e., pH, turbidity, specific conductance, temperature, DO and ORP), although useful, is not mandatory during this **Initial Time Period** (i.e., 15 to 20 minutes with the #16 tubing, or 40 minutes if the tubing was changed to #14) when attempts are being made to adjust the flow rate and stabilize the drawdown rate.

   If the desired drawdown rate cannot be achieved by the end of the Initial Time Period, then begin collecting the indicator field parameters **at a minimum frequency of five-minute intervals**, if not already doing so. Make sure the purge water is still being collected in the graduated bucket as part of the total purge volume.

   When using the YSI, if minimum fluctuation of the indicator field parameters is observed during the Initial Time Period, then go ahead and begin recording indicator field parameter data, as it may help reduce the time spent purging.

   Make a notation on the field worksheet (when using the YSI) "NR" for "no reading" at times when only partial data is being collected (e.g., water level only) during the Initial Time Period while adjusting pump speed and stabilizing the drawdown rate.

   Measure the purge volume of a bladder pump cycle with a 250 ml, or smaller size, graduated cylinder and record on the worksheet.

7. Well Conditions
   a. The well will NOT be considered suitable for low flow sampling under the following conditions:
      1) The initial water level was above the top of the screen prior to starting the pump but the water level falls below the top of the well screen during purging.
      2) The water level continues to drop at the 2-hour time limit at a rate that is greater than the maximum allowable drawdown rate in the chart above, with the pump setting at the lowest level, including the use of the #14 silicon tubing when using a peristaltic pump.

Under these conditions, sampling may occur immediately if the total purge volume is greater than one tubing volume, and enough water remains above the tubing intake to collect the samples, as follows:

1) Collect one last set of field parameters before disconnecting the three-way stopcock and collecting the samples.
2) To collect the samples, refer to the Sample Collection steps below in Step 12- *Sample Collection*.
3) If the water level in the well drops to the tubing intake, discontinue sampling at that location and return once the well has sufficiently recharged to collect the remaining samples.

   If the well has to recharge overnight, refer to the *Modified Sampling Procedure* section in this SOP for instruction on sampling the well. One tubing volume of water must be removed immediately before resumption of sample collection the next day.

   If this type of problematic situation persists in a well (e.g., for a minimum of two consecutive sampling rounds), then the modified sampling procedure should be evaluated and approved for future sampling events at the well by the project manager if it's consistent with the site's DQOs or a new well should be installed.

b. If the well is not under any of the conditions in Step a, then continue to the next step (Field Parameter Monitoring).

8. Field Parameter Monitoring

In addition to the water level, drawdown, pumping rate and any adjustments, and if not already doing so, now begin recording the indicator field parameters (pH, turbidity, specific conductance, temperature, DO and ORP) at a frequency of five-minute intervals (or greater if using the YSI; e.g., every ten minutes until the indicator field parameters start to stabilize, then every five minutes. **Readings shall not be less than five minutes apart.**

a. When recording pH and DO data, round off data to one decimal place (i.e., nearest tenth). When DO is less than 0.5 mg/L, data should be recorded as "< 0.5" or "less than 0.5". When recording specific conductance, temperature, turbidity, and ORP data, record only whole numbers (round off to the nearest whole number). When turbidity data is less than 5 NTU, data should be recorded as "< 5" or "less than 5".

b. Periodically check the probes and the top of the flow-through cell for air bubbles and eliminate any that are found. Be sure to tilt the flow-through cell with the outflow connection facing upward to minimize, if not eliminate and prevent air bubbles.
c. Rinse the turbidity vial with DI water before collecting the first sample. Rinse with fresh purge water or DI water between readings to eliminate any sediment that may have collected on the bottom.
d. Condensation (fogging) of Turbidity Vial: Condensation may occur on the outside of the sample cell when measuring a cold sample in a warm, humid environment. Condensation interferes with turbidity measurement, so all moisture must be thoroughly wiped off of the sample cell before measurement. If fogging recurs, let the sample warm slightly at ambient temperature or immerse the sample cell in a container of ambient temperature water for a short period. After warming, wipe the sample cell dry and gently invert the sample cell to thoroughly mix the contents before measurement.
e. If the flow-through cell needs to be cleaned during purging operations, then continue pumping, while letting purge water discharge directly to the graduated bucket and disconnect the flow-through cell for cleaning; reconnect after cleaning and continue monitoring activities. Record start and stop times for cleaning and document with a brief description of cleaning activities.
f. The flow rate used to achieve a stable pumping level should remain constant while monitoring the indicator parameters for stabilization and while collecting the samples. However, it may be necessary to reduce the flow rate to collect volatile samples (e.g., VOCs, methane, ethane, ethene, carbon dioxide, volatile fatty acids) in order to fill the sample containers by allowing the discharge to flow gently down the inside of the container with minimal turbulence.

9. Stabilization of indicator parameters is considered to be achieved when three consecutive readings at **five-minute intervals** (or longer) are within the limits listed below.

10. Sampling Requirements:

a. When the drawdown rate and indicator field parameters have stabilized, ensure that the PVR has been met before sample collection. On the worksheet, record the total volume that was purged before sample collection begins and then compare that volume to the PVR. The PVR was calculated in the Preliminary Procedures and recorded on the worksheet.If the PVR has not been met, continue purging the well (and recording stabilization parameters) until a minimum of one tubing volume of water (i.e., PVR) is discharged from the flow-through cell. Do not include the volume of water in the flow-through cell as part of the PVR.

b. If all the indicator field parameters have not stabilized within 2 hours of commencing purging, proceed as in Step "a" above, ensuring that the PVR has been met before sample collection. Note: Additional information (i.e., parameters that did not stabilize and total purge volume) will be input in to the 'Test Notes' section during Post Sampling Activities. Record the following information on the field worksheet: indicate that two-hour purge limit was reached, note which specific parameters did not stabilize, the final set of readings, and the total purge volume.

11. Sample Collection

a. Samples for laboratory analyses must be collected before the flow-through cell and the three-way stopcock. This will be done by disconnecting the three-way stopcock from the pump discharge tubing so that the samples are collected directly from the pump tubing.
b. Remove the cap from the sample container and place it on the plastic sheet or in a location where it won't become contaminated.
c. VOC samples are typically collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling projects; the site-specific QAPP shall list the order in which the samples are to be collected based on the project's objectives.
For collecting VOC samples, including 1,4-dioxane (for analytical Methods 8260 SIM), carbon dioxide, methane, ethane and ethene, refer to the *Special Notes* section at the end of this SOP.
d. If dissolved (i.e., filtered) metals samples or other samples collected for dissolved analytes, such as dissolved organic carbon (DOC), are required, then attach a onetime-use-only in-line filter (transparent housing preferred) to the end of the tubing. The filter size (0.45 micron is commonly used) is based on the sampling objective. Make sure the filter is free of air bubbles before samples are collected. Hold the filter upright until the purge water exits the top to allow the water to completely fill the filter. Allow a volume of purge water, roughly equivalent to the volume of the filter, to discharge into the bucket to rinse the filter before collecting the sample. Discard the filter after use. When collecting a duplicate sample, a new filter must be used**.**
Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health or ecological risk calculations.
e. Make sure that all sample containers are properly labeled. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence. Cap sample containers securely after filling each bottle. Sample containers must be wiped dry.

1) If the pump's flow rate is too high to collect the VOC/dissolved gas samples, follow this procedure unless otherwise specified in the QAPP: collect the other samples first, lower the pump's flow rate to a reasonable rate, collect the VOC/dissolved gas samples and record the new flow rate.
2) For bladder pumps that will be used to collect VOC or dissolved gas samples, the pump must be set to deliver long pulses of water so that one pulse will fill a 40 ml VOA vial.

f. Field duplicate and matrix spike/matrix spike duplicate (MS/MSD) samples are collected by filling a separate container for each analysis immediately following the actual field sample collection (e.g., VOC sample, VOC duplicate sample, VOC MS/MSD sample, SVOC sample, SVOC duplicate sample, SVOC MS/MSD sample). Refer to the site-specific QAPP for specific QA sampling requirements.
In general, most MS/MSD samples will be requested by the laboratory as part of their QA requirements. If that is the case, add a note to the comment section on the chain-of-custody (e.g., "Lab MS/MSD") on the same line used for the regular samples at that location. The number of sample containers will also change to accommodate the extra bottle(s) for the MS/MSD sample. MS/MSD samples should not be on a separate line on the chain-of-custody unless they are blind samples. Refer to the Chain of Custody SOP in the QAPP for information on collected Site-related MS/MSD samples.
g. Place samples in re-sealable plastic bags. Samples requiring cooling shall be placed **into loose ice** within a cooler for delivery to the laboratory. Metals samples, after acidification to a pH less than 2, do not need to be cooled.

13. Post Sampling Activities
    a. After sample collection, sample information including sample identification, analysis, sample collection time, and any QC samples if collected, will be entered by selecting "Add Sample" from the bottom of the screen. Move on to the test notes screen by selecting "next" but do not yet select "Complete".
    b. Just prior to turning off the pump, measure and record the water level on the worksheet, then turn off the pump. Disconnect the flow-through cell from the three-way stopcock and discharge the water from the flow-through cell into the graduated bucket.
    c. Record the total purged volume (contained in the graduated bucket) on the Low Flow Sampling Worksheet or "test notes" section of the low-flow test on the tablet. Make sure that the water in the flow-through cell has already been discharged into the bucket before recording the total volume in the bucket.

d. Disconnect all other equipment as needed.
e. Remove the pump and tubing from the well, unless dedicated. Secure dedicated pump and tubing to the inside of the well. Non-dedicated tubing should be discarded.
f. Collect the well depth measurement, if required in the site-specific QAPP. Record the depth measurement on the Low Flow Sampling Worksheet, or in the "test notes" section of the low-flow test on the tablet. Also note variation in total depth of well compared to that previously documented.
g. Secure the well with the locking cap.
h. All non-dedicated equipment (e.g., water level meter) must be decontaminated according to the Decontamination SOP in the site-specific QAPP.
i. Collect an equipment blank if required. Refer to the site-specific QAPP for details and analysis.

MODIFIED SAMPLING PROCEDURE

This procedure is to be used if: (1) the well is under artesian conditions; (2) the water level is at, or below, the top of the screen; (3) the well has been identified in the site-specific QAPP as having insufficient recharge (minimal drawdown cannot be achieved according to the criteria listed in the standard procedure above); or (4) the well had to recharge overnight. Wells where this occurs may be considered for replacement in the future. This modified procedure includes the collection of one set of field parameters before collecting the samples.

Procedure

1. Set up equipment. Be sure to tilt the flow-through cell so that the outflow connection is facing upward, in order to eliminate and prevent air bubble entrapment in the flow-through cell.
2. Measure the water level. Carefully lower a water level indicator to the top of groundwater. Measure and record the water level (to 0.01 feet) before any disturbance to the well. Care shall be taken to minimize suspension of any particulates attached to the sides. Document the data on the field sampling worksheet.
3. Start the pump and allow the flow-through cell to fill.
   If using the YSI, note pump start time on the worksheet.
   a. Start the pump at its lowest speed setting and slowly increase the speed until a flow rate of 50 ml/minute is reached. **Pumping rates shall not be less than 50 ml/minute.**
   b. If a 50 ml/minute flow rate cannot be achieved, then set the pump at the lowest flow rate that the pump is cable of attaining.

If using a peristaltic pump, make sure the correct pump head (thin wall tubing pump head) and the silicon tubing (smallest size silicon tubing, #14) is being used and that the pump head is set at 300 RPM, not 600 RPM.

4. Purge Volume Requirement

   Ensure that the purge volume requirement (PVR) has been met before sample collection. On the worksheet, record the total volume that was purged before sample collection begins and then compare that volume to the PVR. The PVR was calculated in the Preliminary Procedures and recorded on the worksheet.

   Continue purging the well at 50 ml/minute, or lowest flow rate possible above that, until a minimum of one tubing volume of water (i.e., PVR) is discharged from the flow-through cell. Do not include the volume of water in the flow-through cell as part of the PVR.

5. Collect one set of parameter readings from the YSI or In-Situ instrument, and an aliquot of water through the three-way stop cock to analyze for turbidity using the Hach Turbidity meter. Record the YSI and turbidity readings on the sampling worksheet.

6. Sample Collection

   a. Samples for laboratory analyses must be collected before the flow-through cell and three-way stopcock. This will be done by disconnecting the three-way stopcock from the pump discharge tubing so that the samples are collected directly from the pump tubing.

   b. Remove the cap from the sample container and place it on the plastic sheet or in a location where it won't become contaminated.

   c. Begin collecting groundwater samples in the order found in the site-specific QAPP. VOCs are typically collected first.

      For collection of VOC samples, including 1,4-dioxane (for analytical Methods 8260 SIM), carbon dioxide, methane, ethane and ethene, refer to the *Special Notes* section at the end of this SOP.

   d. If dissolved (i.e., filtered) metals samples or other samples collected for dissolved analytes, such as dissolved organic carbon (DOC), are required, then attach a onetime-use-only, 0.45-micron, in-line filter to the end of the tubing. Make sure the filter is free of air bubbles before samples are collected. Hold the filter upright until the purge water exits the top to allow the water to completely fill the filter. Allow a volume of purge water, roughly equivalent to the volume of the filter, to discharge into the bucket to rinse the filter before collecting the sample. Discard the filter after use. When collecting a duplicate sample, a new filter must be used.

e. Make sure that all sample containers are properly labeled. Fill all sample containers by allowing the discharge to flow gently down the inside of the container with minimal turbulence. Cap sample containers securely after filling each bottle. Sample containers must be wiped dry.
f. Field duplicate and matrix spike/matrix spike duplicate (MS/MSD) samples are collected by filling a separate container for each analysis immediately following the actual field sample collection (e.g., VOC sample, VOC duplicate sample, 1,4-dioxane sample, 1,4-dioxane duplicate sample and/or 1,4-dioxane MS/MSD sample). Refer to the site-specific QAPP for specific QA sampling requirements.
   In general, most MS/MSD samples will be requested by the laboratory as part of their QA requirements. If that is the case, add a note to the comment section on the chain-of-custody (e.g., "Lab MS/MSD") on the same line used for the regular samples at that location. The number of sample containers will also change to accommodate the extra bottle(s) for the MS/MSD sample. MS/MSD samples should not be on a separate line on the chain-of-custody unless they are blind samples. Refer to the Chain of Custody SOP in the QAPP for information on collecting Site-related MS/MSD samples.
g. Place samples in re-sealable plastic bags. Samples requiring cooling shall be placed **into loose ice** within a cooler for delivery to the laboratory. Metals samples, after acidification to a pH less than 2, do not need to be cooled.

7. Post Sampling Activities
   a. After completing the test, sample information including sample identification, analysis, sample collection time, and any QC samples if collected, will be entered by selecting "Add Sample" from the bottom of the screen. Move on to the test notes screen by selecting "next" but do not yet select "Complete".
   b. Just prior to turning off the pump, measure and record the water level on the worksheet, then turn off the pump. Disconnect the flow-through cell from the three-way stopcock and discharge the water from the flow-through cell into the graduated bucket.
   c. Record the total purged volume (contained in the graduated bucket) on the Low Flow Sampling Worksheet or "test notes" section of the low-flow test on the tablet. Make sure that the water in the flow-through cell has already been discharged into the bucket before recording the total volume in the bucket.
   d. Disconnect all other equipment as needed.
   e. Remove the pump and tubing from the well, unless dedicated. Secure dedicated pump and tubing to the inside of the well. Non-dedicated tubing should be discarded.

f. Collect the well depth measurement, if required in the site-specific QAPP**.** Record the depth measurement on the Low Flow Sampling Worksheet, or in the "test notes" section of the low-flow test on the tablet. Also note variation in total depth of well compared to that previously documented.

The following information should have been entered PRIOR to starting the low flow test:
Sampler's full name or first initial and full last name (not just initials)
Pump serial number
Turbidity meter model and serial number
Static water level from measuring point
Total tubing length (inside and5 outside of well)
Check tubing inside diameter shown on template, if not correct, enter correct inside diameter here
PVR (including the calculations)
Maximum allowable drawdown rate
The following information must be captured in the test notes:
Pump start time
Indicator parameters stable? If "no", which parameters were out?
Record total purge volume before sample collection to compare to PVR
Minimum PVR reached?
2-hour time limit reached?
Clock time for switch to Modified Sampling Procedure, if applicable
Condition that triggered the switch to the Modified Sampling Procedure, if applicable
Any adjustments made (including adjustments in flow rates, etc.)

Time at sample collection (as recorded on the bottle) and completion (24-hour clock time)
All samples collected, including QC samples (i.e., VOCs, DUPs, FIELD BLANKs) recorded on the "Sample" page
Final water level
Total volume purged (recorded in test notes only)
Measured Well depth, if required. If measured, note variation in total depth of well compared to that previously documented.
Comments or field observations during sampling event (e.g., condition of well, missing locks, weather)

Notes:

a) If the sampler went directly to the Modified Sampling Procedure without starting the standard low flow procedure, then the information that is **bolded** in the table above will not be applicable.

2) Once this information has been added to the "test notes" for each well, click "**Complete**". **At this point, no additional revisions or additions of information can be made.**

3) Secure the well with the locking cap.

h. All non-dedicated equipment (e.g., water level meter) must be decontaminated according to the *Decontamination* SOP.

i. Collect an equipment blank if required. Refer to the site-specific QAPP for details.

RECORDS AND DOCUMENTATION

A field log must be kept each time ground water monitoring activities are conducted in the field. The field logs/sampling worksheets shall be filled out in black ink.

SPECIAL NOTES

Special Considerations for the Collection of Volatile Organic Compound Samples

Including 1,4-dioxane samples analyzed by Method 8260 SIM.

The proper collection of a sample for volatile organic compound analysis requires minimal disturbance of the sample to limit volatilization and therefore minimize loss of volatiles from the sample. The following VOC procedures shall be followed:

1. Open the vial, set cap in a protected place, and collect the sample by allowing the water to flow gently down the inside wall of the container with minimal turbulence. When collecting quality control samples (duplicates and MS/MSD samples), collect them immediately following the original sample (e.g., VOC sample, VOC duplicate sample, then VOC MS/MSD sample).
2. Do not rinse the vial or excessively overflow it because it likely contains a specific volume of preservative that must not be diluted.
3. Do not collect the initial 10 ml (approximate) of sample in the discharge tubing, as the beginning of the sample has been in contact with air.
4. Be sure the sample flow is laminar and there are no air bubbles in the sample flow.
5. There should be a convex meniscus on the top of the vial prior to capping the vial. The cap may be used to create the convex meniscus for VOC samples, if needed.
   For methane/ethane/ethene and carbon dioxide, the laboratory typically requests that the sample bottle cap is not used to top off the sample vials. These vials should be filled in the shortest time possible and capped immediately. Do not uncap these vials and add more water. Small bubbles are considered normal for these pre-preserved containers; however, every effort shall be made to collect the highest quality (e.g., bubble free) sample possible.
6. Check that the cap has not been contaminated (e.g., splashed) and carefully cap the vial.
7. Place the cap directly over the top and screw down firmly. Do not over-tighten and break the cap.
8. Invert the vial and tap gently. If an air bubble appears, uncap and attempt to add a small volume of sample to achieve the convex meniscus without excessively overfilling the vial. If this has to be repeated more than twice, discard the sample and vial and begin again with a new container and preservative. It is imperative that no entrapped air is in the sample vial.
9. Wipe the vial dry and immediately place the vial into a re-sealable plastic bag and then into loose ice in the cooler.

REFERENCES

USEPA Region I, 1996; Low-Stress (Low-flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells, Revision 4, September 19, 2017, SOP #EQASOP-GW-4.

Vroblesky, Don A., Clifton C. Casey, and Mark A. Lowery, Summer 2007, Influence of Dissolved Oxygen Convection on Well Sampling, *Ground Water Monitoring and Remediation* 27, no. 3: 49-58.

GZA GeoEnvironmental, Inc.