QUALITY ASSURANCE PROJECT PLAN

EPA DOCKET NO. RCRA 05-2013-0011
WEDRON, ILLINOIS

PREPARED FOR:

Wedron Silica Company

and

Lockheed Martin Corporation

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October 15, 2013
GZA File No. 20.0151178.51

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QUALITY ASSURANCE PROJECT PLAN APPROVAL SHEET
EPA DOCKET NO. RCRA 05-2013-0011
WEDRON, ILLINOIS

On behalf of Wedron Silica Company (Wedron Silica), Technisand, Inc. (Technisand) and Lockheed Martin Corporation (Lockheed Martin), this Quality Assurance Project Plan (QAPP) was prepared by GZA GeoEnvironmental, Inc. (GZA) for use during implementation of the Workplan attached to the September 25, 2013 AOC. The QAPP was developed following the guidance presented in the U.S. EPA document QA/G-5 *Guidance for Quality Assurance Project Plans*, dated December 2002.

NAME	DATE
Steven Faryan, USEPA Project Manager	
First Name, Last Name, USEPA QA Reviewer	
Michael Melton, Wedron Silica and Technisand	
William Bath, Lockheed Martin	
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Stella Cuenco, Laboratory Data Consultants, Inc.	

QUALITY ASSURANCE PROJECT PLAN DISTRIBUTION LIST

The following have received a copy of this Quality Assurance Project Plan:

- Steven Faryan, USEPA Project Manager
- First Name, Last Name, USEPA QAPP Reviewer
- Michael Melton, Wedron Silica Company
- William Bath, Lockheed Martin
- Mark J. Krumenacher, GZA Principal In Charge
- Bernard Fenelon, GZA Project Manager
- Jay Karls, GZA QA Manager
- Jessica Esser, ECCS, Inc., Laboratory Project Manager
- Daniel Milewsky, Pace Analytical Services, Inc.
- Stella Cuenco, Laboratory Data Consultants, Inc.

ACRONYM LIST

AOC - Administrative Order on Consent
ASTM - American Society for Testing and Materials
CFR - Code of Federal Regulations
COC - Chain of Custody
DAR - Data Assessment Report
DI - Deionized
DQOs - Data Quality Objectives
GZA - GZA GeoEnvironmental, Inc.
ECCS - Environmental Chemistry Consulting Services, Inc.
HASP - Health and Safety Plan
IEPA - Illinois Environmental Protection Agency
LDC - Laboratory Data Consultants, Inc.
LCS - Laboratory Control Sample
MDL - Method Detection Limit
MS/MSD - Matrix Spike/Matrix Spike Duplicate
OSHA - United States Department of Labor, Occupational Safety and Health Administration
PARCCS - Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity
PE - Performance Evaluation
PID - Photoionization Detector
PPE - Personal Protective Equipment
QA - Quality Assurance
QAPP - Quality Assurance Project Plan
QA/QC - Quality Assurance/Quality Control
QC - Quality Control
RPD - Relative Percent Difference
RSD - Relative Standard Deviation
RL - Reporting Limit
SOPs - Standard Operating Procedures
SRM - Standard Reference Materials
SROs - soil remediation objectives
TACO - Tiered Approach to Corrective Action Objectives
USEPA - United States Environmental Protection Agency
USTs - Underground Storage Tanks
VOCs - Volatile Organic Compounds

1.0 PROJECT DESCRIPTION

The purpose of this document is to describe the personnel, procedures and methods for ensuring the quality, accuracy and precision of data associated with implementation of the Workplan made part of the September 25, 2013 AOC for conducting sampling, analysis and monitoring at property currently or historically owned and/or operated by Wedron Silica Company (Wedron Silica), Technisand, Inc. (Technisand) and Martin Marietta Corporation (Martin Marietta) in Wedron, Illinois and at other locations in Wedron, Illinois. The Workplan was prepared in response to requests made by the United States Environmental Protection Agency (USEPA) to Wedron Silica, Technisand and Lockheed Martin Corporation (Lockheed Martin), and in accordance with the terms of an Administrative Order on Consent (AOC) to which the Workplan was attached.

Data will be collected pursuant to procedures outlined in this Quality Assurance Project Plan (QAPP). This QAPP will be valid for the duration of the Workplan implementation and may be used for follow-up data collection activities as necessary. If use of the QAPP is extended for work beyond implementation of the Workplan, it will be reviewed annually (from the date of approval) and updated as necessary. This annual review will be documented and, if changes to the QAPP are necessary, revisions will be sent to all recipients of the QAPP with updated materials (current laboratory certificates, resumes for new key staff, etc.) to insert into the QAPP. If substantial changes are anticipated during the project period (new laboratories, additional analyses, new field methods, etc.), the parties that reviewed this QAPP will be contacted to evaluate the most appropriate way to revise this document.

1.1 PROJECT SCOPE

The purpose of the Workplan is to evaluate the nature and extent of the presence and/or release of hazardous wastes and/or hazardous constituents at certain locations on Wedron Silica property using a phased approach, comparing soil analytical results to Illinois Tiered Approach to Corrective Action Objectives (TACO)[1] Tier 1 Class I migration to groundwater soil remediation objectives (SROs) to evaluate whether potential source areas exist which require additional characterization and/or investigation.

The general areas included for investigation are:

1. the Tech Center;

2. the area around Illinois Environmental Protection Agency (IEPA) boring GP-10 drilled in 2012 and IEPA monitoring well G103 installed in 1984 near the former Scale-House well;

3. the two former 4,000-gallon gasoline underground storage tanks (USTs) and dispensers closed in 1998 near the main office, which are being investigated under

[1] Title 35, Subtitle G, Chapter I, Subchapter F, Part 742 of the Illinois Administrative Code.

the oversight of the Illinois Office of State Fire Marshal (OSFM), pursuant to a separate Workplan;

4. the former 6,000-gallon gasoline UST near the current Screen House that was closed in 1982;

5. the Pit 2 reclamation area; and

6. evaluation of community-wide groundwater and surface water elevations.

The six areas of investigation are identified on Figure 1 and are discussed further in Section 1.3. The work scopes proposed for the six areas were developed through discussions among representatives of the USEPA, Wedron Silica and Lockheed Martin, and will be performed pursuant to the terms of the AOC. A detailed description of the project scope is provided in the Workplan incorporated in the September 25, 2013 AOC and is not repeated in detail here. The AOC is provided in Appendix A. The Workplan, to be implemented in the six areas listed above, consist of one or more of the following tasks for each of the areas:

- Drilling of Geoprobe® soil borings;
- Field screening and describing soil samples;
- Collection and analysis of soil samples;
- Collection and analysis of grab groundwater samples;
- Installation of groundwater piezometers;
- Measurement of surface and groundwater levels; and
- Evaluation of soil and groundwater data with respect to TACO Tier 1 Class I Migration to Groundwater SROs.

1.2 PROJECT ORGANIZATION AND RESPONSIBILITY

Figure 2 presents the general organizational structure to be used for implementation of the Workplan. The lines of communication, management activities and technical direction within this project team will follow this organization arrangement. Directions or communications from the USEPA will be given to the Wedron Silica or Lockheed Martin Project Managers. The Wedron Silica and/or Lockheed Martin Project Managers will subsequently communicate directions to the GZA Project Manager. The USEPA Project Manager will be notified of proposed changes in personnel. Responsibilities of key project personnel are outlined below.

USEPA Project Manager:

1. Direct, review and approve QAPP and data quality objectives (DQOs).

2. Review progress reports detailing work accomplished.

3. Review final reports.

USEPA Quality Assurance (QA) Reviewer:

1. Review and approve the QAPP.

2. Assist in review of workplans.

Mr. Steven Faryan will serve as the USEPA Project Manager and Mr./Ms. First Last Name will served as the USEPA QAPP reviewer.

Wedron Silica/Lockheed Martin:

1. Direct project activities.

2. Responsible for review of project deliverables, development of project planning and the overview of project strategies.

3. Review site reports for consistency with objectives stated in the Workplan.

4. Provide approval on reports.

Mr. Michael Melton and Mr. William Bath are the Project Managers for Wedron Silica and Lockheed Martins.

GZA Principal-In-Charge:

1. Responsible for assignment of appropriate personnel for completing project.

2. Responsible for review of environmental reports and documents.

3. Provide final signature on reports.

GZA Project Manager:

1. Responsible for planning, coordinating, monitoring and evaluating of project field activities.

2. Before sampling, meet with QA manager and field staff to discuss and verify sampling purposes, sampling methodology, number of samples, size of

samples, sample preservation methods, chain-of-custody (COC) requirements, analyses required and which sample locations will be selected for field duplicates.

3. Maintain a record of samples collected and the sample identification information on each sample.

4. Manage data acquired from field assessments and laboratory analyses and assemble data into computer format.

5. Resolve technical problems that may arise.

6. Responsible for preparation of environmental reports and documents.

7. Provide final signature on reports.

GZA QA Manager:

1. Oversee assessment activities to confirm sampling methodology, sample preservation methods and COC procedures are being followed.

2. Assist in any QA issues with field or laboratory questions, as needed.

3. Conducts field audits.

4. Maintain a record of samples submitted to the laboratory, the analyses being performed on each sample, the final analytical results and data validation reports.

5. Prepare a Data Assessment Report (DAR).

6. Annual review of the QAPP.

GZA Field Team Leader:

1. Review on-site Health and Safety Plan (HASP).

2. Review applicable Workplan as it pertains to each field task.

3. Be responsible for oversight of field activities to confirm that procedures for the field activities related to the QAPP are executed and documented properly.

4. Submit data generated during field assessment to the project manager.

5. Procuring, coordinating and qualifying subcontractors.

GZA Field Technical Staff:

1. Before sampling, meet with GZA Project Manager to discuss and establish sampling purposes, sampling methodology, number of samples, sample preservation methods, COC requirements, analyses required and which samples will be duplicated in the field.

2. Be responsible for collection of equipment needed for property assessment work, which would include personal protective equipment (PPE), sampling equipment, sample containers and coolers, water-level meters, monitoring devices and any other equipment deemed necessary.

3. Oversee drilling and soil boring work to during monitoring well installation and soil sample collection from borings.

4. Monitor for hazardous conditions while conducting field operations.

5. Submit COC records and field paperwork to field team leader.

Mr. Mark Krumenacher, P.G. will serve as the GZA Principal-In-Charge. Mr. Bernard Fenelon will serve as the GZA Project Manager and GZA Data Manager. Dr. Jay Karls will serve as the GZA QA Manager. The GZA Field Team Leader will be Mr. David Bauer. The Field Technical Staff may include: Mr. Christopher Ainsworth, Mr. Daryl Lamb, Ms. Carlene Polk and Ms. Elizabeth Stapleton, although other supporting staff from GZA may be assigned on an as-needed basis. Resumes for key GZA personnel are included in Appendix B.

GZA site personnel will be trained as mandated by the United States Department of Labor, Occupational Safety and Health Administration (OSHA) Act regulations (29 Code of Federal Regulations [CFR] 1910.120). When working on mine property, at least one of GZA's personnel will be trained in accordance with Mine Safety and Health Administration (MSHA) 30 CFR Part 46 requirements and will have gone through site-specific training on at least an annual basis. Additionally, site personnel will be properly trained in the procedures for collecting, labeling, packaging and shipping of liquid and solid environmental samples. Mr. Michael McCoy, the GZA Waukesha, Wisconsin office Health and Safety Coordinator, maintains personnel training records. Field personnel will be trained to use monitoring devices and other equipment used in the field.

Laboratory Data Consultants, Inc. (LDC) - Project Manager:

1. Responsible for ensuring that the data validation is conducted in accordance with Level III and 10% Level IV requirements.

2. Responsible for meeting the data validation requirements within the three-week turnaround time requirements.

Ms. Stella Cuenco, senior chemist, will serve as the LDC overall program manager

Environmental Chemistry Consulting Services, Inc. (ECCS) - Project Manager:

1. Responsible for samples submitted to ECCS, including those released to a subcontracted laboratory.

2. Responsible for summarizing QA/quality control (QA/QC) requirements for the project, including those samples analyzed by subcontracted laboratories.

3. Maintain laboratory schedule and communicate technical requirements to laboratory personnel.

4. Provide technical guidance to the GZA Project Manager.

5. Verify accuracy of the laboratory data.

ECCS QA Manager:

1. Responsible for evaluating adherence to policies and ensuring that the systems are in place to provide QA/QC, as defined in the QAPP.

2. Initiate and oversee audits of corrective action procedures.

3. Perform data reviews.

4. Maintain documentation of training.

The primary laboratory selected to perform the analytical work required under the Workplan is ECCS located in Madison, Wisconsin. However, ECCS will also be subcontracting certain laboratory analyses to an Illinois-certified laboratory (Pace Analytical Services, Inc. [Pace]). The Project Managers and QA Managers at these subcontractor laboratories will have the same responsibilities as the primary laboratories, as described above.

Ms. Jessica Esser will serve as the ECCS Project Manager, and Mr. Mike Linskens will serve as the ECCS QA Manager. The ECCS Project Manager will be ultimately responsible for ensuring the quality of the laboratory data. Mr. Daniel Milewsky will serve as Pace's Project Manager and will be ultimately responsible for ensuring the quality of the Pace laboratory data. The Pace QA Manager will be Ms. Kate Grams.

Because of the strict schedule to which implementation of the Workplan must adhere and the unknown availability of the subcontractors at the time the work is required to be conducted, the drilling subcontractors have not yet been selected for this project. However, on-site drilling personnel shall have been provided the applicable OSHA training. Additionally, drilling personnel will be required to comply with site safety

regulations covered in the site-specific HASP, provided under separate cover to this QAPP.

1.3 BACKGROUND INFORMATION

The purpose of the Workplan is to evaluate the nature and extent of the presence and/or release of hazardous wastes and/or hazardous constituents at certain locations on Wedron Silica property using a phased approach, comparing soil analytical results to Illinois TACO Tier 1 Class I migration to groundwater soil remediation objectives (SROs) to evaluate whether potential source areas exist which require additional characterization and/or investigation. The general areas included for investigation are:

1) the Tech Center;

2) the area around Illinois Environmental Protection Agency (IEPA) boring GP-10 drilled in 2012 and IEPA monitoring well G103 installed in 1984 near the former Scale-House well;

3) the two former 4,000-gallon gasoline underground storage tanks (USTs) and dispensers closed in 1998 near the main office, which are being investigated under the oversight of the Illinois Office of State Fire Marshal (OSFM), pursuant to a separate Workplan;

4) the former 6,000-gallon gasoline UST near the current Screen House that was closed in 1982;

5) the Pit 2 reclamation area; and

6) evaluation of groundwater and surface water elevations.

The Tech Center and on-site wastewater treatment system layout are shown on View 1 on Figure 1.

The area of IEPA 2012 boring GP-10 and 1984 monitoring well G103 near the former Scale-House well is shown on View 2 on Figure 1. During a 1982 to 1984 groundwater investigation by IEPA, petroleum constituents were reported in the Scale-House well. IEPA noted odors in soil samples collected between the depths of 14.7 feet and approximately 15.8 feet where clay was encountered in the boring (B5) drilled for monitoring well G103. Petroleum constituents were reported at concentrations below TACO Tier 1 SROs in 2012 in soil collected from a depth of 15.5 feet (estimated to be from near or below the water table) in boring GP-10. Refusal was encountered at a depth of 18 feet in boring GP-10.

The former 4,000-gallon gasoline UST system is shown on View 3 on Figure 1. Site assessment soil samples were collected from the base and sidewalls of the UST tank basin at the time the USTs were pulled in 1998, but soil samples were not taken beneath the

short section of piping runs between the USTs and dispensers or beneath the dispensers. Based on information in the UST closure report and historical aerial photography, the locations of the two USTs and two dispensers were identified to within a couple of feet.

The former 6,000-gallon gasoline UST near the current Screen House is shown on View 4 on Figure 1. The 6,000-gallon gasoline UST was installed in approximately 1975, and pulled in approximately 1982 to allow for construction of the current Screen House. Based on information in Wedron Silica files, the UST was located north of the "Paper Shed" and was 29 feet 2 inches long and 6 feet in diameter. The location of the Paper Shed was identified in historical aerial photography.

The partially reclaimed Pit 2 is shown on Figure 1. After sand extraction from the quarry ended, the southern portion was reclaimed with various materials. Further evaluation of the groundwater-flow conditions in and around Pit 2 will be conducted before soil samples are collected from soil borings for laboratory analysis. One groundwater-level monitoring location will be completed. We understand that additional wells will be installed by USEPA and IEPA in 2013, and water levels obtained from these wells will also be used to refine groundwater-flow conditions in and around Pit 2.

In order to further evaluate and define the groundwater-flow regime throughout the Wedron community, three additional piezometers will be installed and two rounds of groundwater and surface water levels measured at the locations described in the Workplan.

1.4 PROJECT SCHEDULE

The schedule for the various tasks specified in the Workplan is detailed in the AOC, which is provided in Appendix A.

1.5 DATA QUALITY OBJECTIVES

DQOs are qualitative and quantitative statements that clearly state the objective of a proposed project, define the most appropriate type of data to collect, determine the appropriate conditions for data collection and specify acceptable decision error limits that establish the quantity and quality of data needed for decision making. The DQOs are based on the use of the data that will be generated. Different data uses may require different quantities of data and levels of quality.

1.5.1 Analytical Quality Objectives

Analytical quality objectives are used so that analyses will accurately and adequately identify the contaminants of concern, and analyses will be able to achieve the quantitation limits less than or equal to the target cleanup levels.

1.5.1.1 Field Screening

Field-screening instruments provide rapid "real-time" results for field personnel in order to help guide field decision-making processes. These

techniques are often used for health and safety monitoring, initial site characterization to locate areas for more detailed assessment, to guide the selection of soil and groundwater samples for obtaining higher quality analytical data in a laboratory and preliminary comparison of remedial objectives. However, field screening methods have significant inherent limitations and provide a much lower quality of analytical data compared to laboratory equipment.

Field-screening data can include measurements of organic vapor concentrations, pH, temperature, conductivity, oxidation-reduction (ORP) potential, dissolved oxygen (DO), turbidity, and other similar field monitoring data. During sampling activities, the breathing space of site personnel and the head space of soil samples will be monitored for the presence of organic vapors using a photoionization detector (PID) instrument. The PID will be used to monitor the breathing space in accordance with the requirements of the site-specific HASP. The PID will be used to perform field screening of soil samples in order to assist in the selection of samples to be submitted for laboratory analysis. Generally, the two soil intervals with the highest PID readings and/or field indications of odors, staining, etc., from each soil boring will be submitted to the laboratory for analyses, as summarized on Table 1. If volatile organic vapors are not detected by the PID or other field indications are not noted, samples will be selected for laboratory analyses from the two intervals identified in the Workplan specific to each area being evaluated, as specified on Table 1.

1.5.1.2 Illinois-Certified Laboratories

Analyses (volatile organic compounds [VOCs], lead, pH and fraction organic carbon [f_{oc}]) will be performed by ECCS or Pace at their respective Madison and Green Bay, Wisconsin locations, respectively. Table 2 summarizes the analyses to be performed by each of the two laboratories for groundwater and soil samples.

The State of Illinois formally recognizes that ECCS is technically competent to perform analyses for VOCs in accordance with the method specified in this QAPP and has issued Certificate No. 003174 to ECCS for their Madison, Wisconsin laboratory. A copy of the State of Illinois certificate issued to ECCS is provided in Appendix C.

The State of Illinois formally recognizes that Pace is technically competent to perform analyses for f_{oc}, lead and pH in accordance with the methods specified in this QAPP and has issued Certificate No. 003263 to Pace for their Green Bay, Wisconsin laboratory. A copy of the State of Illinois certificate issued to Pace is provided in Appendix C

1.5.2 Project Quality Objectives

The project quality objectives process is a series of planning steps designed so that the type, quantity and quality of environmental data used in decision-making are appropriate for the intended application. Five steps can be considered in the project quality objectives process and include problem statement, decision identification, decision inputs, assessment boundary and the decision process. The details of these steps are provided in the following sections.

1.5.2.1 Problem Statement

Petroleum constituents were reported in several drinking water wells in the southeastern portion of the Wedron community in 1982, and more recently, in 2011, have been reported in additional drinking water wells at concentrations that exceeded TACO Class I Tier I residential groundwater levels.

1.5.2.2 Decision Identification

Evaluation of the nature and extent of the presence and/or release of hazardous wastes and/or hazardous constituents at certain locations on Wedron Silica property using a phased approach, comparing soil analytical results to Illinois TACO Tier 1 Class I Migration to Groundwater SROs will permit evaluation of whether potential source areas exist which require additional characterization and/or investigation.

1.5.2.3 Decision Inputs

Data obtained through the collection and analysis of soil and possibly groundwater samples, as described in the Workplan, from the various locations on Wedron Silica property will be used to evaluate whether potential source areas exist which require additional characterization and/or investigation. The soil analytical data will be compared to TACO Tier 1 Class I Migration to Groundwater SROs.

Data obtained through the measurement of groundwater levels within the installed monitoring well network and specific area surface water levels will be used to assess the groundwater-flow configuration across the Wedron community. The groundwater-flow configuration will provide data for the evaluation of fate and transport of petroleum constituents released from soil to groundwater.

1.5.2.4 Assessment Boundary

For the portions of the Workplan with objectives of evaluating the presence of petroleum constituents in soil, the horizontal assessment boundary for each target evaluation area is identified by the area covered by the proposed

borings identified on Figure 1. If TACO Tier 1 Class I Migration to Groundwater SROs are exceeded in soil samples submitted for laboratory analyses, the horizontal boundary may expand in a follow-up evaluation to cover the area of TACO Tier 1 Class I Migration to Groundwater SRO exceedances, assuming that the area does not overlap with an adjacent area of soil containing petroleum constituents from a separate release area not associated with former or current Wedron Silica operations.

The assessment boundary for evaluating groundwater-flow configuration is bounded by the area of groundwater and surface water measurements and covers nearly the entire Wedron community.

The vertical assessment boundary for soil will vary depending on the specific potential petroleum source area evaluated, as identified in the Workplan (e.g. water table, depth of refusal, 2 to 3 feet below a UST, or 2 feet below the top of the former USTs). The vertical assessment for evaluating groundwater-flow configuration is the water table.

1.5.2.5 Decision Process

A decision for follow-up environmental activities will be based on a comparison of detected VOC constituents to Illinois TACO Tier 1 Class I migration to groundwater SROs. If soil VOC concentrations for samples collected are less than TACO Tier 1 Class I migration to groundwater SROs, additional investigation will be unnecessary. If TACO Tier 1 Class I migration to groundwater soil SROs are exceeded, additional activities such as development of Tier 2 TACO levels and/or additional soil investigation and possible investigation of groundwater will be considered. If groundwater investigation is proposed, consideration of the community-wide presence of petroleum constituents in groundwater will be taken into consideration in the interpretation of results. Specific information to be considered will include: 1) soil data collected at other potential source areas; 2) soil data collected as part of this investigation; 3) groundwater flow direction assessment utilizing piezometers and surface water gaging collected as part of this investigation; and 4) other available data. If additional investigation of soil and/or groundwater is warranted, a separate work plan will be prepared and submitted to the USEPA.

The applicable soil and groundwater levels for VOCs, which will be used to evaluate the need for follow-up environmental activities, are presented in Tables 3a and 3b for groundwater and soil, respectively. The applicable soil levels for lead, which will be used to evaluate the need for follow-up environmental activities, are presented in Table 3c.

1.6 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT

The overall QA objective for conducting the Workplan is to implement procedures for field sampling, COC and laboratory analysis identified in this QAPP. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventative maintenance of field equipment and corrective action are described in various sections of this QAPP.

Measurement DQOs for this project will be addressed in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). The DQOs and resulting PARCCS parameters will require that the sampling be performed using standard methods with properly operated and calibrated equipment, and conducted by trained personnel.

1.6.1 Precision

Precision is the degree of agreement among repeated measurements of the same parameter under the same or similar conditions. Precision is reported as either relative percent difference (RPD) or relative standard deviation (RSD), depending on the end use of the data.

1.6.1.1 Field Precision Objectives

Field precision will be assessed through the collection and analysis of field duplicate samples. RPDs will be calculated for the detected analytes from investigative and field duplicate samples. Water matrix samples can be readily duplicated due to their homogeneous nature; conversely, the duplication of soil or sediment samples is much more difficult due to their non-homogeneous nature. Due to this difficulty, RPDs of ±35 percent (%) and ±50% for water and soil sample field duplicates, respectively, will be used as advisory limits for analytes detected in both investigative and field duplicate samples at concentrations greater than or equal to five times its quantitation limit. A summary of duplicate samples to be collected is presented in Table 4, along with the other QC samples.

1.6.1.2 Laboratory Precision Objectives

For ECCS and Pace, precision of laboratory analyses will be based upon laboratory matrix spike/matrix spike duplicate (MS/MSD) analyses. Precision is reported as RPD or RSD, and the equation to be used to determine precision is presented in Section 4.3.1. MS/MSD analyses will be either at a rate of 1 per 20 samples received by the laboratory or in accordance with laboratory SOPs. Tables 3a, 3b, 3c and 3d list the MSD and RPDs used by ECCS and Pace. The QA Manuals for the laboratories to be utilized are included in Appendix D.

1.6.2 Accuracy

Accuracy is the extent of agreement between an observed or measured value and the accepted reference, or true, value of the parameter being measured.

1.6.2.1 Field Accuracy Objectives

The objective for accuracy of the field sample collection procedures is to minimize the potential that samples will be affected by sources external to the sample, such as sample contamination by ambient conditions or inadequate equipment decontamination procedures. Sampling accuracy will be assessed by evaluating the results of equipment and trip blank samples for contamination.

A trip blank will consist of a laboratory-prepared sample of reagent-grade water. Trip blanks will accompany sample containers and be subjected to the same handling procedures as the field samples, but will not be opened and will be shipped back to the laboratory with the samples. Trip blanks are required only when VOCs will be analyzed for water samples. Trip blanks will be submitted at the rate of one trip blank per shipping container containing water samples for laboratory VOC analysis. The trip blank samples will provide a measure of potential cross-contamination of samples by VOCs during shipment and handling.

A methanol blank will consist of a laboratory-prepared sample of laboratory-grade methanol. Trip blanks will accompany sample containers and be subjected to the same handling procedures as the field samples and will be shipped back to the laboratory with the samples. Methanol blanks are required only when VOCs will be analyzed for soil samples. Methanol blanks will be submitted at the rate of one per shipping container containing soil samples for laboratory VOC analysis. The methanol blank samples will provide a measure of potential cross-contamination of samples with VOCs during use, handling, storage and shipment.

Equipment blanks will be collected by pouring laboratory-prepared water or distilled water over or through the field sampling equipment and collecting the rinsate in the proper analytical containers. Equipment blanks must be submitted to the laboratory with investigative samples and analyzed for the same parameters as the investigative samples. The minimum required under the USEPA is one per 20 field samples per matrix, or, if less than 20 samples are collected, one equipment blank per day per sample matrix. If only disposable equipment is used to conduct the sampling, an equipment blank will be unnecessary and will not be collected.

1.6.2.2 Laboratory Accuracy Objectives

Laboratory accuracy will be assessed by determining percent recoveries from the analysis of laboratory control samples (LCSs) or standard reference materials (SRMs). The analyses of MS/MSD samples are also utilized to determine laboratory accuracy by determining percent recoveries from the analysis of MS/MSD samples. MS/MSD samples will be collected for organic and inorganic analyses at a minimum frequency of 1 per 20 or fewer samples. The equation used to determine accuracy for this project is presented in Section 4.3.2.3.

The accuracy of the organics analyses also will be monitored through analysis of surrogate compounds. Surrogate compounds are added to each standard, blank and QC sample prior to sample preparation and analysis. Surrogate compounds are not expected to be found occurring naturally in the samples, but behave analytically similar to the compounds of interest. Consequently, surrogate compound percent recoveries will provide information on the effect that the sample matrix exhibits on the accuracy of the analyses.

Laboratory QA Manuals for ECCS and Pace are provided in Appendix D. The QA objectives for each laboratory are presented in their respective QA Manuals.

1.6.3 Representativeness

Representativeness is a qualitative term that describes the extent to which a sampling design adequately reflects the environmental conditions of the site. It also reflects the ability of the sample team to collect samples and laboratory personnel to analyze those samples in such manners that the data generated accurately and precisely reflect the conditions at the site.

1.6.3.1 Measures of Representativeness of Field Data

Representativeness will be achieved by establishing the level of allowable uncertainty in the data and then statistically determining the number of samples needed to characterize the population through the DQO process. Representativeness will also be achieved through the proper selection of sampling locations. Representativeness is dependent upon the proper design of the sampling program and will be accomplished by through following this QAPP, the Workplan and standard procedures. The QA goal will be to have all samples and measurements representative of the media sampled. Field testing for pH, temperature and specific conductivity stabilization is conducted prior to groundwater sampling to confirm that representative samples are collected. Soil intervals will be homogenized for analyses other than VOCs so that representative soil samples are collected.

1.6.3.2 Measures of Representativeness of Laboratory Data

Representativeness of laboratory data cannot be quantified. However, adherence to the prescribed analytical methods and procedures, including holding times, blanks and duplicates, will maximize the representativeness of laboratory data.

1.6.4 Completeness

Completeness is defined as the measure of the quantity of valid data obtained from a measurement system compared to the quantity that was expected under normal conditions. While a completeness goal of 100% is desirable, an overall completeness goal of 90% may be realistically achieved under normal field sampling and laboratory analysis conditions.

1.6.4.1 Field Completeness Objectives

The field-sampling team will take measures to have data generated in the field be valid data. However, some samples may be lost or broken during handling and transit. Therefore, field completeness goals for this project will be to have 90% of all samples be valid data. The equation for calculating completeness is presented in Section 4.3.5.1.

1.6.4.2 Laboratory Completeness Objectives

Laboratory completeness will be a measure of the quantity of valid data measurements and analyses obtained from all the measurements and analyses completed for the project. The laboratory completeness goal is for 90% of the samples analyzed to be valid data. The procedure for determining laboratory data validity is provided in Section 4.2.2. The equation for calculating completeness is presented in Section 4.3.5.1.

1.6.5 Comparability

The confidence with which one data set can be compared to another is a measure of comparability. The ability to compare data sets is particularly critical when a set of data for a specific parameter is compared to historical data for determining trends.

1.6.5.1 Measures of Comparability of Field Data

Ensuring that this QAPP and the Workplan are adhered to and that all samples are properly handled and analyzed will satisfy the comparability of field data. Additionally, efforts will be made to have sampling completed in a consistent manner by the same sampling team.

1.6.5.2 Measures of Comparability of Laboratory Data

Analytical data are comparable when the data are collected and preserved in the same manner followed by analysis with the same standard method and reporting limits. Data comparability is limited to data from the same environmental media. Analytical method quality specifications have been established so that the data will produce comparable results. Tables 3a, 3b, 3c and 3d summarize the laboratory reporting limits.

1.6.6 Sensitivity

Sensitivity is the ability of a method or instrument to detect a parameter to be measured at a level of interest.

1.6.6.1 Measures of Field Sensitivity

The sensitivity of the field instruments selected to measure pH, temperature and specific conductivity of groundwater for this project will be measured by analyzing calibration check solutions, where appropriate, that equate to the lower end of the expected concentration range. The sensitivity of the PID instrument used to screen samples for organic vapors is relative to background readings in ambient air.

1.6.6.2 Measures of Laboratory Sensitivity

The sensitivity requirements for laboratory analyses are to be such that they meet Illinois TACO Tier 1 Class I Migration to Groundwater SROs for soil and TACO Tier 1 Class I Groundwater SRO for groundwater. Laboratory reporting and method detection limits (MDLs) are presented on Tables 3a, 3b, 3c and 3d. If analytical methods are deemed to be insufficiently sensitive for the petroleum constituents of concern previously detected in groundwater from water-supply wells and monitoring wells, alternative analytical methods may be utilized. If the compound is not expected to be a chemical of concern and the MDL exceeds the TACO Tier 1 Class I Migration to Groundwater SROs, the constituent will be considered not detected.

1.7 DOCUMENTATION AND RECORDS

Records generated during field activities are a critical part of investigation. GZA will use select documents for recording information during project activities. Records to be used for project documentation include field forms, field books, laboratory data sheets, COC forms and technical papers. GZA will retain the records generated during the investigation for as long as required under the AOC. At a minimum, the investigation report will include the following:

- Text describing field-sampling methodologies, analytical results, conclusions and recommendations;
- Figures showing property location, property boundaries, sampling locations and summaries of impacted areas, as warranted;
- Tables comparing laboratory data to the applicable standards;
- Tables summarizing QA/QC analytical results;
- Complete laboratory data reports, including copies of COC records;
- Copies of soil boring, groundwater, sediment and surface water sampling logs;
- Third-party data validation report;
- DAR that discusses and compares overall field duplicate precision data from multiple data sets collected for the project for each matrix, analytical parameter and concentration level;
- Figures depicting groundwater-flow direction;
- Figures depicting locations of detected constituents, if any; and
- Conclusions and recommendations.

2.0 DATA GENERATION AND ACQUISITION

The purpose of the QAPP is to produce reliable data throughout the investigation by:

- Ensuring the validity and integrity of the data;
- Ensuring and providing mechanisms for ongoing control of data quality;
- Evaluating data quality in terms of PARCCS; and
- Providing usable, quantitative data for analysis, interpretation and decision-making.

Field instrumentation and field methods SOPs describing the soil sample collection and field screening, groundwater collection, COC and water-level measurement procedures to be used to obtain representative data are provided in Appendix E.

2.1 SAMPLING PROCESS DESIGN

Sample locations, analytical parameters and frequency of sampling are discussed previously in this QAPP. Laboratory test parameters for the sampling program will include analysis for the following parameters:

- Soil and Groundwater VOCs (Method 8260B);
- Soil Lead (Method 6010C);
- Soil pH (Method 9045C); and
- Soil f_{oc} (Method ASTM D2975-87).

Note that the analytical versions for Methods 6010 and 9045 presented in the QAPP (Method 6010C and Method 9045C) are different from those presented in the Workplan (Method 6010B and Method 9045D) provided in the AOC. The analytical methods presented in this QAPP will be the methods used for the project. The laboratory SOPs for these analytical parameters are presented in Appendix D.

QA/QC samples will be submitted in accordance with the QAPP protocols presented in the following sections. Requirements for QA/QC samples are presented in Table 4.

2.2 ANALYTICAL METHODS REQUIREMENTS

In order to preserve the integrity of samples both before and during analyses, specific analytical methods and requirements for those methods will be followed. Samples will be collected, prepared and analyzed in accordance with the analytical methods outlined in the SOPs for ECCS and Pace. These laboratories will perform the analytical services for this investigation. The specific analytical method and reporting limits for each parameter are

presented in Tables 3a, 3b, 3c and 3d. Preparatory methods for analytical parameters are discussed in the SOPs for each laboratory and are included in Appendix D.

Proper sample containers, preservation, holding times and volumes for each analytical parameter are outlined in Table 5. The laboratories identified for use in this QAPP will provide the sample containers and preservatives. Sample containers for groundwater VOC analysis will be pre-preserved with acid by the laboratory. Soil samples to be analyzed for VOCs will be preserved with methanol at a ratio of approximately 1:1 (soil mass in grams to methanol in milliliters).

Sample containers supplied by the project laboratories will be cleaned according to USEPA standards. QC documentation will be supplied with the sample containers and preservatives in order to verify their purity. The containers and preservatives can be traced back to their certificate of analysis from their lot number. The QC documentation/certificate of analysis shall be maintained on file with the particular laboratory. Additionally, the project laboratory shall provide the GZA Field Team with trip blanks for VOC analysis and laboratory-grade deionized (DI) water for rinsing field equipment and instruments for preparation of equipment blanks.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Proper sample handling and custody procedures are crucial to ensuring the quality and validity of data obtained through field and laboratory analyses. For example, the admissibility of environmental data as evidence in a court of law is dependent on the custody of the data. Custody procedures will be used to document the authenticity of data collected during this scope of work. The data requiring custody procedures include field samples and data files that can include field books, logs and laboratory reports. An item is considered in custody if it is:

- In a person's possession;
- In view of the person after being in their possession;
- Sealed in a manner that it cannot be tampered with after having been in physical possession; or
- In a secure area restricted to authorized personnel.

2.3.1 Sample Collection Documentation

Sample-handling procedures include field documentation, COC documentation, sample shipment and laboratory sample tracking. Various aspects of sample handling and shipment, as well as the proposed sample identification system and documentation, are discussed in the following sections.

2.3.1.1 Field Books

Detailed records of the field activities will be maintained in field books dedicated to the projects. Entries will be dated and signed by personnel recording the data. The entries will be made in ink. If more than one field book is used, each field book will have a unique numerical identifier permanently attached and each page will be numbered, permitting indexing of key data. At a minimum, information recorded in the field books will include documentation of sample locations, sampling times, types of samples collected, weather conditions and other pertinent.

2.3.1.2 Field Identification System

Samples collected during the investigation will be given a unique identification code. The unique sample identification will consist of the following:

- *Project Identification Code.* The two-letter designation, WS, will be used to identify the project for which the sample was collected.

- *Sample Matrix Code.* Each sample will be further identified by a code corresponding to the sample matrix:

  SS - surface soil sample
  SB - subsurface soil sample
  GW - groundwater sample
  SW - surface water sample
  SD - sediment sample
  TB - trip blank sample
  EB - equipment blank sample
  FD - field duplicate sample

- *Location Code.* Lastly, each sample will be identified by a location code and interval as follows:

  MW-## - monitoring well location
  GP-## - location of Geoprobe® or other direct-push boring
  SB-## - location of soil borings completed by methods other than direct-push

- *Depth.* For subsurface soil samples a sampling depth interval will be provided.

- *Example.*

  WS-SB-GP-04 (6'-8') = subsurface soil from Geoprobe boring GP-04 over the depth interval of 6 to 8 feet below grade.

Sample bottle labels appropriate for the size and type of containers shall be provided by the project laboratories. The sample containers will be labeled at the time of sample collection but prior to being filled. Each label will indicate, at a minimum:

- Sample identification;
- Date/time of sample collection;
- Sampler's initials;
- Required analyses; and
- Type of preservative.

Labels will be completed in waterproof ink. A sample label is included in Appendix F.

2.3.1.3 Field Sample Handling

The possession and handling of samples will be documented from the time of collection to delivery to the laboratory. GZA field personnel are responsible for ensuring that COC procedures are followed. Field personnel will maintain custody of the samples until they are relinquished to another custodian, the laboratory, or to the freight shipper with a custody seal on the cooler.

Samples must be catalogued on a COC form using sample identification codes. A copy of the ECCS COC form is provided in Appendix F. The date and time of collection will be recorded on the form, as well as the number of each type of sample, the method of preservation and the type of analysis. The COC SOP is provided in Appendix E.

2.3.1.4 Field Sample Packaging and Shipping

Samples will be packaged and transported in a manner that maintains the integrity of the sample and permits the analysis to be performed within the prescribed holding time. Prior to shipment, each sample container will be inspected for a label with the proper sample identification code.

Samples will be either shipped either via courier or via overnight delivery to ECCS. The laboratory will be contacted in advance to expect shipment so that holding times of the samples will be conserved. The COC forms will be sealed in a plastic bag and transported inside the sample cooler. In addition, shipping receipts will be incorporated into the COC documentation. Samples will be packed in the cooler using bubble-wrap packing materials and ice will be sealed in a Ziploc®-type bag. Samples suspected of being highly contaminated will additionally be sealed in a Ziploc®-type bag. The cooler will be taped closed using custody seals provided by the laboratory to prevent tampering during transport when a commercial transport company is used. Upon relinquishing the sample cooler to the laboratory, GZA field personnel will sign custody of the samples over to the laboratory by signing and dating the bottom of the COC form. One copy of the COC documentation will be retained by the GZA data manager and a second copy will be retained by the laboratory. The integrity of the custody seals shall be noted by the laboratory on the COC form upon arrival.

2.3.1.5 Field Documentation

Field COC procedures will be followed for the proper documentation of each sample from collection in the field to delivery at the laboratory. Custody of samples will be maintained and documented at all times. The documentation for each sample will include the following information:

- COC form;
- Sample label with sample identification code; and
- Shipping documents.

This documentation will allow for proper identification and verification of samples upon arrival at the project laboratory.

2.3.2 Laboratory Chain-of-Custody

ECCS will perform laboratory custody procedures for sample receiving and log-in, sample storage, tracking during sample preparation and analysis, and storage of data in accordance with their SOPs. The laboratory Project Manager will be responsible for ensuring that laboratory COC protocol is maintained. The SOPs for sample custody are presented in the laboratory QA manuals provided in Appendix D.

2.3.3 Final Evidence Files Custody Procedure

GZA will be responsible for the custody of the evidence files and maintain and update the contents of the files during the project. The evidence files will include records relevant to sampling and analysis activities such as boring logs, field books, photographs, subcontractor reports, laboratory data deliverables, COC forms and data reviews. GZA will retain this file for a period of time required under the AOC.

2.4 QUALITY CONTROL REQUIREMENTS

The QC requirements are followed so that the environmental data collected is of the highest standard feasible, as appropriate for the intended application. Facets of the QC requirements are provided in the following sections.

2.4.1 Field Quality Control Requirements

Where applicable, QC checks will be strictly followed during the assessment through the use of replicate measurements, equipment calibration checks and data verification by GZA field personnel. Field-sampling precision and data quality will be evaluated through the use of sample duplicates, equipment blanks and trip blanks. Sample duplicates provide precision information regarding homogeneity, handling, transportation, storage and analysis. Equipment blanks will be used to document that proper decontamination procedures have been performed and that cross-contamination is minimized during sampling or transportation. Trip blanks will be used with groundwater samples targeted for VOCs, to evaluate whether storage and transportation of samples may have contaminated the samples. Methanol blanks will be used with soil samples targeted for VOCs only to evaluate whether use, storage or transportation of samples may have contaminated the samples. If there is any discrepancy in the sample data, the GZA project manager will be notified and, if deemed necessary, re-sampling of the questionable point scheduled. Requirements for field QA/QC samples are listed in Table 4.

2.4.2 Laboratory QC Requirements

The laboratory QA Manager will be responsible for ensuring that the laboratory's data precision and accuracy are maintained in accordance with specifications. Internal laboratory duplicates and calibration checks are performed on one of every 20 samples submitted for analysis. Other internal laboratory QA/QC is performed according to laboratory SOP. Soil and water samples that are submitted for laboratory MS/MSD or spike and duplicate analyses will have an additional set of samples collected from the sample locations. In the case of VOCs, double the amount will be collected. Typically, laboratories require two to three sample containers for each sample location, therefore, four to six sample containers will be collected for laboratory MS/MSD analyses. If soil VOCs are preserved in the field with methanol, additional sample volume is not required for the MS/MSD analyses.

2.5 INSTRUMENT CALIBRATION AND FREQUENCY

The calibration procedures to be employed for both the field and laboratory instruments are referenced in this section. Measuring and testing equipment used in the field and laboratory will be subjected to a formal calibration program. The program will require equipment of the proper type, range, accuracy and precision to provide data compatible with the specified requirements and the desired results. Calibration of measuring and test equipment may be performed internally using in-house reference standards, or externally by agencies or manufacturers.

The responsibility for the calibration of laboratory equipment rests with project laboratories. GZA field personnel are responsible for the calibration of GZA and rental field equipment.

Documented and approved procedures will be used for calibrating measuring and testing equipment. Widely accepted procedures, such as those published by the USEPA and American Society for Testing and Materials (ASTM), or procedures provided by manufacturers in equipment manuals, will be adopted.

Calibrated equipment will be uniquely identified by the manufacturer's serial number, a GZA equipment identification number, or by other means. This identification, along with a label indicating when the next calibration is due (only for equipment not requiring daily calibration), will be attached to the equipment. If this is not possible, records traceable to the equipment will be readily available for reference. It will be the responsibility of all equipment operators to check the calibration status from the due date labels or records prior to using the equipment.

Measuring and testing equipment will be calibrated at prescribed intervals and/or as part of operational use. Frequency will be based on the type of equipment, inherent stability, manufacturer's recommendations, values given in national standards, intended use and experience. Equipment will be calibrated whenever possible using reference standards having known relationships to nationally recognized standards or accepted values of physical constants. If national standards do not exist, the basis for calibration will be documented.

Physical and chemical reference standards will be used only for calibration. Equipment that fails calibration or becomes inoperable during use will be removed from service, segregated to prevent inadvertent use and tagged to indicate the fault. Such equipment will be recalibrated and repaired to the satisfaction of the laboratory personnel or GZA field personnel, as applicable. Equipment that cannot be repaired will be replaced.

Records will be prepared and maintained for each piece of calibrated measuring and test equipment to document that established calibration procedures have been followed. Records for rented field equipment and GZA equipment used for this specific project will be kept in the project files. The project laboratories will maintain laboratory calibration records.

2.5.1 Field Instrument Calibration

Instruments used to gather, generate, or measure field environmental data will be calibrated with sufficient frequency and in such manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Field measurement instruments will include PID units used to detect VOC vapors, pH meters and conductivity meters. As applicable, field instruments will be calibrated daily prior to use. The calibration will be consistent with the standard procedure. The field calibration procedures are presented in the field SOPs provided in Appendix E.

Calibration procedures will be documented in the field logbook and field sampling sheets. Documentation will include the following:

- Date and time of calibration;
- Identity of the person performing the calibration;
- Reference standard used, if applicable;
- Reading taken and adjustments to attain proper reading; and
- Corrective action, if required.

Trained personnel will operate field measurement equipment in accordance with the appropriate standard procedures or manufacturer's specifications. GZA field technical staff members will examine field measurement equipment used during field sampling to verify that they are in operating condition. The GZA field team leader will periodically audit the calibration and field performance of the field equipment to show that the system of field calibration meets the manufacturer's specifications.

2.5.2 Laboratory Instrument Calibration

The proper calibration of laboratory equipment is a key element in the quality of the analysis done by the laboratory. Each type of instrumentation and each USEPA-approved method have specific requirements for the calibration procedures, depending on the analytes of interest and the sample medium.

The calibration procedures and frequencies of the equipment used to perform the analyses will be in accordance with requirements established by the USEPA. The laboratory QA Manager will be responsible for ensuring that the laboratory instrumentation is maintained in accordance with specifications. Individual laboratory SOPs will be followed for corrective actions and preventative maintenance frequencies. Laboratory QC, calibration procedures and corrective

action procedures are discussed in the individual QA Manuals, which are provided in Appendix D.

2.6 DATA MANAGEMENT

GZA field technical staff members will manage raw data during field activities. Data, such as geologic profiles, field-screen readings and pH readings, will be recorded on the appropriate field forms (examples of which are located in Appendix F) or in field logbooks. The GZA Data Manager will periodically collect data gathered during the investigation in order to maintain results. As appropriate, the GZA data manager will coordinate transfer of raw data to computer formats such as Microsoft® Excel to better organize and track incoming data. This will enable the GZA Data Manager to identify data gaps. Flaws in field QA/QC will be brought to the attention of the GZA QA Manager.

The individual laboratory Project Managers will be responsible for laboratory data management. Laboratory procedures for data review and data reporting are discussed in the various laboratory QA Manuals provided in Appendix D. Analytical data reports generated by the individual laboratories will present sample results, including QA/QC samples. The data reports will include a laboratory narrative for the data set describing any out of control analyses and their effect on sample results, an explanation of laboratory-applied qualifiers, sample results including the percent moisture content for soil samples, the method blank results, the calibration blank results, the spike and duplicate analysis results (or MS/MDS results) including the percent recoveries and RPDs, the lab control sample (LCS) results including percent recoveries, summaries of daily calibration check samples (including notation of any outliers), surrogate results including percent recoveries (as applicable per analysis), etc. Soil results will be reported on a dry weight basis. The data, including QA/QC results, will become part of the project files and will be maintained by the GZA Data Manager. Upon report delivery, GZA personnel will analyze laboratory data in accordance with accepted statistical methodologies and will be supervised by the GZA Data Manager.

3.0 ASSESSMENT/OVERSIGHT

Performance and system audits will be conducted to document that the field sampling activities and laboratory analyses are performed following the procedures established in this QAPP, including in the attached SOPs. The audits may be both internally and externally led, as further described below.

3.1 TECHNICAL SYSTEMS AUDITS

Generally, system audits are a qualitative measure of adherence to sampling QA measures overall, including sample collection handling, decontamination procedures, COC and recording requirements in the field, as well as sample receiving, log-in and instrument operating records in the laboratory.

3.1.1 Field Data

A GZA field technician will be present at the site during sampling activities. The GZA representative will provide the on-site supervision required during the project and will be in daily contact with the GZA field team leader who will then review compliance with the project objectives and sampling protocol outlined in this QAPP. Any anticipated modifications to the sampling or measuring procedures will be reported to the GZA Project Manager. GZA field technical staff members will report modifications to the GZA Project Manager and document the modification in the field logbook.

Sample data precision will be determined by the collection and subsequent analysis of sample duplicates, equipment blanks and trip blanks to verify reproducibility.

3.1.2 Field Screening Instruments

GZA field technical staff members will audit and maintain the performance field-screening instruments. Instruments will be calibrated according to the standard procedures, and regular preventative maintenance will be performed as described in Table 6.

3.1.3 Report Preparation

Prior to submittal to the USEPA, reports will undergo a peer review conducted by a project team within GZA and with the Wedron Silica and Lockheed Martin Project Managers.

3.1.4 Laboratory Data

Laboratory results will be reviewed for compliance against the DQO criteria for the level of reporting required.

3.2 PERFORMANCE EVALUATION AUDITS

Generally, performance audits are a quantitative measure of field sample collection and laboratory analyses quality.

3.2.1 Field Audits

The GZA QA Manager will conduct audits of field activities. At least one field audit will be conducted near the beginning of the sample collection activities. If a second phase of field activities is necessary and the second phase starts more than six months following the initial phase, then a second field audit will be conducted. The field audit will include the following checklist:

Item	Description of Field Audit Activities	QA Manager Initials
1.	Review of field-sampling records	
2.	Review of field-measurement procedures	
3.	Examination of the application of sample identifications following the specified protocol	
4.	Review of field instrument calibration records and procedures	
5.	Recalibration of field instruments to verify calibration to the manufacturer's specifications	
6.	Review of the sample handling and packaging procedures	
7.	Review of COC procedures	

If deficiencies are observed during the audit, the deficiency shall be noted in writing and a follow-up audit may be completed if deemed necessary by the Project QA Manager. Corrective action procedures may need to be implemented due to the findings from the audit. Such actions will be documented in the field logbook.

3.2.2 Laboratory Audits

The laboratories will perform the analytical services required during the assessments. As discussed in Section 1.4.1.2, ECCS and Pace are Illinois-certified laboratories. Copies of the applicable laboratory certificates are provided in Appendix C. The laboratory QA managers will be responsible for ensuring that the laboratory data precision and accuracy are maintained in accordance with specifications and laboratory SOPs.

4.0 DATA VALIDATION/USABILITY

This section describes the QA activities that will be performed so that the data collected are scientifically defensible, properly documented and of known quality, and meet project objectives.

The following three steps will be followed to show that project data quality needs are met.

1. **Data Verification** - Data verification is a process of evaluating the completeness, correctness and contractual compliance of a data set against the method standard, SOP, or contract requirements. Data verification will be performed internally by the analytical group or laboratory generating the data. Additionally, data may be checked by an entity external to the analytical group or fixed laboratory. Data verification may result in accepted, qualified, or rejected data.

2. **Data Validation** - Data validation is an analyte- and sample-specific process that extends the qualification of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of specific data sets. Data validation criteria are based on the measurement performance criteria of the project QAPP. LDC, the Third-Party Data Validator, will perform data validation. Data validation results are accepted, qualified, or rejected data.

3. **Data Usability Assessment** - Data usability assessment is the process of evaluating validated data to determine if the data can be used for purposes of the project. Data usability will include the following sequence of evaluation:

    - First, individual data sets will be evaluated to identify the measurement performance/usability issues or problems affecting the ultimate achievement of project DQOs.

    - Second, an overall evaluation of the data generated for the project will be performed.

    - Finally, the project-specific measurement performance criteria and data validation criteria will be evaluated to determine if they were appropriate for meeting project DQOs.

In order to perform the data evaluation steps above, the reported data will be supported by complete data packages, which include sample receipt and tracking information, COC records, tabulated data summary forms and raw analytical data for field samples, standards, QC checks and QC samples, and other project-specific documents that are generated.

4.1 INSTRUCTIONS FOR DATA REVIEW, VALIDATION AND VERIFICATION REQUIREMENTS

This section describes the process for documenting the degree to which the collected data meet the project objectives, individually and collectively. GZA will estimate the potential effect that each deviation from this QAPP may have on the usability of associated data items, its contribution to the quality of reduced and analyzed data and its effects on the decision.

The following procedures will be implemented to verify and validate data collected during the project:

- *Sampling Design* - How closely a measurement represents the actual environment at a given time and location is a complex issue. Each sample will be checked for compliance with the specifications, including type and location. GZA will note deviations from the specifications. Deviations of the sampling design (e.g. boring locations, sample depth, etc.) required as a result of field conditions will be documented.

- *Sample Collection Procedures* - Sample collection procedures identified in this QAPP will be followed. Deviations required as a result of field conditions will be documented.

- *Sample Handling* - Deviations from the planned sample handling procedures will be noted on the COC forms and in the field logbooks. Data collection activities will indicate the events that occurred during sample handling affecting the integrity of the samples.

  GZA field technical staff members will evaluate the sample containers and the preservation methods used to confirm that they are appropriate to the nature of the sample and the type of data generated from the sample. Checks on the identity of the sample will be made so that that the sample continues to be representative of its native environment as it moves through the analytical process.

- *Analytical Procedures* - Each sample will be verified to confirm that the procedures used to generate the data were implemented as specified. Data validation activities will be used to determine how seriously a sample deviated beyond the acceptance limit so that the potential effects of the deviation can be evaluated.

- *Quality Control* - QC checks that are to be performed during sample collection, handling and analysis are previously specified in this QAPP. For each specified QC check, the procedures, acceptance criteria and corrective action should be specified. During data validation, the corrective actions that were taken, which samples were affected and the potential effect of the actions on the validity of the data will be documented.

- *Calibration* - Field and laboratory instrument calibrations will be documented to confirm that calibrations:

  - Were performed within an acceptance time prior to generation of measurement data;

  - Were performed in proper sequence;

  - Included the proper number of calibration points;

  - Were performed using a standard that bracketed the range of reported measurement results as required; and

  - Had acceptable linearity checks and other checks to show that the measurement system was stable when calibration was performed.

  When calibration problems are identified, any data produced between the suspect calibration event and any subsequent recalibration will be flagged to alert data users.

- *Data Reduction and Processing* - Checks on data integrity will be performed to evaluate the accuracy of raw data and include the comparison of important events and duplicate re-keying of data to identify data entry errors. Refer to the QA Manuals in Appendix D for the laboratory data reduction procedures.

4.2 INSTRUCTIONS FOR VALIDATION AND VERIFICATION METHODS

This section describes the process that will be followed to verify and validate the project data.

4.2.1 Verification

Field data will be verified by the GZA QA Manager by reviewing field documentation and COC records. Data from direct-reading instruments used to measure organic vapors, conductivity and pH will be internally verified by reviewing calibration and operating records. The laboratory data will be verified in respect to the COC, units of measure and citation of analytical methods. Data verification procedures followed by the laboratories are provided in their respective QA Manuals (Appendix D) and will include reviewing and documenting sample receipt, sample preparation, sample analysis (including internal QC checks), data reduction and reporting. Any deviations from the acceptance criteria corrective actions taken, and data determined to be of limited usability (i.e., laboratory-qualified data), will be noted in the case narrative of the laboratory report. The QA manager will also verify the use of blanks and duplicates. The applicable reference and identification codes and numbers will be reviewed as part of the documentation.

4.2.2 Validation

Data validation will be conducted by LDC consistent with USEPA Level 3 and Level 4 data validation elements and its procedures and provided in Appendix G. Level 3 validation will be conducted on 100% of the data generated and Level 4 validation will be conducted on 10% of the data generated. The data verification/validation procedure will identify data as being acceptable, of limited usability qualified or estimated, or rejected. The results of the data validation will be provided in a data validation report provided to GZA's Project Manager. Sampling, handling, field analytical data and fixed-laboratory data will be validated by entities external to the data generator.

Data determined to be unusable may require that corrective action be taken. Potential types of corrective action may include re-sampling by the field team or re-analysis of the samples by the laboratory. The corrective actions taken are dependent on the ability to mobilize the field team and whether the data are critical for the project DQOs to be achieved. Should GZA's QA officer identify a situation requiring corrective action during data verification/validation, GZA's Project Manager will be responsible for approving the implementation of the corrective action.

4.3 INSTRUCTIONS FOR RECONCILIATION WITH DATA QUALITY OBJECTIVES

This section describes the scientific and statistical procedures/methods that will be used to determine whether data are of the right type, quality and quantity to support environmental decision making for the project.

The Data Quality Assessment (DQA) process is described in *Guidance for the Data Quality Assessment Process: Practical Methods for Data Analysis*, EPA QA/G-9, July 1996. EPA QA/G-9 will be used to guide the data assessment on this project. The DQA process will consist of five steps:

1. Review DQOs and sampling design;

2. Conduct preliminary data review;

3. Select statistical test;

4. Verify assumptions; and

5. Draw conclusions from the data.

While the formal DQA process presented in the guidance may not be followed in its entirety, a systematic assessment of the data quality will be performed. This process will

include a preliminary data review. Data will be presented in tables and figures to identify the trends, relationships and anomalies.

The overall usability of the data for the project will be assessed by evaluating the PARCCS of the data set to the measurement performance criteria in Section 1.6 of this QAPP using statistical quantities as applicable. The procedures and statistical formulas to be used for these evaluations are presented in the following sections.

4.3.1 Precision

In order to meet the needs of the project, data must meet the measurement performance criteria for precision. Project precision will be evaluated by assessing the RPD data from the field duplicate samples. Analytical precision will be evaluated by assessing the RPD data from either duplicate spiked sample analyses or duplicate sample analyses. The RPD between two measurements is calculated using the following simplified formula:

$$RPD = \frac{|R_1 - R_2|}{(R_1 + R_2)/2} \times 100$$

where: R1 = value of first result
R2 = value of second result

Overall precision for the sampling programs will be determined by calculating the mean RPD for field duplicates in a given sampling program. This will provide an evaluation of the overall variability attributable to the sampling procedure, sample matrix and laboratory procedures in each sampling program.

The overall precision requirement will be the same as the project precision. It should be noted that the RPD of two measurements can be very high when the data approach the quantitation limit of an analysis. The calculation of the mean RPD will include only the RPD values for field duplicate sample analyte data that are greater than or equal to five times the quantitation limit for an analysis.

Poor overall precision may be the result of one or more of the following:

- Field instrument variation;
- Analytical measurement variation;
- Poor sampling technique;
- Sample transport problems; and
- Heterogeneous matrices.

In order to identify the cause of the imprecision, the field-sampling design rationale and sampling techniques should be evaluated by the reviewer, and both field and analytical duplicate/replicate sample results should be reviewed. If poor precision is indicated in both the field and analytical duplicates/replicates, then the laboratory may be the source of error. If poor precision is limited to the field duplicate/replicate results, then the sampling technique, field instrument variation, sample transport, or heterogeneous sample matrices may be the source of error.

If the data validation report indicates that analytical imprecision exists for a particular data set, then the impact of that imprecision on data usability must be discussed in the DAR. It should be noted that the data validation report is considered to be the QA/QC report supplied by the analytical laboratory and supplemented with the results provided by the Third-Party data validator and the DAR will be prepared by GZA and submitted as part of the report.

When project-required precision is not achieved and project data are not usable to adequately address environmental questions and to support project decision-making, then the DAR should address how this problem will be resolved and discuss the need for re-sampling.

4.3.2 Accuracy/Bias

In order to meet the needs of the data users, project data will follow the measurement performance criteria for accuracy/bias established in Section 1.6.2.

4.3.2.1 Sample Contamination

QC check sample data will be reviewed to evaluate the accuracy and potential bias of sample results. If field contamination exists, then the impact of field contamination on data usability will be discussed in the DAR, and the GZA Project Manager and field team leader should be notified. Differentiate field sample collection and transport contamination from contamination introduced at the time of sample preparation and analysis. Note that sample contamination may result in either negative or positive bias. For example, improperly cleaned sample containers for metals analysis may result in the retention of metals on interior container walls. This would result in lower metals concentrations being reported than are actually present in the environmental sample, which is a negative bias. A positive bias would occur when sample container contamination results in an additive effect, meaning that reported analyte concentrations are higher than the true sample concentrations for that analyte.

4.3.2.2 Analytical Accuracy/Bias

The data from method/preparation blank samples, field blank samples, trip blank sample, surrogate spikes, MS/MSD samples and LCSs will be used to determine accuracy and potential bias of the sample data. If the data validation report indicates that contamination and/or analytical inaccuracies/bias exist for a particular data set, then the impact of that contamination and/or analytical inaccuracies/bias on data usability will be discussed on the DAR.

4.3.2.3 Overall Accuracy/Bias

The data from the method/preparation blank samples provide an indication of laboratory contamination that may result in bias of sample data. Sample data associated with method/preparation blank contamination will have been identified during the data verification/validation process. Sample data associated with method/preparation blank contamination are evaluated during data validation procedures to determine if analytes detected in the samples and the associated method/preparation blanks are "real" or are the result of laboratory contamination. The procedure for this evaluation involves comparing the concentration of the analyte in the sample to the concentration of the method/preparation blank taking into account adjustments for sample dilution and dry-weight reporting. In general, the sample data are qualified as not detected if the sample concentration is less than five times (10 times for common laboratory contaminants) the method/preparation blank concentration. Typically, the common quantitation limit for the affected analyte is elevated to the concentration detected in the sample.

The data from the field blanks and trip blanks provide an indication of field and transportation conditions that may result in bias of sample data. Sample data associated with contaminated field and trip blank samples have been identified during the data verification/validation process. The evaluation procedure and qualification of sample data associated with field blank and trip blank contamination is performed in the same manner as the evaluation procedure for method blank sample contamination, taking into account the difference in units for aqueous field blank samples collected during soil sampling programs.

Surrogate spike recoveries provide information regarding the accuracy/bias of the organic analyses on an individual sample bias. Surrogate compounds are not expected to be found in the samples and are added to every sample prior to sample preparation/purging. The percent recovery data provide an indication of the effect that the sample matrix may have on the preparation and analysis procedure. Sample data exhibiting matrix effects will have been identified during data verification/validation process.

Matrix spike sample data can provide information regarding the accuracy/bias of the analytical methods relative to the sample matrix. Matrix spike samples are field samples that have been fortified with target analytes prior to sample preparation and analysis. The percent recovery data provide an indication of the effect that the sample matrix may have on the preparation and analysis procedure. Sample data exhibiting matrix effects will have been identified during the data verification/validation process.

Analytical accuracy/bias will be determined by evaluating the percent recovery data of LCSs. LCSs are artificial samples prepared in the laboratory using a blank matrix that is fortified with analytes from a standard reference material that is independent of the calibration standards. LCSs are prepared and analyzed in the same manner as the field samples. The data from LCS analyses will provide an indication of the accuracy and bias of the analytical method for each target analyte.

Percent recovery is calculated using the following formula:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where: SSR = Spiked Sample Result
SR = Sample Result or Background
SA = Spike Added

The percent recovery of LCSs is determined by dividing the measured value by the true value and multiplying by 100.

Overall accuracy/bias for the sampling events will be determined by calculating the percent accuracy measurements that meet the measurement performance criteria specified in Section 1.6.2 of this QAPP. Overall accuracy will be considered acceptable if the surrogate percent recoveries are met for at least 75% of the samples and the LCS percent recoveries are met for the samples and the MS/MSD percent recoveries are met for at least 75% of the samples.

The DAR will discuss and compare overall contamination and accuracy/bias data from multiple data sets collected for the project for each matrix, analytical parameter and concentration level. The DAR will describe the limitations on the use of the project data if extensive contamination and/or inaccuracy/bias exist, or when it is limited to a specific sampling or laboratory analytical group, data set, analytical parameter, or concentration level. The DAR will identify qualitative and/or quantitative bias trends in multiple performance evaluation (PE) sample results for each matrix, analytical parameter and concentration level. The

impact of any qualitative and/or quantitative trends in bias on the sample data will be discussed. Any PE samples that have false positive and/or false negative results should be reported and the impact on data usability will be discussed in the DAR.

When project-required accuracy/bias is not achieved and project data are not usable to adequately address environmental questions and to support project decision-making, then the DAR will address how this problem will be resolved and the potential need for re-sampling.

4.3.3 Sample Representativeness

In order to meet the needs of the data users, project data must meet the measurement performance criteria to sample representativeness specified in Section 1.6.3.

Representativeness of the samples will be assessed by reviewing the results of field audits and the data from field duplicate samples. If field duplicate precision checks indicate potential spatial variability, then additional sampling may be required in order to collect data that are more representative of a non-homogeneous site. Overall sample representativeness will be determined by calculating the percent of field duplicate sample data that achieved the RPD criteria specified in Section 1.6.3 of this QAPP. Overall sample representativeness will be considered acceptable if the results of the field audits indicate that the approved sampling methods or alternate acceptable sampling methods were used to collect the samples, and the field duplicates RPD data are acceptable for at least 75% of the samples.

The DAR will discuss and compare overall representativeness for each matrix, parameter and concentration level. DARs will describe the limitations on the use of project data when overall non-representative sampling has occurred or when non-representative sampling is limited to a specific sampling group, data set, matrix, analytical parameter, or concentration level. If data are not usable to adequately address environmental questions and/or support project decision making, then the DAR will address how this problem will be resolved and discuss the potential need for re-sampling.

4.3.4 Sensitivity and Quantitation Limits

In order to meet the needs of the data user, project data must meet the measurement performance criteria for sensitivity, as specified. Low point calibration standards should produce a signal at least ten times the background noise levels and should be part of a linear calibration curve. Procedures for calculating MDLs and reporting limits (RLs) should be documented.

4.3.4.1 Overall Sensitivity and Quantitation Limits

The quantitation limits for the sample data will be reviewed to confirm that the sensitivity of the analyses was sufficient to achieve TACO Tier 1 Class I soil migration to groundwater SROs and TACO Tier 1 Class I groundwater SROs for constituents of concern. The method/preparation blank sample data and LCSs percent recovery data will be reviewed to assess compliance with the measurement performance criteria specified in Section 1.6.6.

Overall sensitivity will be assessed by comparing the sensitivity for each monitoring program to the detectability requirements for the analyses. Overall sensitivity will be considered acceptable if quantitation limits for constituents of concern are less than the applicable SROs.

It should be noted that quantitation limits may be elevated as a result of high concentrations of target compounds, non-target compounds and matrix interferences (collectively known as sample matrix effects). In these cases, the sensitivity of the analyses will be evaluated on an individual sample basis relative to the applicable evaluation criteria. The need to investigate the use of alternate analytical methods may be required if the sensitivity of the analytical methods identified in this QAPP cannot achieve the evaluation criteria because of sample matrix interference.

If the data validation report indicates that sensitivity and/or RLs were not achieved, then the impact of that lack of sensitivity and/or higher RLs on data usability will be discussed in the DAR.

The DAR will discuss and compare overall sensitivity and RLs from multiple data sets collected for the project for each matrix, analytical parameter and concentration level. The DAR will describe the limitations on the use of the project data if project-required sensitivity and RLs were not achieved for all project data or when it is limited to a specific sampling or laboratory/analytical group, data set, matrix, analytical parameter, or concentration level.

When project-related RLs are not achieved and project data are not usable to adequately address environmental questions and to support project decision making, then the DAR will address how this problem will be resolved and discuss the potential need for re-sampling. In this case, the DAR will clearly differentiate between usable and unusable data for the users.

4.3.5 Completeness

In order to meet the needs of the data users, project data will follow the measurement performance criteria for data completeness outlined in Section 1.5.4.

4.3.5.1 Overall Completeness

Completeness will be assessed by comparing the number of valid (usable) sample results to the total possible number of results within a specific sample matrix and/or analysis. Percent completeness will be calculated using the following formula:

$$\% \text{ Completeness} = \frac{\text{Number of Valid (usable) measurements}}{\text{Number of Measurements Planned}} \times 100$$

Overall completeness will be assessed by calculating the mean percent completeness for the entire set of data obtained for each sampling program. The overall completeness for the project will be calculated when all sampling and analysis are concluded. Overall completeness will be considered acceptable if at least 90% of the data are determined to be valid.

The DAR will discuss and compare overall completeness of multiple data sets collected for the project for each matrix, analytical parameter and concentration level. The DAR will describe the limitation on the use of the project data if project-required completeness was not achieved for the overall project or when it is limited to a specific sampling or laboratory/analytical group, data set, analytical parameter, or concentration level.

When project-required completeness is not achieved and sufficient data are not available to adequately address environmental questions and support project decision making, then the DAR will address how this problem will be resolved and discuss the potential need for additional re-sampling.

4.3.6 Comparability

In order to meet the needs of the data users, project data will follow the measurement performance criteria for comparability outlined in Section 1.5.5.

The comparability of data sets will be evaluated by reviewing the sampling and analysis methods used to generate the data for each data set. Project comparability will be determined to be acceptable if the sampling and analysis methods specified in this QAPP are used for generating the soil and groundwater data.

The DAR will discuss and compare overall comparability between multiple data sets collected for the project for each matrix, analytical parameter and concentration level. The DAR will describe the limitation on the use of project data when project-required data comparability is not achieved for the overall project or when it is limited to a specific sampling or laboratory/analytical group, data set, matrix, analytical parameter, or concentration level.

Overall comparability of data from split samples (samples that are collected at the same time from the same location and split equally between two parties using sample containers from the same source or vendor) will be evaluated by determining the RPD of detected analytes in both samples following data verification/validation. Analytes that are detected in only one of the two samples will be assessed by reviewing the data verification/validation reports for both data sets and determining the cause of the discrepancy. Overall comparability of split sample data will be considered acceptable if the RPD for detected analytes with concentrations greater than or equal to five times their respective quantitation limits does not exceed RPD acceptance criteria for field duplicate samples.

If screen/confirmatory comparability criteria are not met, then this will be documented in the DAR and the effect on data usability will be discussed. If oversight split-sampling comparability criteria are not met, then this will be documented in the DAR and the effect on data usability will be discussed. If data are not usable to adequately address environmental questions and/or support project decision making, then the DAR will address how this problem will be resolved and discuss potential need for re-sampling.

4.3.7 Data Limitations and Actions

Sources of sampling and analytical error will be identified and corrected as early as possible to the onset of sample collection activities. An ongoing data assessment process will be incorporated during the project, rather than just as a final step, to facilitate the early detection and correction of problems, ensuring that project quality objectives are met.

Data that do not meet the measurement performance criteria specified in this QAPP will be identified and the impact on the project quality objectives will be assessed and discussed in the DAR. Specific actions for data that do not meet the measurement performance criteria depend on the use of the data and may require that additional samples are collected or the use of the data to be restricted.

TABLES

TABLE 1
SOIL AND GROUNDWATER SAMPLING MATRIX
Wedron, Illinois

Investigation Area	Target Number of Soil Borings	Target Soil Boring Depths	Target Number of Soil Samples	Target Soil Sample Locations	Target Number of Groundwater Samples	Target Analytical Suite
Tech Center	2	Total Depth	4	Base locations on field Indications or if no such indications, collect a sample from the 6- to 8-foot depth interval and from Total Depth	0	VOCs and F_{OC}
IEPA Soil Boring GP-10 and IEPA Monitoring Well G103 Area	3	Total Depth	6	Base locations on field Indications or if no such indications, collect a sample from the 6- to 8-foot interval and from the depth interval immediately above the water table or refusal	0	VOCs and F_{OC}
Former 4,000-Gallon Gasoline USTs	4	7 feet	4	Base locations on field Indications or if no such indications, collect a sample from the 5- to 7-foot depth interval	0	VOCs, F_{OC}, lead and pH
Former 6,000-Gallon Gasoline UST	5	11 feet	5	Base locations on field Indications or if no such indications, collect a sample from the 9- to 11-foot depth interval	0	VOCs, F_{OC}, lead and pH
Pit 2 Reclamation Area	3	Total Depth	6	Base locations on field Indications or if no such indications, collect a sample from the 6- to 8-foot interval and from the depth interval immediately above the water table	3	VOCs and F_{OC}
Total No. of Borings/Samples	17		25		3	

Notes:

1. Field Indications denotes observations made from field screening, odors, staining, etc.
2. Total Depth denotes the depth of refusal or groundwater, whichever is encountered first.
3. VOCs denotes volatile organic compounds analyzed in accordance with USEPA Method 8260B.
4. Lead will be analyzed in accordance with USEPA Method 6010C.
5. pH will be analyzed in accordance with USEPA Method 9045C.
6. Fraction organic carbon (Foc) will be analyzed in accordance with ASTM D2974-87.
7. F_{oc} samples will be collected from each soil interval selected for VOC analysis and at least two soil sample from each soil type encountered in each investigation area will be submitted for Foc analysis.

TABLE 2
LABORATORY ANALYSES PERFORMED BY LABORATORY
Wedron, Illinois

Laboratory Name	Analyses to be performed/Method number/media, if other than soil and water
Environmental Chemistry Consulting Services, Inc. (ECCS, Madison, WI)	VOCs (8260B)
Pace Analytical Services, Inc. (Green Bay, WI)	Lead (6010C), pH (9045C), Fractional Organic Carbon (F_{OC}) (ASTM D2974-87)

Notes:

1. Laboratory analyses and corresponding methods listed for each laboratory were obtained from the provided SOPs.
2. VOCs will be analyzed in soil and groundwater samples. Lead, pH, and F_{OC} will be analyzed in soil samples only.

TABLE 3a
GROUNDWATER ANALYTICAL METHOD DETECTION LIMITS, REPORTING LIMITS, AND TACO TIER I REMEDIATION OBJECTIVES
Wedron, Illinois

ANALYTE AND US EPA METHOD	CAS Registry No.	TACO Tier I Class I Groundwater Remediation Objectives	Groundwater Method Detection Limit	Groundwater Reporting Limit	MS/MSD	MS/MSD
VOCs (8260B)		**µg/L**	**µg/L**	**µg/L**	**% R**	**% RPD**
1,1,1-Trichloroethane	71-55-6	200	0.10	0.5	63.3 - 152	20
1,1,2,2-Tetrachloroethane	79-34-5	4.3	0.099	0.5	75.6 -130	20
1,1,2-Trichloroethane	79-00-5	5	0.10	0.5	78.3 128	20
1,1,2-Trichlorotrifluoroethane	76-13-1	210,000	0.13	0.5	61.9 -152	20
1,1-Dichloroethane	75-34-3	700	0.12	0.5	76.5 - 139	20
1,1-Dichloroethene	75-35-4	7	0.14	0.5	66.6 - 141	20
1,2,3-Trichlorobenzene	87-61-6	5.6	0.045	2	72.6 - 123	20
1,2,4-Trichlorobenzene	120-82-1	70	0.077	2	72.4 - 123	20
1,2-Dibromo-3-chloropropane	96-12-8	0.2	0.25	0.5	59.6 - 138	20
1,2-Dibromoethane (EDB)	106-93-4	0.05	0.13	0.5	78.4 - 129	20
1,2-Dichlorobenzene	95-50-1	600	0.076	0.5	88.3 - 114	20
1,2-Dichloroethane	107-06-2	5	0.078	0.5	63 - 155	20
1,2-Dichloropropane	78-87-5	5	0.10	0.5	79.5 - 125	20
1,3-Dichlorobenzene	541-73-1	NE	0.096	0.5	88.1 - 114	20
1,4-Dichlorobenzene	106-46-7	NE	0.070	0.5	88.7 - 113	20
2-Butanone (Methyl ethyl ketone)	78-93-3	4,200	3.0	20	58.8 - 141	20
2-Hexanone	591-78-6	35	0.95	20	69.8 - 131	20
4-Methyl-2-pentanone	108-10-1	560	0.77	20	75 - 127	20
Acetone	67-64-1	6,300	3.4	20	36.4 - 159	20

TABLE 3a
GROUNDWATER ANALYTICAL METHOD DETECTION LIMITS, REPORTING LIMITS, AND TACO TIER I REMEDIATION OBJECTIVES
Wedron, Illinois

ANALYTE AND US EPA METHOD	CAS Registry No.	TACO Tier I Class I Groundwater Remediation Objectives	Groundwater Method Detection Limit	Groundwater Reporting Limit	MS/MSD	MS/MSD
VOCs (8260B)		**µg/L**	**µg/L**	**µg/L**	**% R**	**% RPD**
Benzene	71-43-2	5	0.089	0.5	79 - 128	20
Bromochloromethane	74-97-5	NE	0.31	0.5	71.4 - 138	20
Bromodichloromethane	75-27-4	0.2	0.077	0.5	65.1 - 142	20
Bromoform	75-25-2	1	0.088	0.5	46.4 - 145	20
Bromomethane (methyl bromide)	74-83-9	9.8	0.59	5	27.7 - 194	20
Carbon disulfide	75-15-0	700	0.053	0.5	70.9 - 136	20
Carbon tetrachloride	56-23-5	5	0.038	0.5	44.7 - 168	20
Chlorobenzene	108-90-7	100	0.073	0.5	88.8 - 116	20
Chloroethane	75-00-3	NE	0.25	5	52.8 - 164	20
Chloroform	67-66-3	0.2	0.062	0.5	70.6 - 142	20
Chloromethane (methyl chloride)	74-87-3	NE	0.16	2	64.4 - 141	20
cis-1,2-Dichloroethene	156-59-2	70	0.11	0.5	73.6 - 134	20
cis-1,3-Dichloropropene[4]	10061-01-5	1[4]	0.061	0.5	69.6 - 132	20
Dibromochloromethane	124-48-1	140	0.091	0.5	60.2 - 140	20
Dichlorodifluoromethane	75-71-8	1,400	0.11	0.5	28.3 - 181	20
Ethylbenzene	100-41-4	700	0.054	0.5	88 - 123	20
Isopropylbenzene	98-82-8	700	0.081	0.5	86.7 - 124	20
m,p-Xylene[5]	108-38-3, 106-42-3	10,000[5]	0.057	1	88.3 - 114	20
Methyl t-Butyl Ether	1634-04-4	NE	0.14	0.5	64.7 - 140	20

TABLE 3a
GROUNDWATER ANALYTICAL METHOD DETECTION LIMITS, REPORTING LIMITS, AND TACO TIER I REMEDIATION OBJECTIVES
Wedron, Illinois

ANALYTE AND US EPA METHOD	CAS Registry No.	TACO Tier I Class I Groundwater Remediation Objectives	Groundwater Method Detection Limit	Groundwater Reporting Limit	MS/MSD	MS/MSD
VOCs (8260B)		**µg/L**	**µg/L**	**µg/L**	**% R**	**% RPD**
Methylene chloride	75-09-2	5	0.14	2	62.4 - 137	20
o-Xylene[5]	95-47-6	10,000[5]	0.058	0.5	84 - 115	20
Styrene	100-42-5	100	0.065	0.5	76.5 - 118	20
Tetrachloroethene	127-18-4	5	0.081	0.5	71.7 - 123	20
Toluene	108-88-3	1,000	0.053	0.5	73 - 121	20
trans-1,2-Dichloroethene	156-60-5	100	0.11	0.5	69.8 - 140	20
trans-1,3-Dichloropropene[4]	10061-02-6	1[4]	0.096	0.5	54.6 - 152	20
Trichloroethene	79-01-6	5	0.062	0.5	76.3 - 129	20
Trichlorofluoromethane	75-69-4	2,100	0.13	0.5	69.4 - 148	20
Vinyl chloride	75-01-4	2	0.16	0.5	72.9 - 137	20

Notes:
1. Laboratory limits provided are compared to Illinois Tiered Approach to Corrective Action Objectives (TACO) Tier I Class I Groundwater Remediation Objectives.
2. Concentrations are provided in micrograms per liter (µg/L), as shown.
3. "NE" = Standard is not established for the parameter.
4. The laboratory limits for cis-1,2-Dichloropropene and trans-1,2-Dichloropropene were provided separately by the laboratory. However, the TACO Tier I Class I Groundwater Remediation Objective (1 µg/L) is for 1,2-Dichloropropene and should therefore be compared to the combined sum of the cis- and trans-1,2-Dichloropropene concentrations.
5. The laboratory limits for m,p-Xylene and o-Xylene were provided separately by the laboratory. However, the TACO Tier I Class I Groundwater Remediation Objective (10,000 µg/L) is for Xylenes and should therefore be compared to the combined sum of the m,p-Xylene and o-Xylene concentrations.

TABLE 3b
SOIL ANALYTICAL METHOD DETECTION LIMITS, REPORTING LIMITS, AND TACO TIER I REMEDIATION OBJECTIVES
Wedron, Illinois

ANALYTE AND US EPA METHOD	CAS Registry No.	TACO Tier I Soil Component of Groundwater Ingestion Remediation Objectives	Soil Method Detection Limit	Soil Reporting Limit	MS/MSD	MS/MSD
VOCs (8260B)		**µg/kg**	**µg/kg**	**µg/kg**	**% R**	**% RPD**
1,1,1-Trichloroethane	71-55-6	2,000	7.3	25	86.7 - 126	20
1,1,2,2-Tetrachloroethane	79-34-5	3.5	6	25	59.4 - 148	20
1,1,2-Trichloroethane	79-00-5	20	6.3	25	89 - 122	20
1,1,2-Trichlorotrifluoroethane	76-13-1	450,000	4.1	25	58.7 - 157	20
1,1-Dichloroethane	75-34-3	23,000	9.3	25	81.7 - 139	20
1,1-Dichloroethene	75-35-4	60	7.3	25	78.2 - 131	20
1,2,3-Trichlorobenzene	87-61-6	460	5.5	100	74.8 - 125	20
1,2,4-Trichlorobenzene	120-82-1	5,000	5.5	100	84.8 - 119	20
1,2-Dibromo-3-chloropropane	96-12-8	2	11	25	61.7 - 142	20
1,2-Dibromoethane (EDB)	106-93-4	0.4	4.9	25	82.8 - 124	20
1,2-Dichlorobenzene	95-50-1	17,000	2.7	25	87.8 - 117	20
1,2-Dichloroethane	107-06-2	20	4.6	25	71.7 - 142	20
1,2-Dichloropropane	78-87-5	30	7.7	25	81 - 126	20
1,3-Dichlorobenzene	541-73-1	NE	4.7	25	88.3 - 114	20
1,4-Dichlorobenzene	106-46-7	NE	4	25	86.1 - 119	20
2-Butanone (Methyl ethyl ketone)	78-93-3	17,000	180	1,000	77.3 - 139	20
2-Hexanone	591-78-6	160	29	1,000	76.3 - 134	20
4-Methyl-2-pentanone	108-10-1	2,500	39	1,000	82.6 - 129	20

TABLE 3b
SOIL ANALYTICAL METHOD DETECTION LIMITS, REPORTING LIMITS, AND TACO TIER I REMEDIATION OBJECTIVES
Wedron, Illinois

ANALYTE AND US EPA METHOD	CAS Registry No.	TACO Tier I Soil Component of Groundwater Ingestion Remediation Objectives	Soil Method Detection Limit	Soil Reporting Limit	MS/MSD	MS/MSD
VOCs (8260B)		**µg/kg**	**µg/kg**	**µg/kg**	**% R**	**% RPD**
Acetone	67-64-1	25,000	150	1,000	80.7 - 148	20
Benzene	71-43-2	30	1.6	25	71.2 - 129	20
Bromochloromethane	74-97-5	NE	9.6	25	78.6 - 133	20
Bromodichloromethane	75-27-4	600	3.4	25	85.6 - 122	20
Bromoform	75-25-2	800	15	25	90 - 115	20
Bromomethane (methyl bromide)	74-83-9	200	250	---	42.1 - 191	20
Carbon disulfide	75-15-0	32,000	2.3	25	66 - 130	20
Carbon tetrachloride	56-23-5	70	4.1	25	62.9 - 157	20
Chlorobenzene	108-90-7	1,000	3.7	25	78.1 - 127	20
Chloroethane	75-00-3	NE	250	250	50.8 - 198	20
Chloroform	67-66-3	600	3.8	25	77.2 - 135	20
Chloromethane (methyl chloride)	74-87-3	NE	7.9	50	56.5 - 154	20
cis-1,2-Dichloroethene	156-59-2	400	8	25	56.5 - 155	20
cis-1,3-Dichloropropene[(4)]	10061-01-5	4[(4)]	5.6	25	72.3 - 126	20
Dibromochloromethane	124-48-1	400	5.3	25	81 - 117	20
Dichlorodifluoromethane	75-71-8	43,000	5.1	25	44.3 - 175	20
Ethylbenzene	100-41-4	13,000	2.1	25	70.4 - 132	20
Isopropylbenzene	98-82-8	91,000	2.3	25	90.6 - 111	20
m,p-Xylene	108-38-3, 106-42-3	210,000	3.1	50	83.3 - 117	20

TABLE 3b
SOIL ANALYTICAL METHOD DETECTION LIMITS, REPORTING LIMITS, AND TACO TIER I REMEDIATION OBJECTIVES
Wedron, Illinois

ANALYTE AND US EPA METHOD	CAS Registry No.	TACO Tier I Soil Component of Groundwater Ingestion Remediation Objectives	Soil Method Detection Limit	Soil Reporting Limit	MS/MSD	MS/MSD
VOCs (8260B)		**µg/kg**	**µg/kg**	**µg/kg**	**% R**	**% RPD**
Methyl t-Butyl Ether	1634-04-4	NE	4.3	25	80.5 - 130	20
Methylene chloride	75-09-2	20	7	100	40.2 - 153	20
o-Xylene	95-47-6	190,000	3	25	11.6 - 167	20
Styrene	100-42-5	4,000	4	25	82.3 - 117	20
Tetrachloroethene	127-18-4	60	5.7	25	74.5 - 127	20
Toluene	108-88-3	12,000	4	25	76.4 - 121	20
trans-1,2-Dichloroethene	156-60-5	700	4.5	25	52.3 - 166	20
trans-1,3-Dichloropropene[(4)]	10061-02-6	4[(4)]	4.3	25	84.7 - 115	20
Trichloroethene	79-01-6	60	4.1	25	73.8 - 133	20
Trichlorofluoromethane	75-69-4	34,000	5.4	25	56.5 - 168	20
Vinyl chloride	75-01-4	100	5.8	25	64.3 - 137	20

Notes:
1. Laboratory limits provided are compared to Illinois Tiered Approach to Corrective Action Objectives (TACO) Tier I Soil Component of Groundwater Ingestion Remediation Objectives.
2. Concentrations are provided in micrograms per kilogram (µg/kg), as shown.
3. "NE" = Standard is not established for the parameter.
4. The laboratory limits for cis-1,2-Dichloropropene and trans-1,2-Dichloropropene were provided separately by the laboratory. However, the TACO Soil Component of Groundwater Ingestion Remediation Objective (4 µg/kg) is for 1,2-Dichloropropene and should therefore be compared to the combined sum of the cis- and trans-1,2-Dichloropropene concentrations.

TABLE 3c
SOIL ANALYTICAL METHOD DETECTION LIMITS, REPORTING LIMITS, AND TACO TIER I REMEDIATION OBJECTIVES
Wedron, Illinois

ANALYTE AND US EPA METHOD	CAS Registry No.	TACO Tier I Soil Component of Groundwater Ingestion Remediation Objectives pH = 4.5 to 6.24	TACO Tier I Soil Component of Groundwater Ingestion Remediation Objectives pH = 6.25 to 8.74	TACO Tier I Soil Component of Groundwater Ingestion Remediation Objectives pH = 8.75 to 9.0	Soil Method Detection Limit	Soil Reporting Limit	MS/MSD	MS/MSD
Lead (6010C)		**mg/kg**	**mg/kg**	**mg/kg**	**mg/kg**	**mg/kg**	**% R**	**% RPD**
Lead	7439-92-1	23	107	282	0.2927	1.0	75-125	20

Notes:
1. Laboratory limits provided are compared to Illinois Tiered Approach to Corrective Action Objectives (TACO) Tier I Soil Component of Groundwater Ingestion Remediation Objectives.
2. Concentrations are provided in milligrams per kilogram (mg/kg), as shown.

TABLE 3d
SOIL ANALYTICAL METHOD DETECTION LIMITS, REPORTING LIMITS
Wedron, Illinois

ANALYTE AND US EPA METHOD	CAS Registry No.	TACO Tier I Soil Component of Groundwater Ingestion Remediation Objectives	Soil Method Detection Limit	Soil Reporting Limit	MS/MSD	MS/MSD
pH (9045C)		**pH units**	**pH units**	**pH units**	**% R**	**% RPD**
pH	NA	NA	NA	NA	NA	NA
FOC (ASTM D2974-87)		**% w/w**	**% w/w**	**% w/w**	**% R**	**% RPD**
Fractional Organic Carbon (FOC)	NA	NA	0.058	0.058	NA	NA

Notes:
1. "NE" = Illinois Tiered Approach to Corrective Action Objectives (TACO) Tier I Soil Component of Groundwater Ingestion Remediation Objective is not established for the parameter.
2. "NA" = The CAS Registry Number or laboratory value is not applicable for the parameter.
3. Fractional Organic Carbon concentrations are provided in percentage weight by weight, (% w/w) as shown.

TABLE 4
FIELD AND LAB QA/QC SAMPLE REQUIREMENTS
Wedron, Illinois

	QC Sample Type	Frequency of Sample/Analysis	Details
Field Samples	Duplicate Samples	1 duplicate per 10 samples per matrix, or 1 duplicate per sample matrix if fewer than 10 samples	Duplicate sample to be collected by the same methods at the same time as the original sample. Used to verify sample and analytical reproducibility.
	Equipment Blanks	1 equipment blank per 10 samples, minimum 1 equipment blank per day per sample matrix	Distilled water placed into contact with sampling equipment. Used to assess quality of data from field sampling and decontamination procedures.
	Trip Blanks	1 trip blank per cooler containing samples for targeted VOC analysis for water samples	Laboratory prepared organic-free blank to assess potential contamination during sample container shipment and storage.
	Field Blanks	1 field blank per cooler containing samples targeted for VOC analysis for soil samples	Laboratory prepared methanol blank to assess potential contamination during sample preparation, storage and shipment.
Lab Samples	Matrix Spike/ Matrix Spike Duplicate	1 MS/MSD per 20 or fewer samples per matrix	Laboratory spiked sample to evaluate matrix and measurement methodology.
	Method Blanks	1 method blank per batch of samples prepared, or per lab SOP	Laboratory blank sample to assess potential for contamination from laboratory instruments or procedures.
	Laboratory Control Samples and Duplicates	Analyzed as per method requirements and laboratory SOPs	Evaluates laboratory reproducibility.

TABLE 5
SAMPLE CONTAINER, PRESERVATION, AND HOLDING TIME REQUIREMENTS[(1)]
Wedron, Illinois

Matrix	Analysis	Container	Preservation	Holding Time
SOIL	Volatile Organic Compounds (VOCs)	Pre-weighed 40 ml glass vial containing 10 ml of methanol. Lock and Load T handle and disposable sample container are used to collect approximately 10 grams of soil. Soil sample is then expelled from disposable container into 40 ml glass vial containing methanol.	Methanol Cool to 4° C	14 days
	Dry Weight (for VOCs)	Pre-cleaned plastic or glass container	None	None
	Lead	Pre-cleaned plastic or glass container	Cool to ≤4° C	6 months
	pH	1 – 4 oz. plastic container	Cool to 4 +/- 2° C	24 hours is recommended (laboratory must qualify results if hold time exceeds 24 hours)
	Fractional Organic Carbon (FOC)	Pre-cleaned plastic or glass container	Cool to ≤6° C	7 days
WATER	Volatile Organic Compounds	3 – 40 ml glass vials with Teflon-lined septum caps eject	No headspace, 0.5 mL 1:1 HCl Cool to 4° C	14 days

Note:
1) The information provided in Table 4 pertains to analyses performed by Environmental Chemistry Consulting Services, Inc. ('ECCS'), or its subcontractor laboratory, Pace Analytical Services, Inc. ('Pace').

TABLE 6
FIELD EQUIPMENT PREVENTATIVE MAINTENANCE
Wedron, Illinois

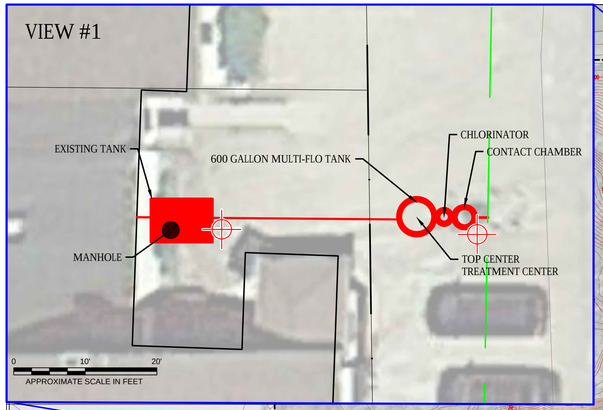
INSTRUMENTS	MAINTENANCE PROCEDURES/SCHEDULE	SPARE PARTS IN STOCK
ppbRAE 3000 Portable VOC Monitor (rental unit)	1. Unit is cleaned, charged, and calibrated by vendor prior to delivery. 2. Calibrate unit at the beginning of each day, and as necessary during use. 3. Charge battery upon returning from field work each day (fully charged battery runs instrument for 16 hours continuously). If needed, charge battery in the field when empty battery icon flashes. 4. Clean PID sensor module, lamp, and lamp housing when needed (if the reading is inaccurate even after calibration or the reading is very sensitive to moisture).	1. Battery charger 2. Calibration gas 3. Spare filter cartridges
Heron Water Level Indicator: Heron Dipper-T (100 ft)	1. Decontaminate sensor and tape at the beginning and end of each day, and as necessary during use (between each well). 2. Check battery, and replace when low. 3. Quarterly inspect sensor unit and send into factory for replacement if necessary.	1. Spare batteries
Geotech Peristaltic Pump	1. Use new polyethylene and silicone tubing at each well. Discard tubing after use. 2. Check electrical cord and associated connectors before and after each use. 3. Charge external battery before each use. 4. Decontaminate/wipe any parts of the pump that come in contact with water samples. 5. Quarterly inspect unit and send into factory for repair if necessary	1. Polyethylene and silicone tubing 2. Check valves (for sampling wells > 25 ft deep)

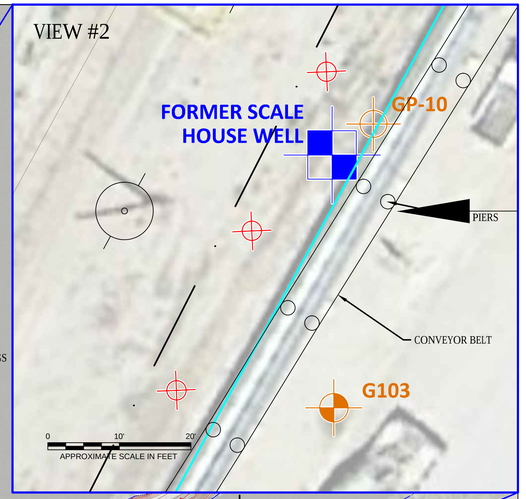
TABLE 7
QA OBJECTIVES FOR FIELD MEASUREMENTS
Wedron, Illinois

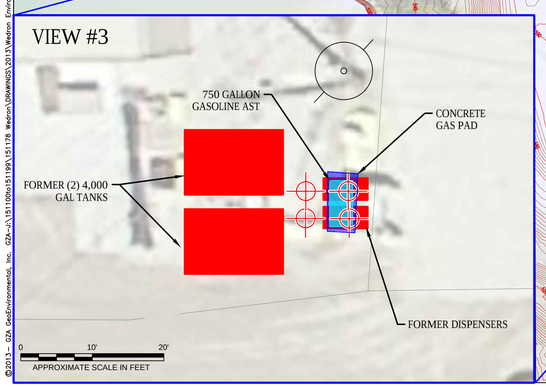
PARAMETER	EQUIPMENT	PRECISION[(1)]	ACCURACY[(1)]	COMPLETENESS
Standing Water Levels	Heron Water Level Indicator	±0.01 ft.	0.005 ft.	90%

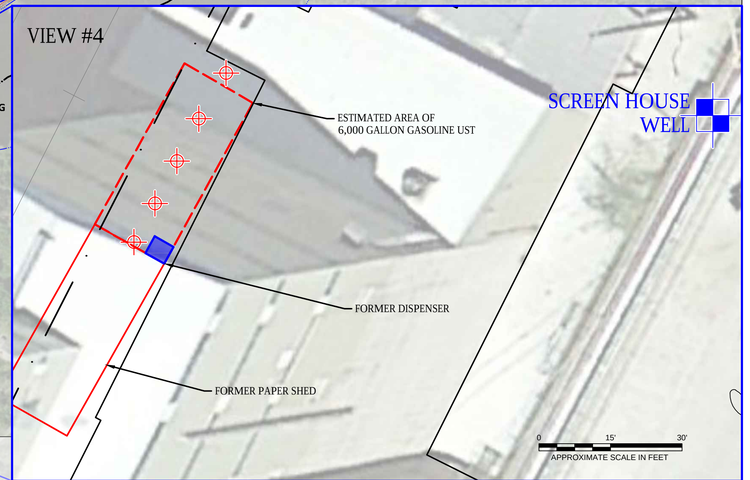
NOTES:
1. Expected based on equipment manufacturer specifications.

FIGURES









LEGEND

- GP-1 GEOPROBE BORING (GZA, 2012)
- MW-3 MONITORING WELL (GZA, 2013)
- PROPOSED GEOPROBE BORING
- PROPOSED PIEZOMETER
- PIT #2 TRANSDUCER OR STAFF GAGE (GZA, 2013)
- WEDRON SILICA PRODUCTION WELL
- GP-10 GEOPROBE BORING (WESTON/IEPA, 2012)
- TMW-6 GEOPROBE BORING AND TEMPORARY MONITORING WELL (WESTON/IEPA, 2012)
- G102 FORMER MONITORING WELL (IEPA, 1984)
- UST-5 WS-7 GEOPROBE BORING (CDM SMITH, 2012)
- TEST PITS (CIVIL & ENVIRONMENTAL CONSULTANTS, INC., 2012)
- HISTORICAL UNDERGROUND STORAGE TANK OR OTHER RELATED FEATURE
- STORMWATER CATCH BASIN
- APPROXIMATE LOCATION OF STORM SEWER
- WATER SUPPLY MAIN
- WEDRON SILICA PROPERTY BOUNDARIES
- COMMUNITY LOT LINES
- INTERMEDIATE CONTOUR (INTERVAL: 2')
- MAJOR CONTOUR (INTERVAL: 10')
- OVERHEAD POWER POLE

N

NOTES

1. BASE MAP INCLUDING TOPOGRAPHIC FEATURES (DATED NOVEMBER 2011) AND PROPERTIES BOUNDARIES WAS DEVELOPED FROM SURVEYS CONDUCTED BY YEGRZYN, SARVER AND ASSOCIATES, INC. OF OTTAWA, ILLINOIS.
2. BACKGROUND AERIAL IMAGERY IS A GOOGLE PROFESSIONAL ELECTRONIC IMAGE FILE. DIGITAL AERIAL ORTHOPHOTOGRAPHY WAS PUBLISHED BY THE U.S.G.S. DATED APRIL 2006.
3. THE USE OF AERIAL PHOTOGRAPHY CAN OFTEN MAKE BUILDINGS AND OTHER SITE FEATURES APPEAR TO BE OVERLAPPING AND DISTORTED WHEN OVERLAID WITH ACTUAL SITE FEATURES.
4. THE LOCATION OF EXPLORATIONS AND OTHER FEATURES WERE APPROXIMATELY DETERMINED BY OVERLAYING IMAGES OF HISTORIC MAPS AND AERIAL PHOTOGRAPHS, MATCHING REFERENCE POINTS, AND TRACING FEATURES. THESE LOCATIONS SHOULD BE CONSIDERED ACCURATE ONLY TO THE DEGREE IMPLIED BY THE METHOD USED.

UNLESS SPECIFICALLY STATED BY WRITTEN AGREEMENT, THIS DRAWING IS THE SOLE PROPERTY OF GZA GEOENVIRONMENTAL, INC. (GZA). THE INFORMATION SHOWN ON THE DRAWING IS SOLELY FOR USE BY GZA'S CLIENT OR THE CLIENT'S DESIGNATED REPRESENTATIVE FOR THE SPECIFIC PROJECT AND LOCATION IDENTIFIED ON THE DRAWING. THE DRAWING SHALL NOT BE TRANSFERRED, REUSED, COPIED, OR ALTERED IN ANY MANNER FOR USE AT ANY OTHER LOCATION OR FOR ANY OTHER PURPOSE WITHOUT THE PRIOR WRITTEN CONSENT OF GZA. ANY TRANSFER, REUSE, OR MODIFICATION TO THE DRAWING BY THE CLIENT OR OTHERS, WITHOUT THE PRIOR WRITTEN EXPRESS CONSENT OF GZA, WILL BE AT THE USER'S SOLE RISK AND WITHOUT ANY RISK OR LIABILITY TO GZA.

NO.	ISSUE/DESCRIPTION	BY	DATE

WEDRON PROPOSED INVESTIGATION LOCATIONS

PREPARED BY:
GZA GeoEnvironmental, Inc.
Engineers and Scientists
20900 SWENSON DRIVE, SUITE 150
WAUKESHA, WISCONSIN 53186
(262) 754-2560

PREPARED FOR:
WEDRON SILICA COMPANY
WEDRON, ILLINOIS

PROJ MGR: BGF	REVIEWED BY: MJK	CHECKED BY: MJK	FIGURE 1
DESIGNED BY: BGF	DRAWN BY: JAH	SCALE:	
DATE: September 6, 2013	PROJECT NO. 20.0151178.50	REVISION NO.	SHEET NO.

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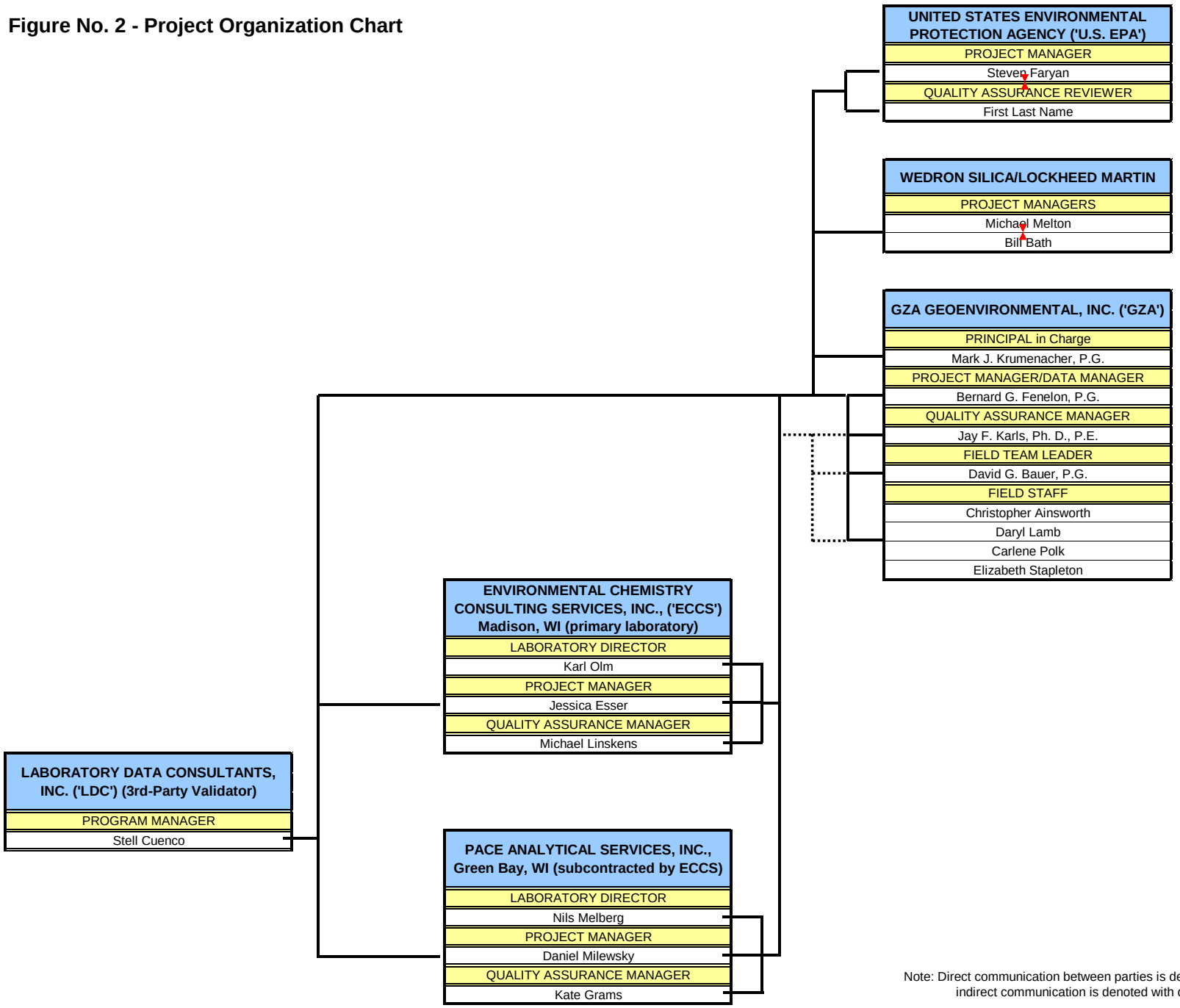
Figure No. 2 - Project Organization Chart



Note: Direct communication between parties is denoted with solid lines; indirect communication is denoted with dashed lines.

APPENDIX A

September 25, 2013
Administrative Order on Consent (AOC)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5

IN THE MATTER OF:	)	ADMINISTRATIVE ORDER ON CONSENT
Wedron Ground Water Contamination Site Wedron, Illinois	)	
Wedron Silica Company; Technisand, Inc.; and Lockheed Martin Corporation	)	EPA Docket No. **RCRA-05-2013-0011**
RESPONDENTS	)	Proceeding under Section 3013(a) of the Resource Conservation and Recovery Act, as amended, 42 U.S.C. § 6934(a)

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I. JURISDICTION

1. This Consent Order is issued pursuant to the authority vested in the Administrator of the Environmental Protection Agency (EPA or Agency) by Section 3013(a) of the Resource Conservation and Recovery Act, (RCRA), as amended, 42 U.S.C. § 6934(a). The authority to enter into this Consent Order has been duly delegated to the Regional Administrator, and further delegated to the Director, Land and Chemicals Division, EPA Region 5.

2. This Consent Order concerns the preparation and performance of monitoring, testing, analysis and reporting relating to property owned and/or operated by Wedron Silica Company and Technisand, Inc. (and formerly owned and/or operated by Martin Marietta Corporation) (the Site) at the Wedron Ground Water Contamination Site located in Wedron, LaSalle County, Illinois (the Wedron Site).

3. This Consent Order is issued to Wedron Silica Company; Technisand, Inc.; and Lockheed Martin Corporation, successor in interest to Martin Marietta Corporation (Respondents). For purposes of the work to be performed as set forth in this Consent Order, Respondents consent to and agree not to contest EPA's authority to issue this Consent Order and to enforce its terms. Further, Respondents will not contest EPA's authority to: compel compliance with this Consent Order in any subsequent enforcement proceedings; require Respondents' full or interim compliance with the terms of this Consent Order; or impose sanctions for violations of this Consent Order; provided, however, that Respondents retain any and all rights they may have to dispute the merits of any such claims.

4. Respondents' participation in this Consent Order shall not constitute or be construed as an admission of liability. Respondents do not admit, and retain the right to controvert, the factual allegations and legal conclusions set forth in this Consent Order (Section IV., Findings of Fact, and Section V., Determinations and Conclusions of Law).

5. This Consent Order is based upon the administrative record compiled by EPA and incorporated herein by reference. The record is available for review by the Respondents and the public at EPA's Regional Office at 77 W. Jackson Blvd. Chicago, IL 60604.

II. PARTIES BOUND

6. The provisions of this Consent Order shall apply to and be binding upon Respondents and their successors and assigns, and to Respondents' officers, directors, employees, agents and contractors acting in their official capacity as representatives of Respondents.

7. Respondents are jointly and severally responsible for carrying out all activities required by this Consent Order. In the event of the insolvency or other failure of any one or more Respondents to implement the requirements of this Order, the remaining Respondents shall complete all such requirements.

8. No change in ownership, corporate, or partnership status relating to property owned or operated by Respondents at the Site will in any way alter the status or responsibility of Respondents under this Consent Order. Any conveyance by Respondents of title, easement, or other interest in property owned or operated by Respondents at the Site, or a portion of such interest, shall not affect Respondents' obligations under this Consent Order. Respondents shall be responsible and liable for any failure to carry out all activities required of Respondents by this Consent Order, irrespective of its use of employees, agents, contractors, or consultants to perform any such tasks.

9. Respondents shall provide a copy of this Consent Order to all contractors, subcontractors, laboratories, and consultants retained to conduct or monitor any portion of the work performed pursuant to this Consent Order within seven (7) calendar days of the effective date of this Consent Order, or on the date of such retention, and Respondents shall condition all such contracts on compliance with the terms of this Consent Order.

10. Any documents transferring ownership and/or operations of property owned or operated by Respondents at the Site to a successor-in-interest shall include written notice of this Consent Order. In addition, Respondents shall, no less than thirty (30) days prior to transfer of ownership or operation of property owned or operated by Respondents at the Site, provide written notice of this Consent Order to such successor-in-interest, and written notice of said transfer of ownership and/or operation to EPA.

III. STATEMENT OF PURPOSE

11. In entering into this Consent Order, the mutual objectives of EPA and Respondents are the protection of human health and the environment through Respondents' implementation of sampling, analysis, monitoring and reporting at property currently or historically owned or operated by Respondents at the Site. In meeting these objectives, Respondents shall prepare and submit for approval to EPA a sampling and analysis workplan (the Workplan) to perform monitoring, testing, analysis, and reporting at the Site in order to ascertain the nature and extent of the presence and/or release of hazardous wastes and/or hazardous constituents at the Site.

IV. FINDINGS OF FACT

12. The Site is in the unincorporated community of Wedron located in LaSalle County, Illinois.

13. Wedron Silica Company (Wedron Silica) currently owns and operates a sand mining facility and related maintenance, storage, and administrative property and previously operated a small laboratory at the Technical Center at the Site.

14. Wedron Silica operations include sand mining pits and ancillary operations where sand is mined, processed, and loaded for shipment by rail and truck.

15. Technisand, Inc. (Technisand) currently owns and operates a resin coating facility at the Site.

16. Technisand operations include a coating facility and loading facilities for shipment by rail and truck.

17. The mining business at the Site operates sand mining pits, which are pumped for water that is used in the mining process.

18. Martin Marietta Corporation (Martin Marietta) owned and operated the sand mining business and related property from 1979 to 1984. Martin Marietta sold the sand mining business to Wedron Silica Company in 1984 and leased the related property to Wedron Silica Company from 1984 to 1991. Martin Marietta sold the related property to Wedron Silica Company in 1991.

19. Lockheed Martin Corporation (Lockheed Martin) is the successor in interest to Martin Marietta.

20. The Illinois Environmental Protection Agency (IEPA) initiated a ground water investigation at the Wedron Site in 1982 after several residents reported gasoline-type odors in waters from their private wells to the Illinois Department of Public Health (IDPH).

21. IEPA's 1982-1985 ground water investigation (the 1982-1985 Investigation) results reported that seven private drinking water wells, and a well at a former Martin Marietta facility, were contaminated with certain volatile organic compounds (VOCs) below the Maximum Contaminant Level (MCL) established under the National Primary Drinking Water Regulations and benzene above the established MCL.

22. IEPA made no determination as to the source of the groundwater and soil contamination during its 1982-1985 Investigation.

23. The 1982-1985 Investigation noted soil vapors were present in the sub-surface soil on the former Martin Marietta scale house property at the Site.

24. In 1985, the Illinois Commerce Commission funded the installation of two new deep drinking water wells at the Site to provide permanent alternate water to residents with contaminated private wells.

25. In 2011, some residents of Wedron, Illinois reported gasoline-type odors in waters from their private drinking water wells to the LaSalle County Health Department.

26. On October 19, 2011, IDPH and IEPA sampled residential wells at the Wedron Site, and the sample results established that two private wells were contaminated with certain VOCs below the MCL established under the National Primary Drinking Water Regulations and benzene above the established MCL.

27. IEPA referred the Wedron Site to EPA and asked for assistance in a letter dated November 10, 2011.

28. EPA collected samples from all of the 45 private drinking water wells at the Wedron Site on a continuous basis from December 2011 through May 2013.

29. EPA's sample results from December 2011 through May 2013 established that eight private drinking water wells, nine homes total, tested positive for certain VOCs below the MCL established under the National Primary Drinking Water Regulations and benzene above the established MCL.

30. The MCL for benzene under the National Primary Drinking Water Regulations is .005 mg/L, which is equivalent to 5 parts per billion or 5 ppb. See 40 C.F.R. § 141.61.

31. On June 11, 2012, EPA issued a request for information to Wedron Silica pursuant to Section 104(e) of the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA), 42 U.S.C. § 9604(e).

32. On June 15, 2012, EPA issued a request for information to Wedron Silica pursuant to Section 9005 of RCRA, 42 U.S.C. § 6991(d).

33. Wedron Silica responded to EPA's June 11, 2012 and June 15, 2012 requests for information on July 24, 2012.

34. Wedron Silica's July 24, 2012 responses to EPA's June 11, 2012 and June 15, 2012 requests for information provided information that Wedron Silica and Martin Marietta managed, handled, stored, treated, disposed of and/or potentially released hazardous wastes and/or hazardous constituents at the Site.

35. On July 26, 2012, EPA issued a request for information to Martin Marietta pursuant to Section 104(e) of CERCLA, 42 U.S.C. § 9604(e), and Section 9005 of RCRA, 42 U.S.C. § 6991d.

36. Lockheed Martin responded to EPA's July 26, 2012 request for information on September 28, 2012, as the successor in interest to Martin Marietta.

37. Lockheed Martin's September 28, 2012 response to EPA's July 26, 2012 request for information provided information that Wedron Silica and Martin Marietta managed, handled, stored, treated, disposed of and/or potentially released hazardous wastes and/or hazardous constituents at the Site.

38. In July and August 2012, EPA and IEPA conducted a site assessment, and took soil and ground water samples that identified soil and ground water contamination in multiple locations at the Wedron Site, many exceeding health based standards.

39. The site assessment reported soil contamination (in levels below applicable heath based standards) at the depth of 15.5 feet below ground surface in one location on property owned by Wedron Silica and operated by Technisand. Benzene was not reported at this location.

40. The ground water contamination plume has the potential to migrate and contaminate additional private drinking water wells at the Wedron Site.

V. DETERMINATIONS AND CONCLUSIONS OF LAW

41. The Wedron Site is a "facility or site" within the meaning of Section 3013(a) of RCRA, 42 U.S.C. § 6934(a).

42. Property currently or historically owned and/or operated by Respondents at the Site is a "facility or site" within the meaning of Section 3013(a) of RCRA, 42 U.S.C. § 6934(a).

43. Each Respondent is a "person" as defined in Section 1004(15) of RCRA, 42 U.S.C. § 6903(15).

44. Each Respondent is an "owner" and/or "operator" or a "previous owner" and/or "previous operator" of a "facility or site" at the Site within the meaning of Section 3013(a) or (b) of RCRA, 42 U.S.C. § 6934(a) or (b).

45. Section 1004(27) of RCRA, 42 U.S.C. § 6903(27) defines the term "solid waste" to mean "any garbage, refuse... and other discarded material, including solid, liquid, semisolid, or contained gaseous material resulting from industrial, commercial, mining, and agricultural operations..."

46. Section 1004(5) of RCRA, 42 U.S.C. § 6903(5), defines the term "hazardous waste" to mean:

    a solid waste, or combination of solid wastes, which because of its quantity, concentration, or physical, chemical, or infectious characteristics may-

    (A) cause or significantly contribute to an increase in mortality or an increase in serious irreversible, or incapacitating reversible, illness; or

    (B) pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, or disposed of, or otherwise managed.

47. Based on the foregoing Findings of Fact, the Administrative Record, and pursuant to Section 3013(a) of RCRA, 42 U.S.C. § 6934(a), EPA has hereby determined the presence of hazardous waste that is or has been managed at the Site, and/or the release of hazardous waste which is or has been stored, treated, or disposed of at the Site.

48. Based on the foregoing Findings of Fact, the Administrative Record, and pursuant to Section 3013(a) of RCRA, 42 U.S.C. § 6934(a), EPA has hereby determined that there may be a substantial hazard to human health or the environment due to the presence and/or release of hazardous wastes at or from property owned and/or operated by Respondents at the Site.

49. Respondents, as current or previous owners and/or operators of property at the Site, agree to conduct the actions ordered herein, which are necessary to ascertain the presence, nature, and extent of potential hazard to human health or the environment.

50. The EPA has further determined that the monitoring, testing, analysis, and reporting set forth in this Consent Order are reasonable to ascertain the presence, nature, and extent of potential hazard to human health or the environment.

VI. ORDER

51. Pursuant to Section 3013(a) of RCRA, 42 U.S.C. § 6934(a), Respondents consent to and are hereby ordered to perform the following actions in the manner and by the dates specified herein:

52. Attached hereto as Exhibit A is an approved Workplan.

53. All samples shall be collected to adequately ascertain the presence and, to the extent present, the horizontal and vertical extent of impacts. All samples shall be analyzed using EPA-approved analytical methods.

54. The Workplan includes a discussion of the following parameters:

a. Rationale and objectives of the Workplan;
b. Field investigation procedures;
c. Field sample collection procedures;
d. Field measurements;
e. Schedule; and
f. Sample analysis and testing.

55. In accordance with the Workplan schedule, or within forty-five (45) days of completion of any additional work required pursuant to Paragraph 60, below, Respondents shall submit a final report to EPA addressing the activities proposed in the Workplan, including a summary of all actions taken to comply with this Order. The report will serve as a Current Conditions Report, summarizing the results of investigations for work completed in accordance with the Workplan.

56. EPA acknowledges that Respondents may have completed some of the tasks required by this Consent Order and/or that Respondents may have available some of the information and data required by this Consent Order. Such previous work may be used to meet the requirements of this Consent Order upon submission to, and formal approval by, EPA.

57. All work undertaken pursuant to this Consent Order shall be developed and performed in accordance with RCRA and its implementing regulations, and all relevant EPA guidance documents, including those found at http://www.epa.gov/epawaste/hazard/correctiveaction/resources/guidance/index.htm. All attachments to this Consent Order are incorporated herein by reference.

58. Health and Safety Plan. Respondent shall develop a Health and Safety Plan and it shall be implemented during the Work performed under this Consent Order. The Health and Safety plan shall comply with applicable Occupational Safety and Health Administration (OSHA) regulations.

59. The Workplan is incorporated into this Consent Order; any other reports, plans, specifications, schedules, other submissions and attachments required by this Consent Order are, upon written approval by EPA, incorporated into this Consent Order. Any noncompliance with such EPA-approved reports, plans, specifications, schedules, other submissions, and attachments shall be considered a violation of this Consent Order and shall subject Respondents to the stipulated penalty provisions included in Section XVII., Delay in Performance/Stipulated Penalties.

VII. ADDITIONAL WORK

60. EPA may determine that additional monitoring, testing, analysis, and/or reporting is necessary to ascertain the nature and extent of any hazard to human health or the environment which may be presented by the presence or release of hazardous wastes and/or hazardous constituents at the Site. If EPA determines that such additional work is necessary, EPA will notify Respondents in writing and specify the basis for its determination that additional work is necessary. Within fifteen (15) days after the receipt of such determination, Respondents shall have the opportunity to meet or confer with EPA to discuss the additional work. If required by EPA, Respondents shall submit for EPA approval a workplan for the additional work. EPA will specify the contents of such workplan. Such workplan shall be submitted by Respondents within thirty (30) days of receipt of EPA's determination that additional work is necessary, or according to an alternative schedule established by EPA.

VIII. MINIMUM QUALIFICATIONS FOR PERSONNEL

61. All work performed by Respondents pursuant to this Consent Order shall be under the direction and supervision of an individual who has demonstrated expertise in hazardous waste and hazardous constituents site investigation. Before any work is performed, Respondents shall submit to EPA, in writing, the name, title, and qualifications of the supervisory personnel and of any contractors or subcontractors to be used in carrying out the terms of this Consent Order. Additionally, Respondents shall ensure that when a license is required, only licensed individuals shall be used to perform any work required by this Consent Order.

IX. SUBMISSIONS/EPA REVIEW

62. EPA will review Respondents' workplans, reports, and any other documents submitted pursuant to this Consent Order ("submissions"), with the exception of progress reports, and will notify Respondents in writing of EPA's approval or disapproval of each such submission. In the event of EPA's disapproval, EPA shall specify in writing any deficiencies in the submission. Such disapproval shall not be subject to the dispute resolution procedures of Section XVII., below.

63. Within thirty (30) calendar days of receipt of EPA's comments on the submission, Respondents shall submit to EPA for approval a revised submission which responds to any comments received and/or corrects any deficiencies identified by EPA. Respondents may request additional time within which to submit a revised submission. In the event that EPA disapproves the revised submission, Respondents may invoke the dispute resolution procedures of Section XVII., below. Otherwise, EPA reserves the right to revise such submission and seek to recover from Respondents the costs thereof, in accordance with any rights that it may have under RCRA, CERCLA and any other applicable law. Any submission approved or revised by EPA or upheld through dispute resolution under this Consent Order shall be deemed incorporated into and made an enforceable part of this Consent Order.

64. Upon disapproval by EPA of a revised submission, and in the event Respondents do not invoke the dispute resolution procedures of Section XVII., below, Respondents may submit to EPA for approval a subsequent revised submission which responds to any comments received and/or corrects any deficiencies identified by EPA. Respondents may request additional time within which to submit a subsequent revised submission.

65. In the event EPA and Respondents cannot resolve issues relating to EPA's comments and EPA disapproves of any subsequent revised submission, Respondents may invoke the dispute resolution procedures of Section XVII., below. Otherwise, EPA reserves the right to revise such submission and to seek to recover from Respondents the costs of revising the subsequent submission in accordance with RCRA, CERCLA and any other applicable law. Any submission approved or revised by EPA or upheld through dispute resolution under this Consent Order shall be deemed incorporated into and made an enforceable part of this Consent Order.

66. Beginning with the first day of the second full month following the effective date of this Consent Order, and every two months thereafter on the first day of the month, for the first six months that this Consent Order is effective, Respondents shall provide EPA with bi-monthly progress reports which contain the information required in the relevant Scope(s) of Work attached hereto. Thereafter, throughout the period this Consent Order is effective, Respondents shall provide EPA with such reports on a quarterly basis.

67. Two (2) copies (one (1) hard copy, double-sided if possible, and one (1) electronic copy) of all submissions (including revised submissions) required to be submitted by this Consent Order shall be delivered to the EPA Project Coordinator designated pursuant to Section XI., Project Coordinator, below.

68. EPA shall endeavor to timely approve or disapprove any deliverable submitted by Respondents for approval pursuant to this Consent Order. Nothing in this paragraph shall be construed to confer any enforceable rights upon Respondents, nor shall any failure to comply with the provisions of this paragraph be subject to the dispute resolution provisions set forth in Section XVII., below.

69. Unless otherwise specified, reports, correspondence, approvals, disapprovals, notices, or other submissions relating to or required under this Consent Order shall be in writing and shall be sent as follows:

a. All documents to be submitted to EPA shall be sent to:

Steve Faryan, On-Scene Coordinator U.S.
Environmental Protection Agency Region 5 (SC-5J)
77 W. Jackson Blvd.
Chicago, IL 60604

b. Documents to be submitted to Respondents shall be sent to:

Mike Melton
Corporate Environmental Manager
Fairmount Minerals, Ltd.
c/o Wedron Silica Company
3450 E. 2056th Rd.
P.O. Box 119
Wedron, IL 60557

with a copy to:

David J. Crandall
VP, General Counsel & Secretary
Fairmount Minerals, Ltd.
8834 Mayfield Road
Chesterland, Ohio 44026

and:

William W. Bath
Project Manager
Lockheed Martin Corporation
2550 North Hollywood Way, Suite 406
Burbank, CA 91505-5047

with a copy to:

Norman A. Varney, Jr.
Associate General Counsel
Environment, Safety & Health
Lockheed Martin Corporation
P.O. Box 61511
King of Prussia, PA 19406-0911

70. Any notice, report, certification, data presentation, or other document submitted by Respondents pursuant to this Consent Order which discusses, describes, demonstrates, supports any finding or makes any representation concerning Respondents' compliance or noncompliance with any requirement of this Consent Order shall be certified by a duly authorized representative of Respondents. A person is a "duly authorized representative" only if: (a) the authorization is made in writing; (b) the authorization specifies either an individual or position having responsibility for overall operation of the regulated facility or activity (a duly authorized representative may thus be either a named individual or any individual occupying a named position); and (c) the written authorization is submitted to the Project Coordinator designated by EPA pursuant to Section XI., Project Coordinator, of this Consent Order.

71. The certification required by Paragraph 70 above, shall be in the following form:

"I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to be the best of my knowledge and belief, true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations."

Signature:_____

Name: _____

Title: _____

X. QUALITY ASSURANCE/QUALITY CONTROL

72. Respondents shall follow EPA guidance for sampling and analysis. Within 20 days of the effective date of this Consent Order, Respondents shall submit to EPA for approval a Quality Assurance Project Plan (QAPP) for all sampling and analysis conducted under this Consent Order. The QAPP shall contain quality assurance/quality control (QA/QC) and chain of custody procedures for all sampling, monitoring, and analytical activities. Any deviations from the QA/QC and chain of custody procedures in the approved QAPP must be approved by EPA prior to implementation; must be documented, including reasons for the deviations; and must be reported in the applicable report.

73. The name, address, telephone number and contact person of each analytical laboratory Respondents propose to use must be specified in the applicable QAPP.

74. Within 20 days of the effective date of this Consent Order, Respondents shall submit to EPA for approval Data Quality Objectives for each data collection activity to ensure that data of known and appropriate quality are obtained and that data are sufficient to support their intended use(s).

75. Respondents shall monitor to ensure that high quality data is obtained by their consultant or contract laboratories. Respondents shall ensure that laboratories used by Respondents for analysis perform such analysis according to the latest approved edition of "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" (SW-846 Third Edition as amended by Update One, July 1992), or other methods deemed satisfactory to EPA. If methods other than EPA methods are to be used, Respondents shall specify and submit all such protocols for EPA approval in the Data Quality Objectives deliverable. EPA may reject any data that do not meet the requirements of the approved Workplan or EPA analytical methods and may require resampling and additional analysis.

76. Respondents shall ensure that laboratories they use for analyses participate in a QA/QC program equivalent to that which is followed by EPA. EPA may conduct a performance and QA/QC audit of each laboratory chosen by Respondents before, during, or after sample analyses. Upon request by EPA, Respondents shall have their laboratory perform analyses of samples provided by EPA to demonstrate laboratory performance. If the audit reveals deficiencies in a laboratory's performance or QA/QC, resampling and additional analysis may be required.

XI. PROJECT COORDINATOR

77. EPA hereby designates as its Project Coordinator:

Steve Faryan, On-Scene Coordinator U.S.
Environmental Protection Agency Region 5 (SC-5J)
77 W. Jackson Blvd.
Chicago, IL 60604
(312) 353-9351

78. Within fifteen (15) calendar days of Respondents' receipt of this Consent Order, Respondents shall designate a Project Coordinator and submit the designated Project Coordinator's name, address, and telephone number in writing to EPA.

79. Each Project Coordinator shall, on behalf of the party that designated him/her, oversee the implementation of this Consent Order and function as the principal project contact.

80. Respondents shall provide EPA with a written notice of any change in their Project Coordinator. Such notice shall be provided at least seven (7) calendar days prior to the change in Project Coordinator.

XII. SAMPLING AND DATA/DOCUMENT AVAILABILITY

81. Respondents shall submit to EPA the results of all sampling and/or tests or other data generated by, or on behalf of, Respondents pursuant to the requirements of this Consent Order and the Attachments appended hereto and incorporated herein.

82. Respondents shall notify EPA, in writing, at least fourteen (14) calendar days in advance of engaging in any field activities at the Site conducted pursuant to this Consent Order. At the request of EPA, Respondents shall provide or allow EPA or its authorized representatives to take split and/or duplicate samples of all samples collected by Respondents pursuant to this Consent Order. Similarly, at the request of Respondents, EPA will allow Respondents or their authorized representatives to take split and/or duplicate samples of any samples collected by EPA under this Consent Order, provided that such sampling shall not delay EPA's proposed sampling activities. Upon request, Respondents shall submit to EPA the results of all sampling and/or tests or other data generated by, or on behalf of, the Respondents pursuant to this Order. Nothing in this Consent Order shall limit or otherwise affect EPA's authority to collect samples pursuant to applicable law, including, but not limited to, RCRA and CERCLA.

XIII. ON-SITE AND OFF-SITE ACCESS

83. Respondents Wedron Silica and Technisand shall provide access at all reasonable times to the Site and to all records and documentation relating to conditions at the Site and the activities conducted pursuant to this Consent Order to EPA and its employees, contractors, agents, consultants, and representatives. These individuals shall be permitted to move freely (subject to applicable Mine Safety Health Act (MSHA) and OSHA requirements) at the Site in order to conduct activities which EPA determines necessary pursuant to this Consent Order.

84. To the extent that activities required by this Consent Order or by any approved workplans prepared pursuant hereto, must be done on property not owned or controlled by Respondents, Respondents will use their best efforts to obtain access agreements in a timely manner from the present owners of such property. Best efforts, as used in this paragraph, shall include the payment of reasonable compensation in consideration for granting access. Respondents shall ensure that EPA's Project Coordinator has a copy of any access agreements.

85. Nothing in this Consent Order limits or otherwise affects EPA's right of access and entry pursuant to applicable law, including, but not limited to, RCRA and CERCLA.

XIV. RECORD PRESERVATION

86. Respondents shall retain, during the pendency of this Consent Order and for a minimum of five (5) years after its termination, a copy of all data, records, and documents now in their possession or control, or in the possession or control of their contractors, subcontractors, representatives, or which come into the possession or control of the Respondents, their contractors, subcontractors, or representatives, which relate in any way to this Consent Order. Respondents shall notify EPA, in writing, at least ninety (90) days in advance of the destruction of any such records, and shall provide EPA with the opportunity to take possession of any such records. Such written notification shall reference the caption, docket number and date of issuance of this Consent Order and shall be addressed to EPA's Project Coordinator, as follows:

Steve Faryan, On-Scene Coordinator U.S.
Environmental Protection Agency Region 5 (SC-5J)
77 W. Jackson Blvd.
Chicago, IL 60604

Additionally, Respondents shall provide data, records and documents retained under this Section at any time before the expiration of the five (5) year period at the written request of EPA.

XV. INFORMATION SUBMITTED TO EPA

87. Respondents may assert a business confidentiality claim in the manner described in 40 C.F.R. § 2.203(b) covering all or part of any information submitted to EPA pursuant to this Consent Order. In accordance with 40 C.F.R. § 2.204(e)(4), any assertion of confidentiality shall be adequately substantiated by Respondents when the assertion is made. Information submitted for which Respondents have asserted a claim of confidentiality as specified above shall be disclosed by EPA only to the extent and manner permitted by 40 C.F.R. Part 2, Subpart B. If no such confidentiality claim accompanies the information when it is submitted to EPA, the information may be made available to the public by EPA without further notice to the Respondents. Respondents agree not to assert any confidentiality claim with respect to any physical, sampling, monitoring, or analytical data.

88. In the event that Respondents wish to assert a privilege with regard to any document which EPA seeks to inspect or copy pursuant to this Consent Order, Respondents shall identify the document, the privilege claimed and the basis therefore in writing. For the purposes of this Consent Order, privileged documents are those documents exempt from discovery from the United States in litigation under the Federal Rules of Civil Procedure and/or any applicable case law. EPA may dispute any such claim of privilege pursuant to the dispute resolution provisions set forth in Section XVII., below.

XVI. DELAY IN PERFORMANCE/STIPULATED PENALTIES

89. Unless there has been a written modification of a compliance date by EPA, or excusable delay as defined below in Section XVIII., Force Majeure, in the event that Respondents fail to comply with any requirement set forth in this Consent Order, Respondents shall pay stipulated penalties, as set forth below, upon receipt of written demand by EPA.

90. Compliance by Respondents shall include commencement or completion, as deemed appropriate by EPA, of any activity, plan, study or report required by this Consent Order, and in the manner required by this Consent Order and within the specified time schedules in and approved under this Consent Order. Stipulated penalties shall accrue as follows:

91. For any failure to commence, perform or complete work as prescribed in this Consent Order: $500 per day for one to fifteen days or part thereof of noncompliance, $1,000 per

day for sixteen to thirty days or part thereof of noncompliance, and $1,500 per day for each day of noncompliance, or part thereof, thereafter;

92. For any failure to submit any draft or final workplans, plans, or reports as required by this Consent Order: $250 per day for one to fifteen days or part thereof of noncompliance, $500 per day for sixteen to thirty days or part thereof of noncompliance, and $1,000 per day for each day of noncompliance, or part thereof, thereafter; and

93. For any failure to submit other deliverables as required by this Consent Order: $250 per day for one to fifteen days or part thereof of noncompliance, $500 per day for sixteen to thirty days or part thereof of noncompliance, and $1,000 per day for each day of noncompliance, or part thereof, thereafter.

94. All stipulated penalties shall begin to accrue the first day that a violation occurs, or the first day after the date that complete performance is due, and shall continue to accrue through the final day of or correction of the violation. Nothing herein shall prevent the simultaneous accrual of separate stipulated penalties for separate violations of this Consent Order.

95. All stipulated penalties owed to EPA under this section shall be due within thirty (30) calendar days of receipt of a demand for payment, unless Respondents invoke the dispute resolution procedures under Section XVII., below. Such demand for payment shall describe the noncompliance and shall indicate the amount of stipulated penalties due.

96. All stipulated penalty payments may be made by certified or cashier's check payable to the Treasurer of the United States of America and shall be remitted to:

    U.S. Department of the Treasury
    Attention: EPA Region 5,
    Office of the Comptroller
    P.O. Box 70753
    Chicago, Illinois 60673

97. All payments shall reference the Respondents' name and address, and the EPA Docket Number of this Consent Order. Copies of the transmittal of payment shall be sent simultaneously to the EPA Project Coordinator and the Regional Hearing Clerk, U.S. Environmental Protection Agency, Region 5 (E-19J), 77 West Jackson Boulevard, Chicago, Illinois 60604.

98. Respondents may dispute EPA's demand for payment of stipulated penalties for any alleged violation of this Consent Order by invoking the dispute resolution procedures below under Section XVII., Dispute Resolution. Stipulated penalties shall continue to accrue, but are not required to be paid, for any alleged noncompliance which is the subject of dispute resolution during the period of such dispute resolution. To the extent that Respondents do not prevail upon resolution of the dispute, Respondents shall remit to EPA within twenty-one (21) calendar days of receipt of EPA's written decision as to said

dispute, any outstanding penalty payment in the manner described above in Paragraph 96 of this Section.

99. Neither the filing of a petition to resolve a dispute nor the payment of stipulated penalties shall alter in any way Respondents' obligation to comply with the requirements of this Consent Order.

100. The assessment of stipulated penalties set forth in this section shall not preclude EPA from pursuing any other remedies or sanctions which may be available to EPA by reason of Respondents' failure to comply with any of the requirements of this Consent Order.

XVII. DISPUTE RESOLUTION

101. If a dispute arises under this Consent Order, the procedures of this section shall apply. The Parties shall make reasonable efforts to informally resolve disputes at the Project Coordinator or immediate supervisor level.

102. If Respondents disagree, in whole or in part, with any EPA disapproval, modification or other decision or directive made by EPA pursuant to this Consent Order, Respondents shall notify EPA in writing of their objections, and the basis therefore, within fourteen (14) calendar days of receipt of EPA's disapproval, decision or directive. Such notice shall set forth the specific points of the dispute, the position which Respondents assert should be adopted as consistent with the requirements of this Consent Order, the basis for Respondents' position, and any matters which they consider necessary for EPA's determination. EPA and Respondents shall have an additional fourteen (14) calendar days from the receipt by EPA of the notification of objection, during which time representatives of EPA and Respondents may confer in person or by telephone to resolve any disagreement. If an agreement is reached, the resolution shall be written and signed by an authorized representative of each party. In the event that resolution is not reached within the twenty-eight (28) calendar day period, EPA will furnish to Respondents, in writing, its decision on the pending dispute. Said written decision shall state the basis and rationale for the decision.

103. Except as provided in Paragraphs 101 and 102 above, the existence of a dispute, as defined in this section, and EPA's consideration of matters placed into dispute, shall not excuse, toll or suspend any other compliance obligation or deadline required pursuant to this Consent Order during the pendency of the dispute resolution process.

104. Notwithstanding any other provisions of this Consent Order, no action or decision by EPA pursuant to this Consent Order shall constitute final agency action giving rise to any right to judicial review.

XVIII. FORCE MAJEURE

105. Respondents shall perform the requirements of this Consent Order in the manner and within the time limits set forth herein, unless the performance is prevented or delayed by

events which constitute a *force majeure.* Respondents shall have the burden of proving such a *force majeure.* A force *majeure* is defined as any event arising from causes not reasonably foreseeable and beyond the control of Respondents, which cannot be overcome by due diligence and which delays or prevents performance in the manner or by a date required by this Consent Order. A *force majeure* does not include: increased costs of performance; changed economic circumstances; failure to obtain federal, State or local permits; reasonably foreseeable weather conditions; or weather conditions which could have been overcome by due diligence.

106. Respondents shall notify EPA, in writing, within ten (10) calendar days after they become or should have become aware of any event which Respondents claim constitutes a *force majeure.* Such notice shall estimate the anticipated length of delay, including necessary demobilization and remobilization, its cause, measures taken or to be taken to prevent or minimize the delay, and an estimated time table for implementation of these measures. Failure to comply with the notice provision of this paragraph shall constitute a waiver of Respondents' right to assert a *force majeure* claim with respect to such event. If, in EPA's sole and unreviewable discretion, EPA determines that the failure to give notice was not prejudicial to EPA's efforts to protect human health or the environment, Respondents' failure to give notice shall not constitute a waiver. In addition to the above notification requirements, Respondents shall undertake all reasonable actions to prevent or to minimize any delay in achieving compliance with any requirement of this Consent Order after it becomes or should have become aware of any event which may delay such compliance.

107. If EPA determines that the failure to comply or delay has been or will be caused by a *force majeure,* the time for performance of that requirement of this Consent Order may be extended, upon EPA approval, for a period equal to the delay resulting from such *force majeure.* This shall be accomplished through an amendment to this Consent Order pursuant to Section XXII., Subsequent Modification of Order. Such an extension shall not alter the schedule for performance or completion of any other tasks required by this Consent Order, unless these tasks are unavoidably affected by the delay. In the event that EPA and Respondents cannot agree that any delay or failure has been or will be caused by a *force majeure,* or if there is no agreement on the length of the extension, Respondents may invoke the dispute resolution procedures set forth in Section XVII., Dispute Resolution.

XIX. EPA'S RESERVATION OF RIGHTS

108. EPA expressly reserves all rights and defenses that it may have, including the right to disapprove of work performed by Respondents pursuant to this Consent Order, to require that Respondents correct and/or re-perform any work disapproved by EPA, and to request that Respondents perform tasks in addition to those stated in the Workplan, or in this Consent Order, consistent with the objectives of this Consent Order.

109. EPA hereby reserves all of its statutory and regulatory powers, authorities, rights and remedies, both legal and equitable, including any which may pertain to Respondents' failure to comply with any of the requirements of this Consent Order. This Consent Order

shall not be construed as a covenant not to sue, or as a release, waiver or limitation of any rights, remedies, defenses, powers and/or authorities, civil or criminal, which EPA has under RCRA, CERCLA, the Clean Water Act (CWA), the Safe Drinking Water Act (SDWA), the Clean Air Act (CAA), or any other statutory, regulatory, or common law enforcement authority of the United States.

110. EPA reserves the right to perform any portion of the work required herein or any additional monitoring, sampling, analysis, or reporting it deems necessary to protect public health or welfare or the environment. EPA reserves the right to seek reimbursement from Respondents for costs incurred by the EPA in connection with any such actions, pursuant to any right it may have under applicable law.

111. EPA reserves whatever rights it may have under any environmental law or authority, or in equity, to seek to recover from Respondents any costs incurred by EPA in overseeing the implementation of this Consent Order.

XX. OTHER APPLICABLE LAWS

112. All actions required to be taken by Respondents pursuant to this Consent Order shall be undertaken in accordance with the requirements of all applicable federal, state, and local laws, regulations, permits, and ordinances.

113. Compliance by Respondents with the terms of this Consent Order shall not relieve Respondents of their obligations to comply with RCRA, or any other applicable federal, state, or local laws, regulations, permits, and ordinances.

114. This Consent Order is not and shall not be interpreted to be a permit, or as a ruling or a determination of any issue related to a permit under federal, state or local law. This Consent Order shall not in any way affect Respondents' obligation, if any, to secure such a permit, nor shall this Consent Order be interpreted in any way to affect or waive any of the conditions or requirements that may be imposed by such permit, nor of Respondents' right to appeal any conditions of such permit. Respondents shall obtain or cause their representatives to obtain all permits and approvals necessary under such laws and regulations.

XXI. OTHER CLAIMS

115. Nothing in this Consent Order shall constitute or be construed as a release from any claim, cause of action, demand, or defense in law or equity, against any person, firm, partnership, or corporation for any liability it may have arising out of or relating in any way to the generation, storage, treatment, handling, transportation, release, or disposal of any solid wastes, hazardous wastes, hazardous constituents, hazardous substances, pollutants, or contaminants found at, taken to, or migrating from the Site.

116. Neither the United States nor EPA shall be deemed a party to any contract involving Respondents and relating to activities at the Site and shall not be liable for any claim or

cause of action arising from or on account of any act, or omission of Respondents, their officers, employees, contractors, receivers, trustees, agents or assigns, in carrying out the activities required by this Consent Order.

XXII. SUBSEQUENT MODIFICATION OF ORDER

117. Except as provided in Paragraph 118 of this section, the provisions of this Consent Order may be amended only by mutual agreement of EPA and Respondents. Any such amendment shall be in writing, shall be signed by an authorized representative of each party, shall have as its effective date the date on which it is signed by EPA, and shall be incorporated into this Consent Order. Any oral agreement between EPA and Respondents, the purpose of which is to modify this Consent Order to address exigent circumstances, and which is subsequently ratified in writing by EPA and Respondents, shall have as its effective date the date of such oral agreement.

118. Minor modifications in the studies, techniques, procedures, designs or schedules utilized in carrying out this Consent Order and necessary for the completion of the project may be made by written agreement of the Project Coordinators. Such modifications shall have as an effective date the date on which the agreement is signed by the EPA Project Coordinator.

119. No informal advice, guidance, suggestions, or comments by EPA regarding reports, plans, specifications, schedules, and any other writing submitted by Respondents shall be construed as relieving Respondents of their obligation to obtain written approval, if and when required by this Consent Order.

XXIII. SEVERABILITY

120. If any provision or authority of this Consent Order, or the application of this Consent Order to any party or circumstances, is held by any judicial or administrative authority to be invalid, the application of such provisions to other Parties or circumstances and the remainder of the Consent Order shall not be affected thereby and shall remain in full force.

XXIV. TERMINATION AND SATISFACTION

121. The provisions of this Consent Order shall be deemed satisfied upon Respondents' receipt of written notice from EPA that Respondents have demonstrated to the satisfaction of EPA that the terms of the Consent Order, including any additional tasks determined by EPA to be required pursuant to this Consent Order, have been satisfactorily completed. Such notice shall not be unreasonably withheld. This notice shall not, however, terminate Respondents' obligations to comply with any continuing obligations hereunder, including, but not limited to, Section XIV., Record Preservation; Section XIX., Reservation of Rights; Section XX., Other Applicable Laws; and Section XXI., Other Claims.

XXV. ATTORNEYS' FEES AND COSTS

122. Except as otherwise provided herein, Respondents shall bear their own costs and attorneys' fees.

XXVI. EFFECTIVE DATE

123. The effective date of this Consent Order shall be the date on which Respondents receive a true and correct copy of the fully executed Consent Order.

IT IS SO AGREED AND

ORDERED: FOR COMPLAINANT

DATE: September 24, 2013 BY: _____

Margaret M. Guerriero,
Director Land and Chemicals
Division
U. S. Environmental Protection Agency
Region 5

IN THE MATTER OF:
Wedron Ground Water Contamination Site,
Wedron, Illinois

FOR RESPONDENT Lockheed Martin Corporation

DATE: 9/17/13 BY: Carol B. Cala

Carol B. Cala
Vice President – Energy, Environment,
Safety & Health
Lockheed Martin Corporation
6801 Rockledge Drive, MP CCT-246
Bethesda, MD 20817

IN THE MATTER OF:
Wedron Ground Water Contamination Site,
Wedron, Illinois

FOR RESPONDENT Wedron Silica Company

DATE: September 17, 2013

BY: _____
David J. Crandall
VP, General Counsel & Secretary
Fairmount Minerals, Ltd.
8834 Mayfield Road
Chesterland, OH 44026

IN THE MATTER OF:
Wedron Ground Water Contamination Site,
Wedron, Illinois

FOR RESPONDENT Technisand, Inc.

DATE: September 17, 2013 BY: David J. Crandall

David J. Crandall
VP, General Counsel & Secretary
Fairmount Minerals, Ltd.
8834 Mayfield Road
Chesterland, OH 44026

RCRA-05-2013-0011

EXHIBIT A

WORKPLAN

GZA
GeoEnvironmental, Inc.

Engineers and Scientists

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WORKPLAN

September 12, 2013

The following Workplan is submitted by GZA GeoEnvironmental, Inc. (GZA) for conducting sampling, analysis, monitoring, and reporting at property currently or historically owned and/or operated by Wedron Silica Company (Wedron Silica), Technisand, Inc. (Technisand), and Martin Marietta Corporation (Martin Marietta). The Workplan is prepared in response to requests made by the United States Environmental Protection Agency (USEPA) to Wedron Silica, Technisand, and Lockheed Martin Corporation (Lockheed Martin), and in accordance with the terms of an Administrative Order on Consent (AOC) to which this Workplan will be attached.

The purpose of the Workplan is to evaluate the nature and extent of the presence and/or release of hazardous wastes and/or hazardous constituents at certain locations on Wedron Silica property using a phased approach, comparing soil analytical results to Illinois Tiered Approach to Corrective Action Objectives (TACO)[1] Tier 1 Class I migration to groundwater soil remediation objectives (SROs) to evaluate whether potential source areas exist which require additional characterization and/or investigation.

The general areas included for investigation are:

1) the Tech Center;

2) the area around Illinois Environmental Protection Agency (IEPA) boring GP-10 drilled in 2012 and IEPA monitoring well G103 installed in 1984 near the former Scale-House well;

3) the two former 4,000-gallon gasoline underground storage tanks (USTs) and dispensers closed in 1998 near the main office, which are being investigated under the oversight of the Illinois Office of State Fire Marshal (OSFM), pursuant to a separate Workplan;

4) the former 6,000-gallon gasoline UST near the current Screen House that was closed in 1982;

5) the Pit 2 reclamation area; and

6) evaluation of groundwater and surface water elevations.

The work scopes proposed for each of the six areas were developed through discussions among representatives of the USEPA, Wedron Silica and Lockheed Martin, and will be performed pursuant to the terms of the AOC.

[1] Title 35, Subtitle G, Chapter I, Subchapter F, Part 742 of the Illinois Administrative Code.

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After conclusion of implementation of the work set forth in this Workplan, a final report will be prepared for submission to USEPA, as required under the AOC.

Tech Center

The Tech Center and on-site wastewater treatment system layout are shown on View 1 on Figure 1 and an expanded view is shown on Figure 2. The following work will be performed:

- Two Geoprobe® soil borings will be drilled to the depth of refusal (bedrock) or groundwater, whichever is encountered first (Total Depth), adjacent to the on-site wastewater treatment system at the approximate locations shown on Figures 1 and 2. One boring will be drilled just east of the holding tank and the other boring will be drilled just west of the connection to the storm sewer line located on the west side of the Jackson Street right-of-way.

- Soil samples will be field-screened for organic compounds with a photoionization detector (PID) capable of measuring in the part per billion (ppb) range, and two soil samples from each boring will be submitted for laboratory analyses for volatile organic compounds (VOCs) in accordance with USEPA Method 8260B. Soil samples will be selected for laboratory analyses based on field indications from field screening, odors, staining, etc. If there are no such indicators in soil from the holding tank boring, one soil sample will be submitted for analysis from the 6- to 8-foot interval (anticipated to be from near the base of the holding tank) and one soil sample will be submitted for analysis from Total Depth. If there are no such indicators in soil from the boring drilled near the connection to the storm sewer, one soil sample will be submitted for analysis from the 4- to 6-foot interval (anticipated to be from near the base of the storm sewer[2]) and one soil sample will be submitted for analysis from Total Depth.

- The work conducted, the field screening and analytical results obtained, a comparison of detected constituent concentrations to TACO Tier 1 Class I migration to groundwater SROs and recommendations for follow-up investigation activities, as warranted, will be documented in a report. The report will be submitted to the USEPA. If soil VOC concentrations are all less than TACO Tier 1 Class I migration to groundwater SROs, additional investigation will be unnecessary. If TACO Tier 1 Class I migration to groundwater soil SROs are exceeded, additional activities such as development of Tier 2 TACO levels and/or additional soil investigation and possible investigation of groundwater will be considered. If groundwater investigation is proposed, consideration of the community-wide presence of petroleum constituents in groundwater will be taken into consideration in the interpretation

[2] Wedron Silica and USEPA have contacted LaSalle County for information on the specifications for the storm sewer. Information obtained from La Salle County will be used to adjust the target intervals of the soil samples, as warranted.

of results. Specific information to be considered will include: 1) soil data collected at other potential source areas; 2) soil data collected as part of this investigation; 3) groundwater flow directions determined from piezometers and surface water gaging collected as part of this investigation; and 4) other available data. If additional investigation of soil and/or groundwater is warranted, a Workplan will be prepared and submitted to the USEPA in advance of conducting additional investigation.

Boring GP-10 and Monitoring Well G103 Area

The area of IEPA 2012 boring GP-10 and 1984 monitoring well G103 near the former Scale-House well is shown on View 2 on Figure 1 and an expanded view is shown on Figure 3. During a 1982 to 1984 groundwater investigation by IEPA, petroleum constituents were reported in the Scale-House well. IEPA noted odors in soil samples collected between the depths of 14.7 feet and approximately 15.8 feet where clay was encountered in the boring (B5) drilled for monitoring well G103. Petroleum constituents were reported at concentrations below TACO Tier 1 SROs in 2012 in soil collected from a depth of 15.5 feet (estimated to be from near or below the water table) in boring GP-10. Refusal was encountered at a depth of 18 feet in boring GP-10. The following work will be performed:

- Three Geoprobe® soil borings will be drilled to Total Depth on approximately 25-foot centers spaced between IEPA 2012 boring GP-10 and 1984 monitoring well G103 at the approximate locations shown on Figures 1 and 3. Note that fixed process equipment exists in the area of the proposed borings which limits access to the area for a Geoprobe® drilling rig.

- Soil samples will be field-screened for organic compounds with a PID capable of measuring in the ppb range, and two soil samples from each boring will be submitted for laboratory analyses for VOCs in accordance with USEPA Method 8260B. Soil samples will be selected for laboratory analyses based on field indications from field screening, odors, staining, etc. If there are no such indicators in soil, one soil sample will be submitted for analysis from the 6- to 8-foot interval (anticipated to be approximately half the distance to the water table) and one soil sample will be submitted for analysis from the depth interval immediately above the water table (approximately 15 to 17 feet) or refusal.

- The work conducted, the field screening and analytical results obtained, a comparison of detected contaminant concentrations to Illinois TACO Tier 1 Class I migration to groundwater SROs and recommendations for follow-up investigation activities, as warranted, will be documented in a report. The report will be submitted to the USEPA. If soil VOC concentrations are all less than TACO Tier 1 Class I migration to groundwater SROs, additional investigation will be unnecessary. If TACO Tier 1 Class I migration to groundwater soil SROs are exceeded, additional activities such as development of Tier 2 TACO levels and/or additional soil investigation and possible

investigation of groundwater will be considered. If groundwater investigation is proposed, consideration of the community-wide presence of petroleum constituents in groundwater will be taken into consideration in the interpretation of results. Specific information to be considered will include: 1) soil data collected at other potential source areas; 2) soil data collected as part of this investigation; 3) groundwater flow directions determined from piezometers and surface water gaging collected as part of this investigation; and 4) other available data. If additional investigation of soil and/or groundwater is warranted, a Workplan will be prepared and submitted to the USEPA in advance of conducting additional investigation.

Former 4,000-Gallon Gasoline UST System

The former 4,000-gallon gasoline UST system is shown on View 3 on Figure 1 and an expanded view is shown on Figure 4. Site assessment soil samples were collected from the base and sidewalls of the UST tank basin at the time the USTs were pulled in 1998, but soil samples were not taken beneath the short section of piping runs between the USTs and dispensers or beneath the dispensers. Based on information in the UST closure report and historical aerial photography, the locations of the two USTs and two dispensers were identified to within a couple of feet. Investigation of this area will be performed under the oversight of the OSFM, pursuant to a separate Workplan, which will generally cover the following work:

- Four Geoprobe® soil borings will be drilled to depths of approximately 7 feet (2 feet below the top of the former USTs) at the approximate locations shown on Figures 1 and 4. The proposed borings target the location of the former dispensers and the approximate locations of the former piping runs between the former USTs and dispensers.

- The current gasoline aboveground storage tank (AST) will be temporarily moved to allow the target locations to be drilled.

- Soil samples will be field-screened for petroleum with a PID capable of measuring in the ppb range, and one soil sample from each boring will be submitted for laboratory analyses for VOCs in accordance with USEPA Method 8260B, lead in accordance with USEPA Method 6010B, and soil pH in accordance with USEPA Method 9045D. Soil samples will be selected for laboratory analyses based on field indications from field screening, odors, staining, etc. If there are no such indicators, a soil sample from the 5- to 7-foot interval will be submitted for the analyses.

- The work conducted, the field screening and analytical results obtained, a comparison of detected constituent concentrations to Illinois TACO Tier 1 Class I migration to groundwater SROs and recommendations for follow-up investigation activities, as warranted, will be documented in a report. The report will be submitted to the OSFM and the USEPA. If soil VOC

concentrations are all less than TACO Tier 1 Class I migration to groundwater SROs, additional investigation will be unnecessary. If TACO Tier 1 Class I migration to groundwater SROs are exceeded, additional activities such as development of Tier 2 TACO levels and/or additional soil investigation and possible investigation of groundwater will be considered. If additional investigation of soil and/or groundwater is warranted, a Workplan will be prepared and submitted to the OSFM in advance of conducting additional investigation.

Former 6,000-Gallon Gasoline UST Near Screen House

The former 6,000-gallon gasoline UST near the current Screen House is shown on View 4 on Figure 1 and an expanded view is provided on Figure 5. The 6,000-gallon gasoline UST was installed in approximately 1975, and pulled in approximately 1982 to allow for construction of the current Screen House. Based on information in Wedron Silica files, the UST was located north of the "Paper Shed" and was 29 feet 2 inches long and 6 feet in diameter. The location of the Paper Shed was identified in historical aerial photography. The following work will be performed:

- Five Geoprobe® soil borings will be drilled on approximate 10-foot centers to depths of approximately 11 feet (estimated 2 to 3 feet below the base of the former USTs) at the approximate locations shown on Figures 1 and 5. The proposed borings target the ends and footprint of the former USTs. Note that the Screen House was constructed adjacent to the former 6,000-gallon gasoline UST which may limit access to the area for a Geoprobe® drilling rig.

- Soil samples will be field-screened for petroleum with a PID capable of measuring in the ppb range, and one soil sample from each boring will be submitted for laboratory analyses for VOCs in accordance with USEPA Method 8260B, lead in accordance with USEPA Method 6010B, and soil pH in accordance with USEPA Method 9045D. Soil samples will be selected for laboratory analyses based on field indications from field screening, odors, staining, etc. If there are no such indicators, the soil sample from the 9- to 11-foot interval (estimated 1 to 3 feet below the former UST) will be submitted for the analyses.

- The work conducted, the field screening and analytical results obtained, a comparison of detected constituent concentrations to Illinois TACO Tier 1 Class I migration to groundwater SROs and recommendations for follow-up investigation activities, as warranted, will be documented in a report. The report will be submitted to the USEPA. If soil VOC concentrations are all less than TACO Tier 1 Class I migration to groundwater SROs, additional investigation will be unnecessary. If TACO Tier 1 Class I migration to groundwater SROs are exceeded, additional activities such as development of Tier 2 TACO levels and/or additional soil investigation and possible investigation of groundwater will be considered. If groundwater investigation is

proposed, consideration of the community-wide presence of petroleum constituents in groundwater will be taken into consideration in the interpretation of results. Specific information to be considered will include: 1) soil data collected at other potential source areas; 2) soil data collected as part of this investigation; 3) groundwater flow directions determined from piezometers and surface water gaging collected as part of this investigation; and 4) other available data. If additional investigation of soil and/or groundwater is warranted, a Workplan will be prepared and submitted to the USEPA in advance of conducting additional investigation.

Pit 2 Reclamation Area

The partially reclaimed Pit 2 is shown on Figure 1. After sand extraction from the quarry ended, the southern portion was reclaimed with various materials. Further evaluation of the groundwater-flow conditions in and around Pit 2 will be conducted before soil samples are collected from soil borings for laboratory analysis. One groundwater-level monitoring location will be completed. We understand that additional wells will be installed by USEPA and IEPA in 2013, and water levels obtained from these wells will also be used to refine groundwater-flow conditions in and around Pit 2. The following work will be performed:

- Install a piezometer in an area north of the existing scale and south of the pipe bridge across the Fox River at the approximate location shown on Figure 1 to aid in the evaluation of groundwater-flow conditions in and around Pit 2. The piezometer will be installed in a boring using rotosonic drilling methods. Continuous soil and rock core samples will be collected for geological characterization. A piezometer will be constructed of 2-inch diameter PVC casing and screened with a 20-foot long, 0.010-inch slot screen placed across the water table and finished with a flush-mount protective casing. Filter pack will be placed around and to approximately 2 feet above the screen, followed by placement of a 2-foot thick bentonite seal and bentonite grout to near grade. The piezometer will be developed to remove residual drill cuttings from the wells to provide hydraulic connection between the aquifer and the well screen. The piezometer will be surveyed for vertical and horizontal control so that groundwater elevations can be determined from the piezometer.

- Two rounds of water levels will be collected from the piezometer, from the piezometers proposed for the task discussed below, from the nine monitoring wells (MW-1 through MW-9) installed by Wedron Silica in 2013, the monitoring well (TW-9) installed by IEPA in 2012, the four surface water monitoring locations containing pressure transducers (Pits 1, 2 and 3 and at the Fox River pump house), the two staff gage locations along Buck Creek, the location in the Fox River at the Highway 21 bridge crossing and the additional monitoring wells to be installed by the USEPA and IEPA in 2013, to which USEPA or IEPA provides access. Each of the water-level monitoring locations, except for those yet to be installed by USEPA and IEPA, are shown on Figure 1.

- Using the water-level data obtained from the above task, locations will be selected in consultation with the USEPA for three Geoprobe® soil borings to be drilled to Total Depth within the reclaimed portion of Pit 2.

- Soil samples will be field-screened for organic compounds with a PID capable of measuring in the ppb range, and two soil samples from each boring will be submitted for laboratory analyses for VOCs in accordance with USEPA Method 8260B. Foreign material encountered in each boring, if any, will be noted. Soil samples will be selected for laboratory analyses based on field indications from field screening, odors, staining, etc. If there are no such indicators in soil, one soil sample will be submitted for analysis from the 6- to 8-foot interval (anticipated to be approximately half the distance to the water table) and one soil sample will be submitted for analysis from the depth interval immediately above the water table (approximately 15 to 17 feet). If groundwater is encountered, a groundwater grab sample will be collected for VOC analysis in accordance with USEPA Method 8260B.

- The work conducted, the field screening and analytical results obtained, a comparison of detected constituent concentrations to Illinois TACO Tier 1 Class I migration to groundwater SROs and recommendations for follow-up investigation activities, as warranted, will be documented in a report. The report will be submitted to the USEPA. If soil VOC concentrations are all less than TACO Tier 1 Class I migration to groundwater SROs, additional investigation will be unnecessary. If TACO Tier 1 Class I migration to groundwater SROs are exceeded, additional activities such as development of Tier 2 TACO levels and/or investigation of soil and possible investigation of groundwater will be considered. If groundwater investigation is proposed, consideration of the community-wide presence of petroleum constituents in groundwater will be taken in the interpretation of results. Specific information to be considered will include: 1) soil data collected at other potential source areas; 2) soil data collected as part of this investigation; 3) groundwater flow directions determined from piezometers and surface water gaging collected as part of this investigation; and 4) other available data. If additional investigation of soil and/or groundwater is warranted, a Workplan will be prepared and submitted to the USEPA in advance of conducting additional investigation.

Evaluation of Groundwater and Surface Water Elevations

In order to further evaluate and define the groundwater-flow regime throughout the Wedron community, three additional piezometers will be installed. The following work will be performed:

- Three piezometers will be installed in a line near the north extent of the Wedron Silica mine operations at the approximate location shown on Figure 1. In addition, surface-water level measurement locations have already been

established in Pits 1, 2 and 3, at two locations on the Fox River, and at two locations along Buck Creek at the approximate locations shown on Figure 1. The piezometers will be installed in borings drilled using rotosonic drilling methods. Continuous soil and rock core samples will be collected for geological characterization. The piezometers will be constructed of 2-inch diameter PVC casing and screen with 20-foot long, 0.010-inch slot screen placed across the water table and finished with a flush-mount protective casing. Filter pack will be placed around and to approximately 2 feet above the screens followed by placement of a 2-foot thick bentonite seal and bentonite grout to near grade. The piezometers will be developed to remove residual drill cuttings from the wells to provide hydraulic connection between the aquifer and each well screen. The piezometers will be surveyed for vertical and horizontal control so that groundwater elevations can be determined from the piezometer.

- Two rounds of water levels will be collected from the three piezometers, from the piezometer proposed in the prior task, from the nine monitoring wells (MW-1 through MW-9) installed by Wedron Silica in 2013, the monitoring well (TW-9) installed by IEPA in 2012, the four surface water monitoring locations containing pressure transducers (Pits 1, 2 and 3 and at the Fox River pump house), the two staff gage locations along Buck Creek, the location in the Fox River at the Highway 21 bridge crossing and in additional monitoring wells to be installed by the USEPA and IEPA in 2013, to which USEPA or IEPA provides access.

- For each round of water-level measurements, water-level data and a groundwater-elevation contour map will be submitted to the USEPA.

Additional Deliverables

Prior to implementation of the work under this Workplan, a Quality Assurance Project Plan (QAPP), Data Quality Objectives (DQOs), and a Health and Safety Plan (HASP) will be prepared for submission to the USEPA, as required under the AOC. After approval by USEPA, the QAPP, DQOs and HASP shall be considered integrated into this Workplan.

After conclusion of implementation of the work set forth in this Workplan, a Final Report will be prepared for submission to USEPA, as required under the AOC.

Schedule

The work set forth in this Workplan will be implemented according to the following schedule, unless otherwise approved by the USEPA:

Activity	Timeframe
Submission of QAPP, DQOs, and HASP	Within 20 days after the Effective Date of the AOC
Commencement of field work at the Tech Center, the Screen House and the Scale House, and commencement of piezometer installations	Within 30 days after USEPA approval of the QAPP and DQOs
Two rounds of water-level measurements	Once per week for two weeks, to begin within 7 days after the later of: – Installation and development of piezometers by Respondents – Installation and development of piezometers by USEPA and IEPA
Submit water-level measurement data, water elevation contour map, and Pit 2 reclamation area boring locations to USEPA	Within 14 days after collection of the first round of water-level measurements
Commencement of borings at Pit 2 reclamation area	Within 14 days after USEPA approval of boring locations
Final Report	Within 45 days after receipt of third-party data validation package for samples from Pit 2 reclamation area borings

Very truly yours,

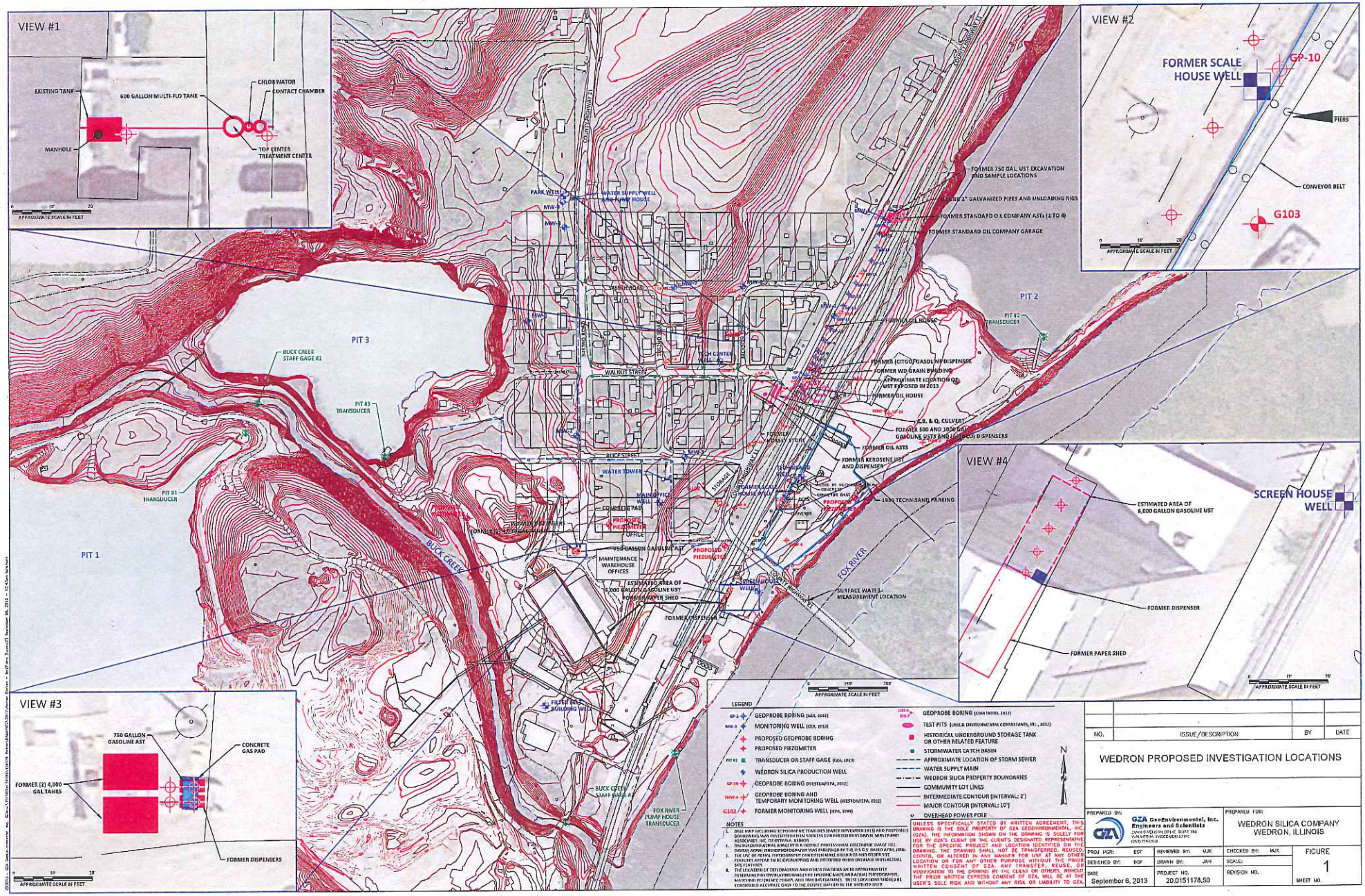
GZA GeoEnvironmental, Inc.

Bernard G. Fenelon, P.G.
Senior Project Manager

Mark J. Krumenacher, P.G
Principal

Attachments

FIGURES


VIEW #1
EXISTING TANK
600 GALLON MULTI-FLO TANK
CHLORINATOR
CONTACT CHAMBER
MANHOLE
TOP CENTER TREATMENT CENTER
APPROXIMATE SCALE IN FEET
VIEW #2
FORMER SCALE HOUSE WELL
GP-10
PIERS
CONVEYOR BELT
G103
APPROXIMATE SCALE IN FEET
VIEW #3
750 GALLON GASOLINE AST
CONCRETE GAS PAD
FORMER (2) 4,000 GAL TANKS
FORMER DISPENSERS
APPROXIMATE SCALE IN FEET
VIEW #4
ESTIMATED AREA OF 6,000 GALLON GASOLINE UST
SCREEN HOUSE WELL
FORMER DISPENSER
FORMER PAPER SHED
APPROXIMATE SCALE IN FEET
PIT 1
PIT 2
PIT 3
BUCK CREEK STAFF GAGE #1
PIT #3 TRANSDUCER
PIT #1 TRANSDUCER
PIT #2 TRANSDUCER
BUCK CREEK
FOX RIVER
WALNUT STREET
BUCK STREET
WATER TOWER
MAIN OFFICE WELL
CONCRETE PAD
PROPOSED PIEZOMETER
OFFICE
750 GALLON GASOLINE AST
MAINTENANCE WAREHOUSE OFFICES
ESTIMATED AREA OF 6,000 GALLON GASOLINE UST
FORMER PAPER SHED
FORMER DISPENSER
SURFACE WATER MEASUREMENT LOCATION
FORMER 750 GAL. UST EXCAVATION AND SAMPLE LOCATIONS
FORMER STANDARD OIL COMPANY ASTs (2 TO 4)
FORMER STANDARD OIL COMPANY GARAGE
FORMER OIL HOUSE
FORMER (CITGO) GASOLINE DISPENSER
FORMER WD GRAIN BUILDING
APPROXIMATE LOCATION OF UST EXPOSED IN 2013
FORMER OIL HOUSE
C.B. & Q. CULVERT
FORMER 500 AND 1000 GAL GASOLINE USTS AND (MOCO) DISPENSERS
FORMER OIL ASTS
FORMER KEROSENE UST AND DISPENSER
1900 TECHNISAND PARKING
FILTER BELT BUILDING WELL
BUCK CREEK STAFF GAGE #2
FOX RIVER PUMP HOUSE TRANSDUCER
APPROXIMATE SCALE IN FEET
LEGEND
GEOPROBE BORING (GZA, 2012)
MONITORING WELL (GZA, 2013)
PROPOSED GEOPROBE BORING
PROPOSED PIEZOMETER
TRANSDUCER OR STAFF GAGE (GZA, 2013)
WEDRON SILICA PRODUCTION WELL
GEOPROBE BORING (WESTON/EPA, 2012)
GEOPROBE BORING AND TEMPORARY MONITORING WELL (WESTON/EPA, 2012)
FORMER MONITORING WELL (EPA, 2004)
GEOPROBE BORING (CRAA SAMPLE, 2012)
TEST PITS (CIVIL & ENVIRONMENTAL CONSULTANTS, INC., 2012)
HISTORICAL UNDERGROUND STORAGE TANK OR OTHER RELATED FEATURE
STORMWATER CATCH BASIN
APPROXIMATE LOCATION OF STORM SEWER
WATER SUPPLY MAIN
WEDRON SILICA PROPERTY BOUNDARIES
COMMUNITY LOT LINES
INTERMEDIATE CONTOUR (INTERVAL: 2')
MAJOR CONTOUR (INTERVAL: 10')
OVERHEAD POWER POLE
N
NOTES
1. BASE MAP INCLUDING TOPOGRAPHIC FEATURES (DATED NOVEMBER 2013) AND PROPERTIES BOUNDARIES WAS DEVELOPED FROM SURVEYS CONDUCTED BY VEGA/VIK SARVER AND ASSOCIATES, INC. OF OTTAWA, ILLINOIS.
2. THE GROUND AERIAL IMAGE IS A GEOSPATIAL PROFESSIONAL ELECTRONIC IMAGE FILE, DIGITAL AERIAL ORTHOPHOTOGRAPHY WAS PUBLISHED BY THE U.S.G.S. DATED APRIL 2006. THE USE OF AERIAL PHOTOGRAPHY CAN OFTEN MAKE BUILDINGS AND OTHER SITE FEATURES APPEAR TO BE OVERLAPPING AND DISTORTED WITH OVERLAID WITH ACTUAL SITE FEATURES.
4. THE LOCATION OF TOPOGRAPHIC AND OTHER FEATURES WERE APPROXIMATELY DETERMINED BY OVERLAYING IMAGES OF EPHEMERIC AMES AND AERIAL PHOTOGRAPHY, MATCHING REFERENCE POINTS, AND TRACING FEATURES. THESE LOCATIONS SHOULD BE CONSIDERED ACCURATE ONLY TO THE DEGREE IMPLIED BY THE METHOD USED.
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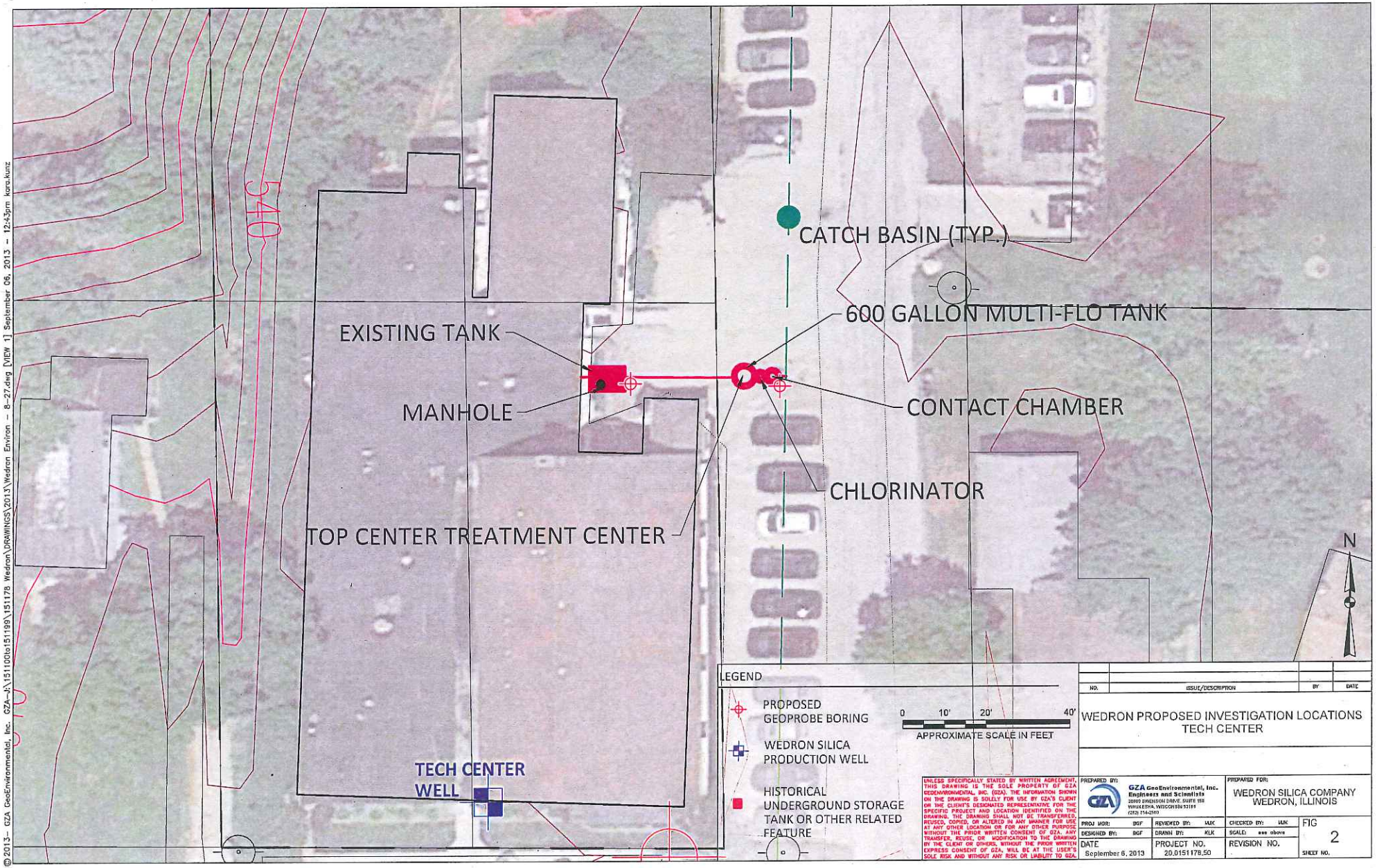
NO.
ISSUE/DESCRIPTION
BY
DATE
WEDRON PROPOSED INVESTIGATION LOCATIONS
PREPARED BY:
GZA GeoEnvironmental, Inc. Engineers and Scientists
PREPARED FOR:
WEDRON SILICA COMPANY
WEDRON, ILLINOIS
PROJ MGR: BGF
REVIEWED BY: MJK
CHECKED BY: MJK
DESIGNED BY: BGF
DRAWN BY: JAH
SCALE:
DATE
September 6, 2013
PROJECT NO.
20.0151178.50
REVISION NO.
FIGURE
1
SHEET NO.



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NO.	ISSUE/DESCRIPTION	BY	DATE

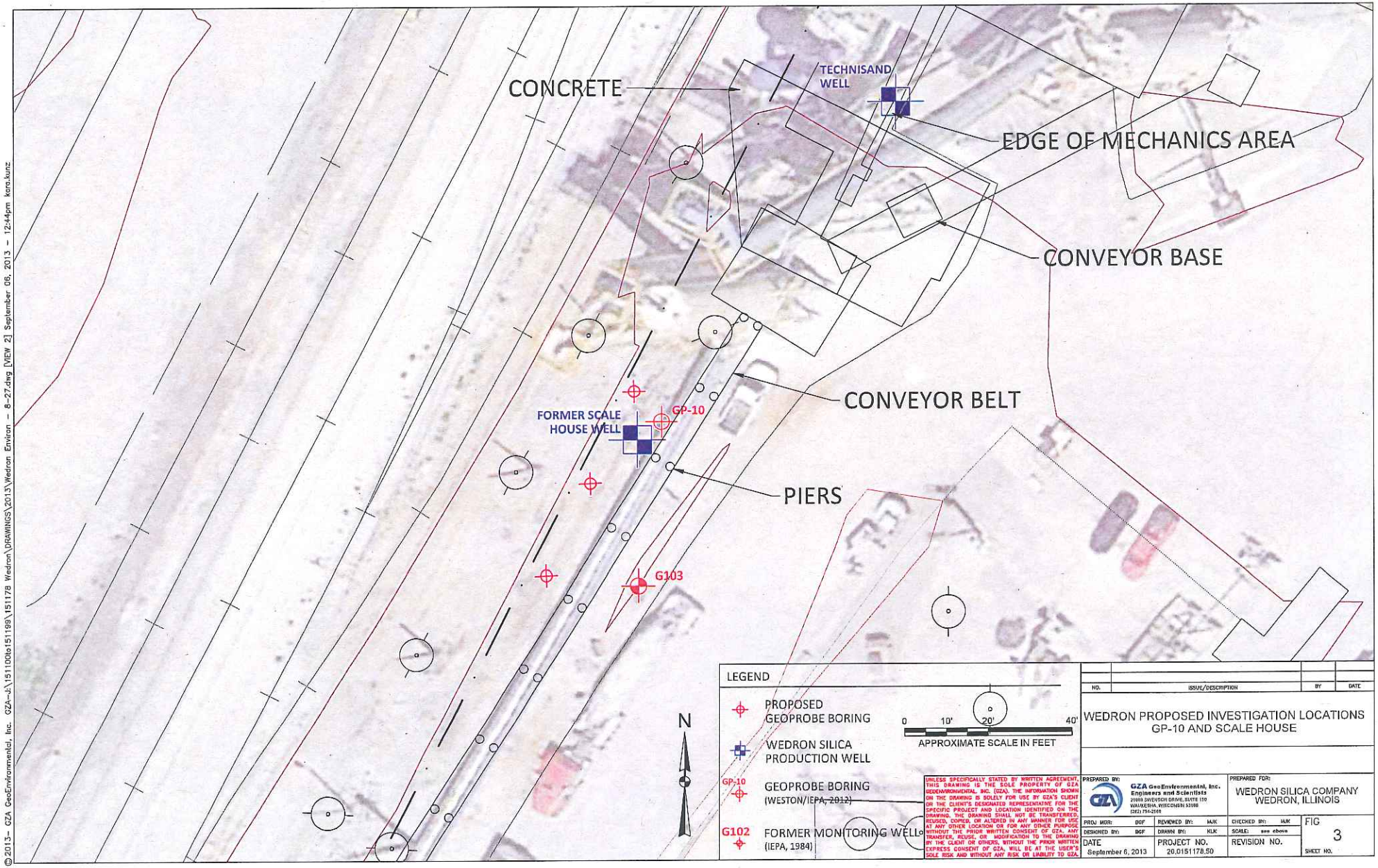
WEDRON PROPOSED INVESTIGATION LOCATIONS
TECH CENTER

PREPARED BY:
GZA GeoEnvironmental, Inc.
Engineers and Scientists
20999 SWENSON DRIVE, SUITE 150
WAUKESHA, WISCONSIN 53186
(262) 754-2580

PREPARED FOR:
WEDRON SILICA COMPANY
WEDRON, ILLINOIS

PROJ MGR: BGF	REVIEWED BY: MJK	CHECKED BY: MJK	FIG	
DESIGNED BY: BGF	DRAWN BY: KLK	SCALE: see above	2	
DATE: September 6, 2013	PROJECT NO. 20.0151178.50	REVISION NO.	SHEET NO.	

© 2013 - GZA GeoEnvironmental, Inc. - G:\151100to151199\151178 Wedron\DRAWINGS\2013\Wedron Environ - 8-27.dwg [VIEW 1] September 06, 2013 - 12:43pm kara.kunz



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NO.	ISSUE/DESCRIPTION	BY	DATE

WEDRON PROPOSED INVESTIGATION LOCATIONS
GP-10 AND SCALE HOUSE

PREPARED BY:
GZA GeoEnvironmental, Inc.
Engineers and Scientists
29000 DIVERSION DRIVE, SUITE 150
WAUKESHA, WISCONSIN 53188
(262) 754-2500

PREPARED FOR:
WEDRON SILICA COMPANY
WEDRON, ILLINOIS

PROJ MGR: BGF	REVIEWED BY: MJK	CHECKED BY: MJK	FIG 3
DESIGNED BY: BGF	DRAWN BY: KLK	SCALE: see above	
DATE September 6, 2013	PROJECT NO. 20.0151178.50	REVISION NO.	SHEET NO.

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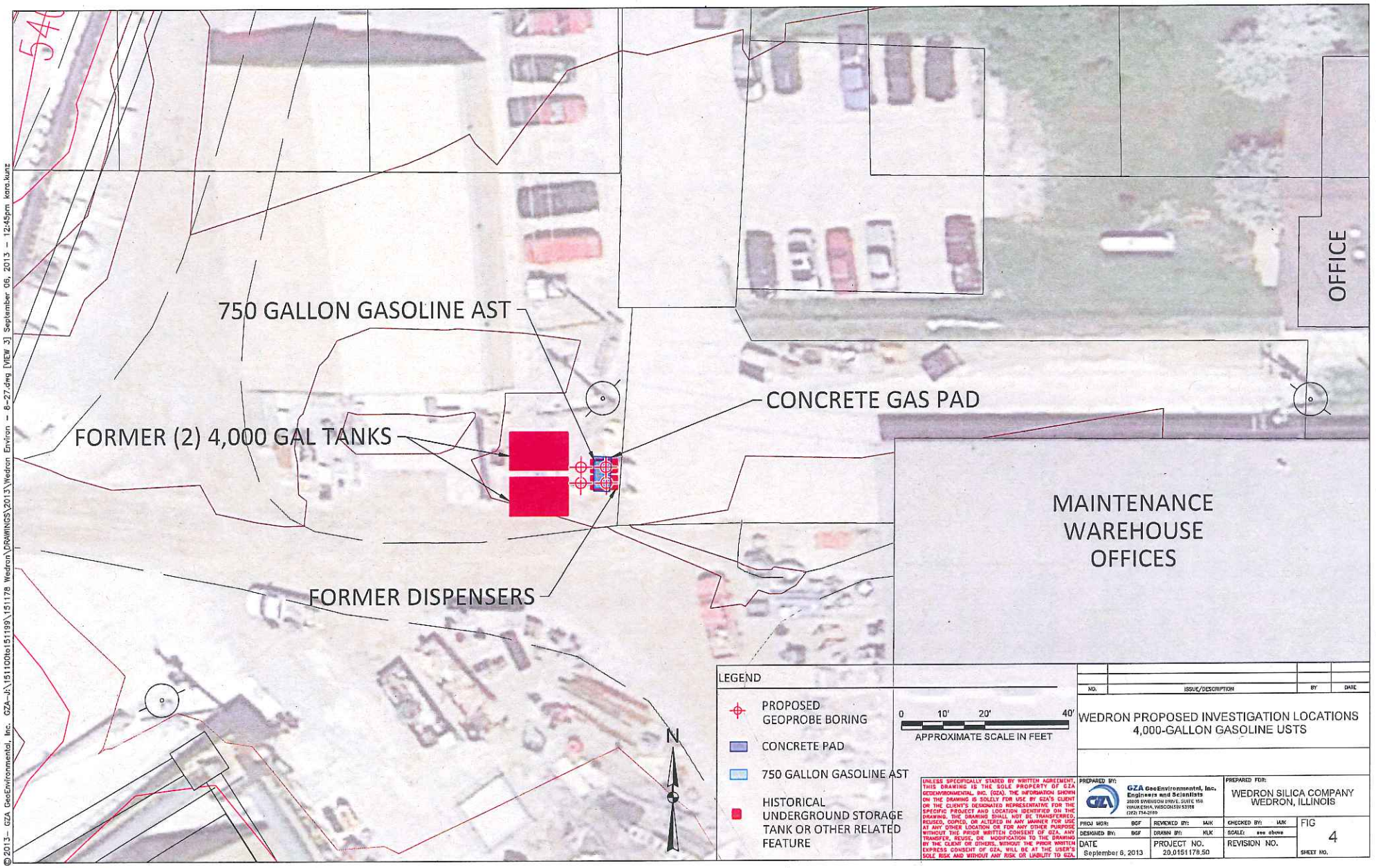

NO. | ISSUE/DESCRIPTION | BY | DATE

WEDRON PROPOSED INVESTIGATION LOCATIONS
4,000-GALLON GASOLINE USTS

PREPARED BY: GZA GeoEnvironmental, Inc. Engineers and Scientists, 2000 EVENSON DRIVE, SUITE 150, VOLK ENA, WISCONSIN 53158, (262) 714-2160

PREPARED FOR: WEDRON SILICA COMPANY, WEDRON, ILLINOIS

PROJ MGR: BGF | REVIEWED BY: MJK | CHECKED BY: MJK | FIG 4

DESIGNED BY: BGF | DRAWN BY: KLK | SCALE: see above

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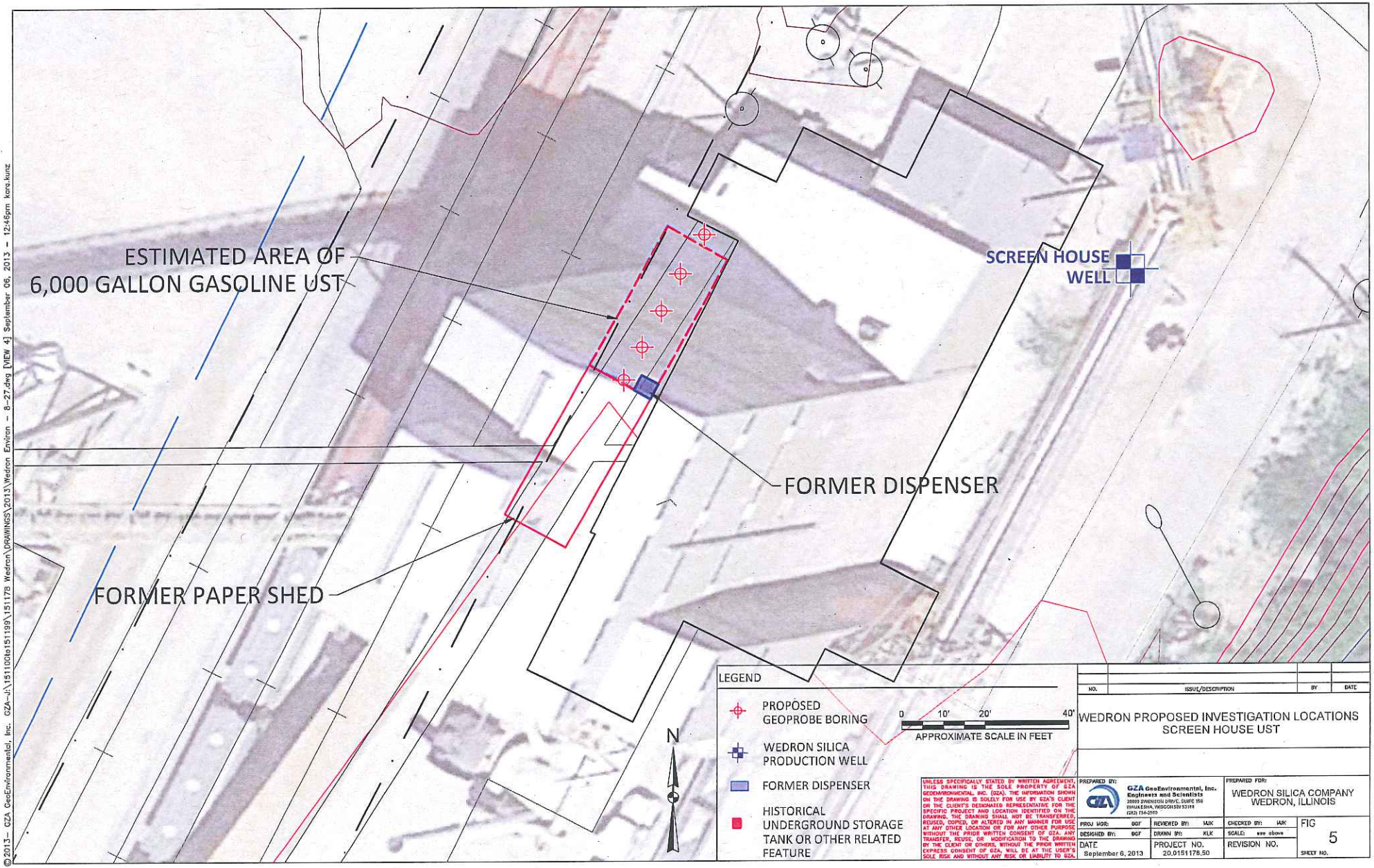

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APPENDIX B

Resumes of Key GZA Team Members

Mark J. Krumenacher, P.G., CPG, CGWP, CHMM

Principal/Senior Vice President/Hydrogeologist

RESUME

Education

B.S., 1985, Geological and Geophysical Sciences, University of Wisconsin-Milwaukee

M.S., 1987, Geological and Geophysical Sciences, University of Wisconsin-Milwaukee

Professional Registration

Professional Geologist, Wisconsin, No. 133

Professional Geologist, Illinois, No. 196-001157

Professional Geologist, Kentucky, No. 2344

Professional Geologist, North Carolina, No. 1936

Professional Geologist, Mississippi, No. 0693

Professional Geologist, Pennsylvania, No. PG004834

Certified Professional Geologist, American Institute of Professional Geologists, No. 10081

Certified Ground Water Professional, Association of Groundwater Scientists and Engineers, No. 450

Certified Hazardous Materials Manager, Institute of Hazardous Materials Management, No. 7749

Areas of Specialization

Mining Geology and Hydrogeology

Midwest geology and Hydrogeology

Pre-Acquisition Environmental Due Diligence

Property Redevelopment Support

Remedial Investigations/Feasibility Studies

Solid and Hazardous Waste RI/FS/RA

Summary of Experience

Mr. Krumenacher has served as Principal, Senior Project Manager and Project Hydrogeologist during the past 23 years on geologic, hydrogeologic, engineering and environmental projects throughout North America. Mr. Krumenacher is a Professional Geologist with licensure nationally and in several states and is a Certified Hazardous Materials Manager (CHMM). He has managed and conducted geologic studies, hydrogeological studies, engineering studies, remedial investigations, environmental assessments, pre-acquisition environmental due diligence and hazardous waste management at various sites including large industrial, commercial and urban redevelopment projects, Federal Superfund sites and state-lead projects.

Experience includes work in 28 states and properties in Canada, Mexico and South America. Responsibilities have involved consultant/owner/designer/developer partnering, contract management and development and implementation of remedial investigations, remedial actions, RCRA Post-Closure Care and Corrective Action, subcontractor management; health and safety monitoring; implementation of remedial technologies including in-situ treatment technologies, slurry cutoff walls, vapor extraction systems and groundwater pumping and treatment systems; design and implementation of focused investigations; monitoring well installation; soil and groundwater sampling and testing; underground storage tank removal; soil-gas surveys; soil and groundwater screening; waste/drum sampling; site reconnaissance; and report preparation.

Mining-related services include more than 100 properties in Wisconsin, Illinois, Indiana, Iowa, Michigan and elsewhere. Services include geologic mapping above and below ground, reserve analysis, mine planning, exploration of reserves, hydrogeologic studies, environmental due diligence, slope stability analyses and engineering, engineering analysis of overburden placement, levee design and construction, foundation engineering and other related services. This work has included open pits and quarries and underground carbonate bedrock and sandstone mines. Geological interpretation in these states can be considered a specialty through education and 20 years experience.

Relevant Project Experience

Geology and Hydrogeology

Mr. Krumenacher has applied his education in general geology, hydrogeology, petroleum geology and geophysics and varied experiences to the interpretation of geologic settings to provide clients and regulators with an understanding of the glacial and bedrock geology, structural geology and hydrogeology on a regional and local level. Those interpretations are necessary for each geologic, environmental and engineering project that involve GZA and range from relatively simple urban settings to expansive multi-acre properties, or multiple properties.

Mining-related services includes work for aggregate producers (sand and gravel and carbonate rock), cement manufacturers (limestone quarries), industrial sand (sandstone) and decorative dimension stone industries (carbonate rock). Services have included work in open pit quarries and carbonate and sandstone underground mines. Mr. Krumenacher also maintains GZA's Mine Safety Health Administration (MSHA) Training Manual for Wisconsin-based staff and has a current training certificate under MSHA Part 46 and Part 48.

Geology and Structural Geology

Complex geologic and engineering projects require an understanding of geologic environments and structure. These types of projects include proposed and existing sand and gravel pits and rock quarries and underground mines and other underground structures such as tunnels and caverns. Work has included evaluation of regional and local structural features that influence migration of groundwater. Examples of various specific projects, most of them confidential, are provided below:

Hydrogeology

Understanding the hydrogeologic setting is essential on every project where groundwater contamination is a concern, sensitive environmental ecosystems are nearby or groundwater is used a resource. Mr. Krumenacher has interpreted the hydrogeologic setting and developed and described hydrogeologic models for hundreds of properties in multiple states and countries. Soil conditions, groundwater characteristics and contaminant migration have been evaluated and described. The properties have included industrial properties that included plating, painting, degreasing, hazardous waste generation and management; sanitary and hazardous waste landfills; and hundreds of underground storage tanks. Oftentimes, the soil and groundwater required development of practical management solutions to enable an engineering design to be implemented. Essentially, all the project descriptions provided below include an evaluation and description of the hydrogeologic setting.

Associate Principal/Geologist - Proposed Theta 13 Neutrino Project, Braidwood, Illinois. The University of Chicago, Fermi Lab and other partners were evaluating the feasibility of constructing two 33-foot diameter vertical shafts and 40-foot span base of shaft caverns to depths of about 650 feet outside the high security perimeter of the Exelon Nuclear Power Generating Station in Braidwood, Illinois. To support the feasibility study, GZA provided geological and engineering field services during rock coring up to 650 feet deep, core hole hydrogeologic and geophysical testing, laboratory testing of soil and rock samples and preparation of a Geotechnical Data Report.

Associate Principal/Geologist - Proposed Underground Aggregate Mine, Illinois; Confidential Client. Provided description of geology, structural geology and hydrogeology for a several hundred-acre proposed underground mine in northeastern Illinois. Interpretations were based on regional studies and reports and rock cores from the property. Challenges included potential complex geologic setting due to location relative to nearby rock quarries, nearby major surface water body, a regional fault zone and sensitive industrial operations.

Associate Principal/Geologist - Active Underground Aggregate Mine, Illinois; Confidential Client. Provided geology, structural geology and hydrogeology interpretation for an active underground mine in northern Illinois. Challenges involved interpretation of groundwater infiltration from several hydrostratigraphic units encountered in and associated with the mine. These units include the overburden, Silurian bedrock and multiple Ordovician bedrock groups; complicated by regional faulting, sensitive nearby ecosystem and local groundwater use.

Associate Principal/Geologist - Geologic and Hydrogeologic Evaluations, Multiple Sites; Confidential Clients. Provided geologic interpretation for multiple clients where a general and specific understanding of the local geology were necessary for assessment and development of open pit and underground mines for aggregate, decorative stone and industrial sand. Interpretations were based on review of regional reports and

site-specific data obtained from site reconnaissance and drilling records. Assessments included evaluation of bedrock thicknesses and overburden thickness, structural geologic concerns and hydrogeologic concerns.

Associate Principal/Geologist - Reserve Analysis, Multiple Sites; Confidential Clients. Provided geologic interpretation at multiple sites for estimating reserves of sand and gravel, carbonate rock, or industrial sand. This work was typically done associated with due diligence of the property associated with potential acquisition. In many cases, this work was performed on a fast-track acquisition schedule with results used for negotiating purchase of the assets.

Remedial Investigations/Feasibility Studies

Associate Principal/Project Manager - Wisconsin; Confidential Client. During construction at an industrial facility in Wisconsin, PCB-containing soil was encountered at the Site. To estimate the volume of PCB-containing soil remaining in the ground at the facility, a site investigation was completed which included approximately 100 soil borings, field screening for PCBs and diesel range organics and analytical testing. The PCB contamination will be addressed by completion of a risk assessment in accordance with the Draft PCB risk assessment guidance established by the USEPA Region 5.

Associate Principal/Project Manager - United Kingdom; Confidential Clients. Plan and manage the implementation of remedial investigations associated with the acquisition or sale of manufacturing facilities across the United States, Canada, Mexico and Brazil. The majority of facilities are various metalworking industries that involve parts degreasing, painting and plating, and also include paint manufacturing, assembly and research and development operations. The predominant chemicals addressed at the contaminated sites include chlorinated solvents, PCBs and metals.

Project Manager - Superfund Site, Spring Arbor, Michigan. Managed two phases of field work and performed report preparation for a hydrogeological and feasibility study that included soil borings, test pits, monitoring well installation/sampling, soil-gas surveys, geophysical seismic surveys, packer/pump groundwater sampling and an extensive residential water supply well sampling program. This study evaluated the hydrogeological condition of a fractured bedrock aquifer and assessed the extent of tetrachloroethylene contamination in the aquifer in Spring Arbor.

Project Manager - Detroit, Michigan; Confidential Client. Developed and implemented a hydrogeological/remedial investigation and remedial action plan for a 17+ acre industrial site in Detroit, Michigan. This study involved over 40 test borings, monitoring wells and soil and groundwater analyses and review of historical geographic and demographic use of the site. The study identified filled-in river channels in an area of industrial fill, which controlled the shallow groundwater flow condition of the site area.

Project Manager - Detroit, Michigan; Confidential Industrial Client. Serving first as Project Geologist and later as Project Manager as part of sale/purchase agreement of a 2+ million square-foot production facility between a large automobile manufacturer and a large engine manufacturer since 1988. Activities associated with the project included a comprehensive environmental site assessment, remedial investigations, remedial engineering design services and remedial system construction oversight. Remedial activities include a groundwater recovery trench, groundwater and oil recovery well systems, removal and closure of underground storage tank systems, and asbestos sampling and abatement. Ongoing activities include remedial system monitoring and interface with the Michigan Department of Natural Resources.

Health and Safety Officer - Bofors-Nobel Superfund Site, Muskegon, Michigan. Level B health and safety monitoring of subcontractor activities associated with installation of field equipment. Implementation of remedial technologies, set-up and utilization of geotechnical soils laboratory, and subcontractor supervision.

Detroit River Study, Detroit, Michigan; Confidential Client. Completed an extensive geophysical study along the Detroit River to determine whether the induced polarization method could be used to detect organic

groundwater contamination. Procedures involved and dated analyzed included the following: data sonics and AquaPulse sub-bottom profilers; Elliot Time Demain Induced Polarization Transmitter; and a Computer-Automated Marine Electrical Resistivity System which consisted of spontaneous potential, longitudinal conductance, apparent resistivity and chargeabilities.

Solid/Hazardous Waste RI/FS/RA

Associate Principal/Project Manager - RCRA Post-Closure Care, Corrective Action and Interim Measures, Former Hallmack Facility, Harrodsburg, Kentucky. Transitioned the project from the previous consultant of six years and developed summary of environmental work dating from the 1970s. Currently manage RCRA Post-Closure Care of three former surface impoundments used to store wastewater sludge from metal plating operations, RCRA Corrective Action consisting of source reduction and hydraulic containment groundwater remediation systems for chlorinated solvent contaminated groundwater from karst limestone bedrock, RCRA Permit Management and general property management. Managed the Interim Measures/Stabilization activities associated with an outdoor area of plating-type waste disposal and a former plating line area; asbestos and lead-based paint abatement, aboveground storage tank closure and industrial hygiene survey. Work completed at the property is overseen by the Kentucky Department for Environmental Protection, Division of Waste Management.

Associate Principal/Project Manager - PCB-Containing Soil Disposal, Wisconsin; Confidential Client. During construction at an industrial facility in Wisconsin, several thousand tons of excavated soil were stockpiled off the property pending evaluation of disposal options. Subsequent analytical testing of the stockpiled soil detected the presence of PCBs. GZA was contracted to oversee the disposal of the excavated soil and subsequent removal of any residual PCB-containing soil from the vicinity of the soil stockpiles. Since the source of the PCB-containing soil was not known, soil containing greater than 25 ppm PCBs required disposal at a facility licensed under the Toxic Substances Control Act (TSCA). Soil containing less than 25 ppm PCBs could be disposed of at a Wisconsin licensed landfill. There was approximately one order of magnitude cost differential between the two disposal options.

In order to reduce disposal costs, a detailed soil sampling and disposal work plan was developed to thoroughly characterize the stockpiled soil and the areas where PCB-containing soil was stockpiled. Utilizing the results of field screening and GC analyses, GZA coordinated with the contractor to minimize the volume of soil requiring disposal at a TSCA facility. PCB-containing soil was removed from beneath and surrounding the soil stockpiles to a concentration less than 1 ppm. During the soil removal process, standing water due to a high groundwater table and considerable precipitation, required implementation of a water management plan. In accordance with USEPA policy, GZA established a water treatment system to remove the majority of PCBs from the water prior to discharge at the local publicly-owned water treatment works (POTW). During the soil removal process, approximately 100,000 gallons of PCB-containing water were treated.

Project Manager - Gratiot County Landfill, Michigan Superfund Site. Responsible for field activities involved with the evaluation of the effectiveness of the present slurry cutoff wall and landfill cap which included test borings, monitoring well installation/sampling, site surveys, packer/pump groundwater sampling, downhole geophysical testing, in-situ testing for determination of hydrogeological properties, design/installation of long-term multiple pressure transducer network and data evaluation. The data collected by GZA was compiled and evaluated with the historical data for the site and a comprehensive final report prepared for the Michigan Department of Natural Resources (MDNR). Also prepared a groundwater monitoring plan for MDNR which is being implemented at the site.

Project Manager - Montmorency/Oscoda Joint Sanitary Landfill, Montmorency County, Michigan. Performed hydrogeologic and engineering analyses of existing site data, prepared a comprehensive hydrogeologic report, and demonstrated to MDNR, on behalf of the County, that perceived groundwater contamination was not an issue due to QA/QC problems associated with groundwater samples and the complex hydrogeologic conditions at the site. Completed a remedial investigation to verify the engineering analysis and

prepared a remedial action plan (RAP) which concluded groundwater monitoring was sufficient to address the regulatory and environmental concerns. The RAP was approved by MDNR. Also prepared a groundwater monitoring plan acceptable to MDNR for implementation. Also responsible for development, implementation and report preparation for a complete hydrogeological investigation and environmental assessment for a proposed 40-acre expansion. The groundwater monitoring plan was revised to provide a comprehensive plan for the existing and proposed landfill areas.

Project Geologist - South Macomb Disposal Authority Landfill Slurry Wall, Macomb Township, Michigan. Managed drilling operations during geotechnical explorations and seismic surveys; aided in preparation of slurry wall design; oversight of slurry wall construction and on-site QA control and field laboratory testing (including API permeability, gradation and slurry and backfill characteristic testing). Assisted with the preparation of a QA/QC report documenting the cutoff wall construction for submittal to the MDNR on behalf of the owner. Managed and implemented geophysical seismic refraction surveys to relate seismic velocities of a highly indurated glacial till to rippability. The objective of the survey was to demonstrate to MDNR that the underlying glacial till which the slurry wall was keyed into had a rippability equivalent to limestone bedrock and that extending the wall through the till was impractical.

Project Manager - RCRA Closure of Hazardous Waste Surface Impoundment, Indianapolis, Indiana; Confidential Industrial Client. Provided QA/QC field and engineering consulting services during the closure of an 8-acre hazardous waste surface impoundment. The surface impoundment formerly received an estimated 100,000 yd^3 of F007 and F009 waste, as defined by RCRA. Activities included pre-remediation sampling and on-site QA control and field laboratory testing (including API permeability, gradation and slurry and backfill characteristic testing) during cutoff wall installation (approximately 120,000 ft^2) surrounding the surface impoundment and keyed into an underlying clay layer (50 to 60 feet deep); and preparation of a QA/QC report documenting the cutoff wall construction for submittal to Indiana Department of Environmental Management on behalf of the owner.

Project Manager - Northern Oaks Recycling and Disposal Facility, Clare County, Michigan. Responsible for development, implementation and report preparation for a complete hydrogeological investigation and an environmental assessment at this 160-acre landfill site in support of the 641 construction permit application to MDNR. Also prepared the Site-Specific Environmental Monitoring Plan for the site which is being implemented in accordance with the operating license. All reports were completed on time and received minimal comment from MDNR despite well organized opposition to the landfill development by a local group.

Project Manager - Michigan; Confidential Landfill Client. Performed engineering and hydrogeologic analyses of site data and a comprehensive hydrogeologic report for an existing 20+ year old landfill site. Initiated a remedial investigation to verify and supplement the engineering analysis and prepared a remedial action plan for the site which was subsequently implemented by the Client.

Pre-Acquisition Environmental Due Diligence

Associate Principal/Project Manager - Former UK-Based Industrial Conglomerate. Completed pre-acquisition and pre-divestiture environmental due diligence of more than 50 sites prior to property transactions throughout the United States, Canada, Mexico and South America. Assessments were generally completed in accordance with ASTM Standards and concentrated on site history reviews, interviews with site personnel, state and local regulatory agency file review where possible, preliminary assessment of on-site hazardous materials and disposal records, underground storage tank compliance issues and preparation of documentation prior to sale or acquisition of properties. Due diligence at industrial locations included a survey of environmental and health and safety compliance and identification of appropriate corrective actions where necessary. Several of the properties involved fast-track Phase II and Phase III site assessments and subsequent remedial action. One site also included remediation of PCB-containing soil on a fast-track basis with issue closure in less than one month. Residual remedial actions are ongoing for the residual companies formed when the industrial conglomerate de-merged in 2000.

Associate Principal/Project Manager - Various Clients. Completed numerous environmental due diligence activities including Phase I ESAs and Environmental and Health and Safety Compliance Surveys for various industries, lenders and legal counsel in the United States and the United Kingdom. The majority of assessments were completed in accordance with the requirements of the ASTM standard for Phase I ESAs. Many ESAs required Phase II and Phase III assessments in order to quantify the environmental liabilities present at the properties. Where remediation was not completed, allocation of costs were evaluated and included in the purchase agreements.

Property Re-Development Support

Project Manager - The University of Toledo, Toledo, Ohio. Prepared and implemented a soil management plan during renovation of an existing building and construction of a new building at the University. The building and property were located at the site of a former glass and plastics research and development facility dating back to the early 1930s, which is listed as a RCRA facility. The site is also situated in an area which was filled between 1900 and 1920, using slag material. Evaluated historical site conditions, hydrogeology and environmental data, completed a geophysical survey and limited site investigation and based on the subsurface conditions present at the site, prepared and implemented a soil management plan which was followed during renovation building construction. Worked closely with the University, their construction management firm, the General Contractors and their subcontractors to manage and integrate subsurface activities such that the construction schedule was not impacted. Through strategic planning and negotiations with the Ohio EPA, demonstrated that the majority of soil which was excavated, could be safely used as fill on University property. Site-specific remediation goals were developed which allowed impacted soil that was not excavated to be left in-place. As such, additional soil was not excavated beyond what was required to facilitate construction. The rationale for allowing impacted soil to remain in-place was based on comparisons of the new building plans, hydrogeologic conditions, and concentrations of the chemical constituents in the soil and groundwater, and an evaluation of the potential health risks to the public and building occupants.

Project Manager/Geologist - Jefferson-Conner Industrial Revitalization Project, Detroit, Michigan. The site consisted of approximately 300 acres of industrial, commercial and residential property in Detroit, Michigan which was targeted for revitalization; the majority of which required characterization and remediation. During the seven-year history of the project, implemented ESAs and parcel sampling plans at approximately 50 industrial and commercial parcels within the project area, developed and implemented strategic Remedial Investigation Plans for each parcel, evaluated the environmental conditions and prepared Remedial Action Plans (RAPs) for each parcel, provided engineering and remedial construction management services during implementation of the RAPs and prepared closure report documentation following completion of remedial activities. Mr. Krumenacher continues to provide assistance to the City of Detroit with issues relating to cost recovery for the environmental aspects of the project.

Professional Activities

Society of Mining, Metallurgy and Exploration
National Stone Sand and Gravel Association
Industrial Minerals Association – North America
Illinois Association of Aggregate Producers
Aggregate Producers of Wisconsin
National ground Water Association
Wisconsin Groundwater Association
Illinois Association of Groundwater Professionals
Federation of Environmental Technologists

Publications

Innovative Application of Induced Polarization for Detecting Organic Ground Water Contamination, Presented at the Petroleum Hydrocarbons and Organic Chemicals in Ground Water: Prevention, Detection and Restoration Conference, November 9-11, 1988, Houston, Texas.

Improved Extraction Efficiency of Polychlorinated Biphenyls from Contaminated Soil Using a Total Halogen Screening Method, Presented at the 13th Annual Waste Testing & Quality Assurance Symposium, July 6-9, 1997, Arlington, Virginia.

RESUME

Education

B.S., 1983, Geological Studies, University of Wisconsin-Milwaukee

M.S., 1987, Geological Services, University of Wisconsin-Milwaukee

Professional Registration

Professional Geologist, Wisconsin, No. 751

Areas of Specialization

Remedial Investigation of Chlorinated Hydrocarbon Sites

Corrective Actions

Groundwater Supply Investigations

Groundwater-Use Impact Assessments

Groundwater and Contaminant Flow Modeling

Bernard G. Fenelon, P.G.
Senior Project Manager/Hydrogeologist

Summary of Experience

Mr. Fenelon has performed remedial investigations and corrective actions, due diligence activities for property transactions, Brownfield redevelopment, groundwater supply development, groundwater-use impact assessments, and geophysical investigations professionally for 25 years.

Remedial investigations and corrective actions for soil and groundwater contamination are performed for industrial, commercial and landfill facilities related to chlorinated solvents, petroleum products, semi-VOCs and metals. The scope of services performed include: 1) evaluating contaminant magnitude and extent in soil, soil vapor and groundwater; 2) evaluating remedial options; 3) implementing remedial actions in soil and groundwater and supervising remedial system operation and maintenance; 4) supervising groundwater monitoring and performing contaminant trend evaluation in support of MNA regulatory closure; 5) evaluating environmental risk and site liabilities as part of pre-acquisition due diligence activities for industry and law firms; and 6) redeveloping urban contaminated Brownfield properties.

Groundwater supply investigations are performed for municipalities, industry, developers, commercial entities and golf courses throughout the upper Midwestern United States in a variety of geological environments including sand and gravel, fractured bedrock (dolomite, limestone, granite, etc.) and sandstone utilizing hydrogeological, geophysical and drilling exploration methods. Well-head protections services and groundwater modeling to optimize well-field development are also performed to aid water supply development.

Groundwater-use impact assessments are performed for the non-metallic mining industry related to dewatering requirements for aggregate extraction, municipalities and industries related to use of groundwater from production wells, agricultural entities related to irrigation wells and developers related to potable groundwater supply requirements and changes in drainage patterns and infiltration characteristics under post-development conditions. Services provided include geologic and hydrogeologic research, soil boring and test well installations, aquifer testing, double-ring infiltrometer testing, groundwater modeling, presentations at zoning and plan commission meetings to provide technical information for projects and for the issuance of conditional-use permits, and answering public concerns regarding various types of proposed projects.

Relevant Project Experience

Remedial Investigations and Corrective Actions

Senior Project Manager, Remediation, 10-Acre Brownfield/ Industrial Site, Confidential Client, Eastern Michigan. GZA has performed several phases of investigation and corrective action at a 10-acre Brownfield site that was formerly an industrial facility with about 60 years of industrial activity.

The cleanup is being performed under an Order of Consent from the USEPA. The site is being prepared for development and the cleanup is subject to State of Michigan Part 201 clean up criteria and final corrective measures are expected to be implemented in 2011 and 2012. The primary risk pathways applicable for the site consist of ecological risk due to metals in soil and metals and volatile organic compounds (VOC) in groundwater discharging to the river that borders the site and vapor intrusion risk related to VOC, primarily trichloroethene (TCE), in homes that border the site and possible future homes built on the site.

Senior Project Manager, Site Investigation, Remediation and Monitoring, Confidential Client, Eastern Wisconsin. GZA is currently conducting an evaluation of potential remedial actions for a chlorinated solvent release of PCE at a manufacturing facility in eastern Wisconsin. The investigation is complicated by the presence of two adjacent manufacturing facilities with similar soil and groundwater contamination issues and solvents detected in a municipal well adjoining the site. Most of the PCE source is bound in a 20-foot deep, nearly-saturated, low permeability clay overlying unsaturated sand and gravel resulting in continuous low solvent loading to the groundwater system. In addition to the presence of solvent in the local water supply aquifer at concentrations above state drinking water standards, PCE has been detected in the shallow groundwater system beneath a residential neighborhood, resulting in potential vapor intrusion through the foundations of the homes and a health risk to the residents. The vapor instruction pathway was evaluated in approximately 15 homes between 2006 and 2011. Mitigation of PCE vapor impacts was necessary for two homes overlying the plume.

Senior Project Manager, Remediation and Monitoring, Former Cool City Cleaners, Two Rivers, Wisconsin. GZA performed remedial actions consisting of MNA groundwater monitoring, PCE vapor assessment and mitigation and enhanced reductive dechlorination (ERD) using emulsified oil at a former dry cleaner under Wisconsin's Drycleaner Environmental Response Fund (DERF). Releases of Stoddard Solvent and migration of petroleum contamination onto the site provided organic carbon substrate for reductive dechlorination of PCE. However, following an increase in chlorinated VOCs in a monitoring well downgradient of the PCE source area, emulsified oil was injected into the aquifer to further enhance reductive dechlorination. Based on the results of post-injection groundwater monitoring, a closure request was prepared and closure with soil and groundwater GIS registries was obtained in 2010.

Senior Project Manager, Site Investigation/Remediation, 20-Acre Former/Industrial Site, Confidential Client, Northern Indiana. In 2008, GZA was asked by this Fortune 100 manufacturing company to review 20 years of soil and groundwater data gathered by other environmental consultants related to multiple trichloroethene (TCE) releases. The site underwent limited soil remediation and about 12 years operation of a 750 gallons per minute (gpm) groundwater pump and treat remedial action under Indiana's voluntary remediation program. Because groundwater remedial progress had slowed considerably and the remedial system was aging and increasingly in need of expensive repair, GZA was asked to evaluate the effectiveness of the remedial system and evaluate remedial alternatives to bring the site to closure. As part of the evaluation, GZA discovered that the groundwater remediation system was improperly designed to control TCE migration beyond the line of pumping wells and was entering residential neighborhoods and resulting in a potential vapor inhalation risk. In 2011, sub-slab soil gas and/or indoor air quality were evaluated in 24 buildings overlying the shallow groundwater plume and 15 ventilation systems were installed in homes with indoor air TCE concentrations exceeding IDEM's 30-year residential indoor air action level. Currently, remedial alternatives are being evaluated for implementation in 2012 to replace the groundwater remediation system which has outlived its useful life and allow redevelopment of the property.

Senior Project Manager, Site Investigation/Remediation, 70-Acre Brownfield/Industrial Site, Confidential Client, Central Pennsylvania. GZA received closure in 2012 for a 70-acre industrial facility with 100 years of industrial activity under the Commonwealth of Pennsylvania Brownfield development program (Act 2) after conducting a comprehensive site characterization and MNA groundwater monitoring. Closure was obtained with minimal active site remediation due to demonstration of limited ecological and vapor intrusion risk from widespread trichloroethene (TCE) contamination across the site and the implementation of institutional controls. Closure was obtained for a budget of less than $1M after a $5.5M pre-sale environmental reserve had been established for the site.

Senior Project Manager, Site Investigation/Remediation, 20-Acre Industrial Site, Confidential Client, Central Kentucky. GZA is currently conducting a comprehensive site characterization for the presence of TCE at an industrial facility in Kentucky. TCE concentrations as high as 650 milligrams per liter have been detected in a 200-foot by 100-foot area of sandstone and shale bedrock. Due to the low-permeability bedrock conditions and lack of nearby receptors to the TCE contamination, a long-term remedial action consisting of ERD has been proposed with placement of 10,000 to 20,000 pounds of organic carbon in dry form on the top of bedrock over the area of highly elevated TCE concentrations. Pilot testing for ERD effectiveness is anticipated for 2009 and 2010, with implementation for the full-scale ERD remedial action sometime in 2011.

Senior Project Manager, Site Investigation/Remediation, Superfund Site, Confidential Client, Central Michigan. In 2003, GZA became the supervising contractor for one the responsible parties (RPs) at a Superfund site in central Michigan. Initially, GZA evaluated data generated over 20 years by numerous consultants for the site that resulted in a significant shift in interpretation of site hydrogeological conditions and the requirement for more aggressive remedial action. Due to the significant change in interpretation of site conditions, GZA made a formal presentation to MDEQ and USEPA, eventually received agency concurrence and saved the RP $1,000,000. Following an extensive evaluation of the groundwater remedial action for compliance with the Consent Decree (CD) and Record of Decision (ROD), GZA proposed replacing the active groundwater remedial action with an MNA remedial scenario. Groundwater pumping was ended and groundwater monitoring performed during a one-year pilot test. Based on the results of the pilot test, the ROD was amended in 2011 to implement an MNA remedial action to meet mixing zone Groundwater-Surface Water Interface Criteria at the surface water discharge, establishment of Alternate Concentration Limits (ACLs) over the groundwater plume to the discharge point and institutional controls over the plume. The amended ROD is expected to save the RP $75,000 to $100,000 per year.

Senior Project Manager, Site Remediation and Monitoring, Confidential Client, South-Central Wisconsin. GZA reviewed almost 15 years of data gathered by another environmental consultant at a site in which an estimated 500,000 pounds of solvent had been released to the surface. The site had undergone about 10 years of active soil and groundwater remediation under Wisconsin's voluntary action program. GZA documented previous site activities and lack of cleanup progress and successfully convinced WDNR that continued operation of the expensive and aging remediation system was not warranted. GZA also identified an improperly constructed well that indicated the false identification of contamination in the regional aquifer and resulted in an incorrect interpretation of the contaminants' migration potential through the low permeability till into the regional aquifer. Given the technical impracticability of source remediation with the current state of remedial technologies, a low technology remedial approach consisting of groundwater monitoring and ERD in the sandstone aquifer was proposed and accepted by WDNR. Continued evaluation of source remediation is being performed on a five-year basis. The change in remedial strategy resulted in substantial savings to the RP on the order of $1,000,000 over 10 years. Implementation of ERD through organic carbon injections began during the Summer/Fall of 2004, with positive results, and a follow-up organic carbon injection and groundwater monitoring were performed in the Fall/Winter of 2008/2009.

Senior Project Manager, Pre-Acquisition Due Diligence, Site Characterization, Remediation and Post-Remediation Groundwater Monitoring, Confidential Client, Central Wisconsin. GZA performed complete turn-key services for the buying party of a manufacturing facility in central Wisconsin that performed plating operations. Due to the detection of chlorinated solvent in the soil and groundwater of the Site during a Phase II ESA and the presence of a municipal water-supply well within 500 feet of the site, GZA provided a pre-acquisition remedial estimate to closure of $1,300,000. As part of the sale, an escrow account was established for the estimated cost of investigation and remediation. GZA performed site investigation and remediation activities at the site between 1995 and the Fall of 2002, and post-remedial groundwater monitoring through 2007. GZA also assisted client with contaminated soil management and UST issues discovered during several post-acquisition building phases. Total environmental cost from pre-acquisition of closure, including environmental activities related to building phases was less than $550,000 resulting in savings of about $750,000 from the initial pre-acquisition remedial estimate. A No Further Action letter was issued by WDNR in 2007.

Senior Project Manager, Groundwater Assessment for Tritium at a Nuclear Power Plant, Southern United States. In 2006 and 2007, GZA performed a review of various plant systems for the potential for an

unmonitored release of impacted or potentially impacted liquid-containing radionuclides in support of the facilities' Groundwater Protection Initiative. The potential for unmonitored releases was combined with the site's hydrogeological conditions and potential receptors to make recommendations for upgrading the site's groundwater monitoring system. Recommendations were also made for collecting additional information to close data gaps identified during the evaluation of site information.

Senior Project Manager, Commercial Brownfield Site Development, Altman, Kritzer & Levick, S.C., Madison, Wisconsin. In 2006, GZA performed pre-acquisition due diligence activities on two adjacent commercial and industrial properties with documented soil and groundwater contamination. The environmental work was performed under the Stet of Wisconsin's VPLE program for redevelopment of a Brownfield property. GZA identified several environmental conditions on the combined properties, the most significant consisting of a release of PCE from a former dry cleaner requiring source remediation. GZA assisted the site developer with the preparation of a Wisconsin Department of Commerce Brownfield Development Grant, which resulted in the award of a $750,000 grant that could be used for development of the site. GZA performed a "not-to-exceed" lump sum remedial action of the PCE source area during construction at the site and performed two years of groundwater monitoring to establish downward contaminant trends in the groundwater system after source remediation. The site received a conditional Certificate of Completion under the VPLE program in 2001, and final closure after placement of the Site on Wisconsin's groundwater GIS registry in 2004.

Senior Project Manager, Site Characterization and Remediation, Sunnyside Mobil, Neviaser Investments, Janesville, Wisconsin. GZA managed the characterization and remediation of a leaking UST site with free-product present in dolomite and sandstone bedrock several hundred feet upgradient and up to 400 feet downgradient of the former USTs. Free product thicknesses were measured at over 2 feet at depths of more than 50 feet. Twelve of the 32 monitoring wells (nine screened in the dolomite and 23 in the sandstone) installed during the investigation were found to have had measurable free petroleum product. Factors complicating the site investigation and remedial action included very large-scale fluctuations in the depth to water in the sandstone (up to 14 feet), upgradient migration of contamination (petroleum product migration above the water table in the dolomite), two aquifers impacted, two adjacent closed leaking UST sites with commingled contamination, fracture flow in the dolomite, separate and isolated groundwater flow systems between the sandstone and the dolomite, and a separate dissolved-phase PCE plume comingling with the petroleum contamination. GZA was complimented by WDNR for its efficient performance of the site investigation and the conclusions made given the complicating geological factors at the site. Due to the technical impracticability of free-product removal in the sandstone and dolomite and the lack of known receptors to the contamination, GZA successfully argued for a low-tech approach to site remediation consisting of passive product collectors and MNA monitoring. The Site received closure in 2006, following free-product collection and groundwater monitoring to support the MNA remedial option.

Senior Project Manager, Site Investigation, Closure, New Waico Development Co., LLP, Milwaukee, Wisconsin. GZA evaluated soil and groundwater conditions in this two-block, multi-family housing development in 2002. As part of the pre-acquisition environmental activities, GZA evaluated Phase II ESA results performed by another environmental consultant and provided "likely" and "worst-case" environmental liability estimates for the buyer, its lending institutions and the Wisconsin Department of Commerce WHEDA program. The "worst-case" environmental liability estimate was required by the WHEDA program as a condition of providing financing for the acquisition. Based on the very limited Phase II ESA data, GZA's "likely" and "worst-case" total investigation and remediation estimates were $90,000 and $720,000, respectively. After completing further environmental investigation activities and evaluating site risk, GZA requested site closure from WDNR. GZA obtained a No Further Action letter for the owner of the site in the Summer of 2003. In the Fall of 2003, the site was placed on Wisconsin's soil GIS Registry and a Deed Restriction was placed on the property specifying site maintenance conditions requirements as detailed in a cap maintenance plan prepared by GZA. GZA was able to complete the environmental investigation through closure for a cost of less than $40,000 resulting in savings of about $50,000 and $680,000 from the "likely" and "worst-case" environmental cost estimate, respectively.

Senior Project Manager, Site Characterization and Remediation of a Federal RCRA Site, Confidential Client, Eastern Tennessee. Managed site investigation, remedial action and risk assessment activities associated with a ¼-mile long chlorinated solvent and plating metals groundwater plume beneath industrial and

residential property originating from a plating operation. Geological conditions consist of sand and gravel over karst limestone and significant DNAPL was detected beneath former process areas of the plant. A $750,000 groundwater recovery system was installed at several on the site and 1,000 feet off the site to remove contaminated groundwater, achieve hydraulic control and restore the condition of the groundwater at off-site locations. The site work was performed under Tennessee Department of Environmental Conservation, RCRA Section, and USEPA.

Senior Project Manager, Milwaukee World Festival Grounds Redevelopment, Milwaukee, Wisconsin. GZA evaluated site-wide environmental and geotechnical conditions prior to two phases of a major reconstruction of the existing Summerfest festival grounds completed between 2001 and 2004. Contamination concerns were focused on the presence of methane, cyanide, PAH and petroleum VOC contamination and the presence of a variety of manufactured gas plant (MGP) and industrial waste/fill that was used to create the land mass several decades ago. GZA obtained a fast-tracked Exemption to Construct on a Landfill through WDNR and prepared a Materials Management Plan to minimize off-site transport and disposal of contaminated residues. GZA performed materials management oversight activities to ensure implementation of the Materials Management Plan during both phases of construction.

Senior Project Manager, Environmental Evaluation for Lakefront Development, Freedom Education Center Wing Addition to the War Memorial Art Museum, Milwaukee, Wisconsin. GZA conducted an environmental evaluation of subsurface conditions during a geotechnical evaluation of the site for the development of this lakeshore facility including a multi-story parking garage and education wing to the War Memorial. Contamination concerns were focused on the presence of methane at two times the lower explosive limit (LEL) in soil beneath the site and petroleum VOC contamination in the groundwater system. Future development on the site will require minimizing off-site disposal of soil generated during construction activities, obtaining an Exemption to Construct on a Landfill from WDNR, engineering controls for the presence of methane below the planned building and proper management of materials (soil and groundwater) generated during site development.

Senior Project Manager, Site Characterization and Remediation, More than 25 Sites, Various Clients, Southeastern Wisconsin and Illinois. GZA managed the characterization and remediation of numerous leaking UST sites in southeastern Wisconsin between 1989 and 2011. Site complexities range from minor petroleum releases in clay environments to free-product present in multiple bedrock aquifers at distances of several hundred feet from the UST release area. Remedial technologies implemented have included soil vapor extraction, groundwater and free product extraction, sparging, excavation and off-site disposal and monitored natural attenuation. The UST sites are administered under Wisconsin's PECFA program. PECFA program reimbursements have typically been in excess of 99% after the responsible party has satisfied the deductible.

Project Manager and Hydrogeologist, Northern States Power Company and James River Corporation Landfills, Ashland, Wisconsin. GZA performed hydrogeological characterization activities in accordance with NR 500 requirements for preparation of Initial Site Reports and Feasibility Reports requiring approval from WDNR. The investigations included soil boring and monitoring well installation, in-situ aquifer testing by measuring groundwater recovery which required more than six months of monitoring, 2-D groundwater flow modeling and leachate generation calculations. Submission of documentation and subsequent review by WDNR project managers resulted in only minimal follow-up on GZA's part to address WDNR concerns and questions.

<u>Groundwater-Use Assessments</u>

Senior Project Manager, Phantom Lake Management District, Town of Mukwonago, Wisconsin. In 2005, GZA performed a hydrogeologic assessment of the Upper Phantom Lake groundwater/surface water basin for the Phantom Lakes Management District (PLMD) Board in order to evaluate the potential impact of pumping from a proposed Village of Mukwonago municipal water supply well located within 1,000 feet of the southern border of Upper Phantom Lake. GZA also provided recommendations to the PLMD and the Village of Mukwonago for pumping test protocols on a test well in 2007 to better assess potential impacts to the Lake and an opinion on the potential to impact the lake based on pumping test results. GZA also provided recommendations to the PLMD in 2012 for conditions for the operating permit by the Village of Mukwonago.

Senior Project Manager, Linnerud Development, Stoughton, Wisconsin. In 2007 and 2008, GZA performed a hydrogeologic assessment of the groundwater/storm water basin for the area of the proposed Linnerud Development in order to evaluate the potential impact of the development's storm water plan on the nearby Virgin Lake basin. The pre-developed conditions of a primarily internally-drained site needed to be maintained after development. GZA aided the engineer in the design of site infiltration in accordance with WDNR's Conservation Practice Standard 1002 *Site Evaluation for Stormwater Infiltration.* GZA performed drilling services and reviewed geotechnical boring logs (more than 150 borings were drilled to support the analysis) and performed grain-size analyses, and conducted double-ring infiltrometer testing in the various proposed infiltration basins.

Senior Project Manager, Pabst Farms, Town of Summit, Wisconsin. In 2005, GZA performed an evaluation of the potential effect of storm water management on the groundwater system and numerous flow-through lakes at the Aurora Hospital proposed for the Pabst Farm property. GZA performed its evaluations using information from USGS topographic maps, area domestic well construction reports, USGS, Wisconsin Geological Survey and SEWRPC hydrogeologic reports, and large-scale pumping tests. GZA's assessment of potential impact to groundwater and surface water was performed with the aid of a ModFlow model constructed for a 16 square-mile area surrounding the Site. The evaluation was successful in showing the negligible impact of the proposed site development on the groundwater system and surrounding lakes through the proper management of storm water.

Senior Project Manager, Various Residential Developments, Town of Richfield, Wisconsin. During 2006 through 2010, GZA performed evaluations of the likely effect water usage from several proposed residential and commercial developments had on the local groundwater system and surrounding surface water bodies. GZA performed its evaluations using information from USGS topographic maps, Wisconsin Geological Survey and Southeastern Wisconsin Regional Planning Commission (SEWRPC) hydrogeologic reports, area domestic well construction reports and incorporated applicable requirements and provisions of the Town of Richfield groundwater protection ordinance, Chapter 59 of the Richfield Municipal Code. GZA's assessments of potential impact to groundwater and surface water were performed by comparing pre-construction to post-construction site infiltration characteristics and water demands and sanitary water return (on-site septic systems) to the area aquifer.

Senior Project Manager, Non-Metallic Mining Industry, More than 15 Sites in Southeastern Wisconsin and Northern Illinois. GZA evaluated the feasibility of carbonate bedrock quarry and sand and gravel pit dewatering, predicted impact of dewatering activities to local wetlands, aquifers and water levels in area domestic wells for numerous sites in southeastern Wisconsin between 1993 and 2010. Site activities have included test well installation, monitoring well and soil boring installation, test pumping and groundwater modeling. GZA has provided professional opinions and answered questions from members of zoning and planning boards and the public in public forums as part of property re-zoning process or issuing of conditional-use permits.

Geophysical Investigations

Senior Geophysicist, Numerous Wisconsin and Illinois Municipalities. Performed seismic refraction and resistivity sounding surveys to evaluate sand and gravel aquifer development potential and to optimize test well drilling locations.

Senior Geophysicist, Numerous Wisconsin and Illinois Municipalities. Performed seismic refraction , azimuthal resistivity, electromagnetic profiling and geothermal surveys, and aerial photography interpretation to evaluate the potential for fractured bedrock aquifer development.

Senior Geophysicist, Numerous sites, Iowa, Illinois, Michigan, Wisconsin. Performed magnetic, magnetic gradiometer, electromagnetic, ground penetrating radar, time-domain electromagnetic, and seismic refraction surveys at environmental sites to detect buried metallic objects groundwater contamination plumes, geologic characteristics, and subsurface disturbed zones.

Project Geophysicist, Southeastern Pennsylvania. Performed seismic refraction depth to bedrock surveys at sites considered for commercial development in southeastern Pennsylvania. Surveys were performed to allow adjustments to site layout plans to minimize the requirements for bedrock excavation to achieve site grades and to estimate the costs to construction related to requirements to excavate bedrock.

Groundwater Supply Investigations

Project Manager, Sand and Gravel Aquifer Exploration for Wisconsin, Illinois and Iowa Municipalities. Performed evaluations for sand and gravel aquifer development by conducting surface geophysical surveys, test drilling and well installation, test well pumping and water sampling. The assessments were performed for municipalities including the Village of Tigerton, Wisconsin **(resulted in the highest capacity production well in the Village by 50% and best water quality in the Village)**; Town of Bristol, Wisconsin; City of Mosinee, Wisconsin; Town of Weston, Wisconsin; City of Kiel, Wisconsin; City of West DeMoines, Iowa; Newton, Iowa; St. Charles, Illinois; Oswego, Illinois, Sioux Falls, South Dakota; and Omaha, Nebraska.

Project Manager, Fractured Bedrock Aquifer Exploration for Wisconsin, Illinois and Iowa Municipalities. Performed aerial photography interpretation and surface geophysical surveys, test drilling and well installation, and test well pumping to evaluate the potential for fractured bedrock aquifer development. The assessments were performed for municipalities including the City of Kiel, Wisconsin **(resulted in the highest capacity production well in the City by more than 200 percent)**; Town of Bristol, Wisconsin; Town of Pewaukee, Wisconsin; City of Plymouth, Wisconsin, Village of Roland, Iowa; and Town of Bolingbrook, Illinois.

Senior Project Manager, Fractured Bedrock and Sand and Gravel Aquifer Exploration for Irrigation Wells in Wisconsin, Illinois and Iowa. Performed exploration services, as described above, for irrigation water supply for private entities consisting of golf courses and green houses.

Senior Project Manager, Groundwater Use Assessment, Industrial and Housing Developments in Southeastern Wisconsin. Assessed the impact of groundwater pumping from the Niagaran dolomite aquifer of proposed developments on existing domestic wells adjacent to the proposed development. Services included review of existing hydraulic information, digital modeling of the proposed groundwater use, and presentation of results at public meetings.

Groundwater Flow Modeling

Senior Project Manager, Proposed Quarry, Northern Illinois. Managed the performance of a 3D ModFlow groundwater flow model in 2006, to assess the affect of quarry dewatering on the local groundwater system and area domestic wells.

Senior Project Manager, Institutional Development, Waukesha County, Wisconsin. Managed the performance of a 3D ModFlow groundwater flow model in 2005, to assess the affect of various storm water management options to the local groundwater system and nearby lakes.

Senior Project Manager, CERCLA Site, Confidential Industrial Client, North-Central Iowa. Managed the performance of a 3D groundwater flow model in 1993, consisting of ModFlow to assess risk of contaminant migration from a CERCLA site to receptors (consisting of a municipal water supply well and surface water body) and to optimize groundwater extraction and hydraulic containment remedial scenarios.

Senior Project Manager, Manufacturing Facility, Amana Appliances, Middle Amana, Iowa. Performed 3D contaminant groundwater flow model in 2000, consisting of ModFlow and MT3D to assess risk of contaminant migration from an industrial site containing chlorinated solvents and petroleum product to a municipal water supply well and potential groundwater recovery scenarios.

Senior Project Manager, Natural Gas Compressor Stations, Several Michigan Sites. Managed 3D contaminant groundwater flow model in 2003, consisting of ModFlow and MT3D to assess risk of contaminant migration to receptors and potential for off-Site migration of contaminants.

Senior Hydrogeologist, Payne and Dolan and Various Developments, Southeastern Wisconsin. Managed 2D and 3D groundwater flow modeling from 1989 to 2000, to predict the impact of proposed quarry dewatering at numerous sites in southeastern Wisconsin using ModFlow to simulate the regional dolomite aquifer.

Professional Activities

Association of Ground Water Scientists and Engineers
Society of Exploration Geophysicists

David G. Bauer, P.G.
Technical Specialist/Hydrogeologist

RESUME

Education

B.S., 1988, Geosciences and Hydrogeology, University of Wisconsin – Milwaukee

2005, Effective Construction Contract and Field Administration

2005, Preventing and Detecting Deficiencies in Design and Construction Documentation

University of Wisconsin-Madison, Department of Engineering

Professional Registration

Professional Geologist, Wisconsin, No. G-175

Asbestos Supervisor, Wisconsin, No. ACS-111287

Asbestos Inspector, Wisconsin, No. AII-111287

Asbestos Supervisor, Virginia, No. 3302 008954

Asbestos Inspector, Virginia, No. 3303 003247

Areas of Specialization

Pre-Demolition Asbestos, Universal Waste and Hazardous Materials Surveys

Building Deconstruction Planning and Scoping

Phase I and II Environmental Site Assessments

Construction Management/Field Superintendent Administration

Corrective Actions

Forensic Due Diligence Relative to Real Estate Transactions

Summary of Experience

Mr. Bauer's projects involve the management of asbestos-containing materials, lead-based paint and other hazardous materials related to renovation and demolition activities in buildings, as well as assisting building owners in the long-term management of asbestos in their buildings. His work includes asbestos and hazardous materials surveys and design specifications; management of asbestos, lead paint and hazardous materials abatement projects; screening and Phase I and II Environmental Site Assessments; monitoring of workplace exposure to chemical and physical hazards; and indoor air quality clearance assessments after asbestos abatement. In addition, his responsibilities include coordination of risk-based, "fast-track" remedial investigation/feasibility studies; evaluation and reduction of extensive field and laboratory analytical data sets as they pertain to conceptual remedial alternative analysis; preliminary remedial design; final remedial design; and quality assurance/quality control. He is responsible for the interpretation and presentation of technical information and has been directly involved in technical negotiations with various state and federal agencies and has participated in public meetings as part of community relations. His experience includes providing services for municipalities, governmental agencies and private sector industrial and non-industrial clients.

Relevant Project Experience

Owner's Representative/Brownfield Remediation Management. Owner's representative and on-site construction manager for the State of Wisconsin's largest Brownfield development at a blighted property located in the Menomonee River Valley in the City of Milwaukee, Wisconsin. Responsibilities to achieve the owner's desired development objective included managing multiple contractors involved with removal and containment of approximately 500,000 cubic yards (cyds) of construction/demolition debris comingled with asbestos, abandonment of extensive sewer networks underlying the future development and implementation of design controls to prevent the migration of chlorinated volatile organic compounds (VOCs), fuel oil product and other contaminants identified in soil and groundwater underlying the site. Acting as on-site "competent person" for the project, responsibilities included the management and assurance of strict conformance to asbestos removal, demolition and remediation specifications, employment of "best management" practices and cost control inventory.

Owner's Representative/Brownfield Due Diligence Management. Owner's representative and field operations manager for a due diligence investigation of a 150-acre, tax delinquent, industrial property located in the Menomonee River Valley in the City of Milwaukee, Wisconsin. Responsibilities included managing and directing multiple contractors and field professionals involved with execution of the soil and groundwater quality investigation work plans and specifications; and assembled, quantified and interpreted multiple environmental media data sets which revealed several identifiable areas of environmental concern,

the technical foundation for development of remedial controls used to prepare the property for redevelopment as a light industrial park and green space.

Owner's Representative/Project Manager. Provide annual asbestos inspections for international avionics manufacturer at three facilities located in Milwaukee, Wisconsin as part of client's corporate asbestos operation and maintenance (O&M) plan.

Owner's Representative/Project Manager. Conducted building surveys of a 125,000 square-foot department store and 15,000 square-foot automotive service business at a mall redevelopment project in Virginia for suspect asbestos-containing materials (ACMs), lead-based paint (LBP), polychlorinated biphenyl (PCB)-containing light ballast and hazardous materials in on-site structures. Collected samples of suspect ACMs and submitted to laboratory for analysis using polarized light microscopy (PLM). Collected samples of suspect LBPs and submitted for analysis. Prepared independent Pre-Demolition Asbestos, Universal and Hazardous Materials Survey Report for use in materials management planning prior to planned demolition.

Owner's Representative/Project Manager. Prepared asbestos, universal waste and hazardous materials abatement scoping document on behalf of developer for competitive bid to multiple contractors involved with the demolition of large department store in Virginia as part of overall site redevelopment, and assisted owner in selecting abatement and demolition contractors.

Owner's Representative/Project Manager. Conducted pre-demolition surveys for ACMs, LBP and hazardous materials at a 65-unit apartment complex to be razed in Rockford, Illinois. Provided abatement oversight and clearance air monitoring prior to building demolition. Prepared Pre-Demolition Asbestos, Universal Waste and Hazardous Materials Survey Report for use in materials management planning prior to demolition. Prepared design specifications for competitive bid to multiple abatement and demolition subcontractors as part of site redevelopment and assisted owner in selecting abatement and demolition contractors.

Owner's Representative/Project Manager. Reviewed pre-demolition survey documents prepared by others for completeness of a 350,000 square-foot industrial building located in Indiana. Prepared full set of abatement scoping and demolition contract documents, administered bid walk-through and assisted owner in selecting abatement and demolition contractors.

Owner's Representative/Project Manager. Conducted a building survey for suspect ACMs, LBP, PCB-containing light ballast and other hazardous materials in a architecturally historic building located at a Brownfield redevelopment of a former industrial site in the City of Milwaukee, Wisconsin. Prepared an Asbestos, Universal Waste and Hazardous Materials Survey Report for use in designing renovations of the building.

Owner's Representative/Project Manager. Conducted a building survey for suspect ACMs, LBP, PCB-containing light ballast and other hazardous materials for a major health care provider at hospitals located in Wisconsin. Prepared Asbestos, Universal Waste and Hazardous Materials Survey Reports for use in designing renovations of multiple wings within the hospitals. Conducted third party supervision of abatement contractors and clearance air sampling within containments after abatement was completed.

Project Hydrogeologist/Project Manager. Managed and directed a Brownfield investigation and remediation of a tax delinquent hazardous waste site property in West Allis, Wisconsin. The site has since been closed and turned over to the City of West Allis pending future beneficial redevelopment.

Owner's Representative/Brownfield Due Diligence Management. Provided owner's representative services and managed environmental due diligence and geotechnical services at a blighted property selected for construction of a $107 million, 450,000 square-foot newspaper printing facility. Responsibilities included assisting the owner in obtaining permits and exemptions to construct on the property, identifying and

implementing design controls in areas of environmental concern and the collection of deep subsurface data pertinent to the facility's press room foundation design.

Owner's Representative/Pre-Demolition Management. Designed, coordinated and managed material handling and demolition specifications for a prominent developer in the City of Milwaukee, Wisconsin relative to the demolition of a 360,000 square-foot tannery facility developed into luxury condominiums, apartments and retail space.

Owner's Representative/Construction Site Management. Provided owner's representative services and managed the completion of a $4 million, 30,000 square-foot distribution center, assisting the construction manager with preparation of trade-specific bid packages, solicitation of those bid packages and selection of the various trade contractors. Coordinated and directly supervised site civil layout and grading work, structural steel erection, concrete and foundation construction, electrical, building control(s), plumbing, heating, ventilation and air-conditioning (HVAC), glass, specialty door, drywall, carpentry, painting, tele/data wiring, fire protection, acoustical ceiling tile, roofing and landscape contractors from groundbreaking through project closeout. Ensured that contractors comply with construction documents and adhere to project schedule and projected project budget.

Owner's Representative/Construction Superintendent. Provided owner's representation and ensured contractor conformance with asbestos abatement, demolition and engineered remedial control specifications during preparation of a 20-acre parcel for construction of a 130,000 square-foot, $95 million motorcycle museum complex.

Professional Activities

OSHA, 40-Hour 29 CFR 1910.120(e) (2) Safety at Hazardous Materials Sites Training
OSHA, 8-Hour Safety at Hazardous Materials Sites Refresher Training, Annually
MSHA, 8-Hour Mine Safety Training Part 46 and Part 48, Annually

Christopher J. Ainsworth
Engineer I

RESUME

Education

B.S., 1993, Geography - Earth Science Option, University of Wisconsin - Stevens Point

Areas of Specialization

Subsurface Investigation
Soils Technician
Landfill Construction Quality Assurance
Soil and Groundwater Investigations

Summary of Experience

Mr. Ainsworth offers over 14 years of professional experience in environmental and geotechnical investigations, environmental and remedial construction, remediation system operation and maintenance, quality control and quality assurance testing, engineering inspection, geology, geotechnical laboratory testing, and soil and groundwater contamination investigations. Mr. Ainsworth's project responsibilities have included all aspects of site investigation and construction quality inspection, soil and groundwater investigations associated with industrial sites, underground storage tanks and industrial landfills; domestic well investigations and construction oversight of remedial activities on a proposed National Priorities List Site. Relevant project experience includes:

Relevant Project Experience

Subsurface Investigations – Multiple Sites. Project work includes all aspects of site investigation and data collection for geotechnical and environmental investigations utilizing hand tools and geophysical methods and subcontracted methods including test pitting with backhoes, Geoprobe® sampling, auger drilling, wash rotary drilling, air rotary drilling and rock drilling and coring. Responsibilities included boring citing, soil and rock classification and boring abandonment. Groundwater investigations included monitoring well installation, development, surveying, abandonment and sampling as well as field permeability testing. Mr. Ainsworth's investigation experience includes sites throughout Wisconsin and several other states including Illinois, Iowa, Minnesota, Michigan, Indiana, West Virginia, Pennsylvania, Missouri, Ohio and Kentucky.

Construction Oversight and Quality Control/Quality Assurance Testing. Project work includes monitoring of construction activities, documenting field conditions and testing results, and ensuring construction details meet quality criteria of the specifications. Day-to-day activities include nuclear density testing, collecting field laboratory samples including soil, concrete and asphalt, soil mechanics and classification, documentation of construction activities, coordination of multiple contractors, interfacing with clients and owner's representatives. Specific site experience includes:

- **Field QA/QC Testing and Inspection, Waste Management, Inc. Solid Waste Landfill Cell Expansion, Chastang, Alabama.** Activities included clay liner installation and fabric membrane liner installation.

- **Field QA/QC Testing and Inspection, Waste Management, Inc. Solid Waste Landfill Cell Expansion, Franklin, Wisconsin.** Activities included clay liner installation, geomembrane and geotextile liner installation, leachate collection system installation and placement of stone drainage layer on four separate 6-acre cell expansions.

- **Field QA/QC Testing and Inspection, Waste Management, Inc. Solid Waste Landfill Capping and Closure, Franklin, Wisconsin.** Activities included clay liner installation and berm construction associated with capping activities.

- **Field Construction Quality Assurance Inspection, Industrial Landfill Closure, Midwestern United States.** Activities included daily supervision of the capping and closure of a 38-acre industrial landfill. Earthwork activities included over 290,000 cyds of soil and waste regrading, the installation and QA/QC testing of over 92,000 cyds of native clay liner, with over 70,000 cyds of cover soil. Daily activities included quality of imported clay material, nuclear density testing, clay sampling and proctor testing and inspection activities for compliance with a Quality Assurance Project Plan approved by USEPA. Responsible for thorough data documentation and reporting.

- **Field Construction Quality Assurance Inspection, Lime Creek Nature Center Industrial Waste Consolidation and Closure, Midwestern United States.** Activities included daily supervision of the capping and closure of a 10-acre industrial landfill. Earthwork activities included over 206,000 cyds of soil and waste regrading, the installation and QA/QC testing of over 38,000 cyds of native clay liner, with over 30,000 cyds of cover soil. Daily activities included quality of imported clay material, nuclear density testing, clay sampling and proctor testing and inspection activities for compliance with a Quality Assurance Project Plan approved by USEPA. Responsible for thorough data documentation and reporting.

- **Field QA/QC Testing and Inspection, Home Depot Building Construction, Racine, Wisconsin**. Activities included nuclear density testing, concrete and asphalt inspection and laboratory sampling and client interaction.

Field Implementation of Biostimulant Injection Remediation Activities, Confidential Client, Watertown, Wisconsin. Directed field activities including delivery of approximately 495,000 gallons of a biostimulant mixture into a network of nine injection wells. On-site activities included network piping construction, mixing approximately 30,000 pounds of injection mass into solution and delivery of mixture to injection wells.

Field Inspection and Installation of Methane Venting System, Bayshore Town Center, Glendale, Wisconsin. Inspected and assisted in the installation of a methane venting system including placement of a permeable crushed stone layer for methane collection, piping installation and geomembrane placement.

Field Inspection and Installation of Methane Venting System, Home Depot, Chicago Ridge, Illinois. Inspected and assisted in the installation of a methane venting system including placement of a permeable crushed stone layer for methane collection and piping installation.

Engineering Technician, Old Kyanize Paints, Springfield, Illinois. Participated in the operations and maintenance of active remediation system and the collection of groundwater samples associated with that system.

Engineering Technician, Confidential Client, Belvidere, Illinois. Directed the collection of groundwater samples and shared responsibilities with the collection of soil samples using a Geoprobe® system.

Engineering Technician, Operations and Maintenance, Various Sites. Participated in the operations and maintenance of active remediation systems.

Engineering Technician, Town of Lisbon, Wisconsin. Directed the quarterly domestic well sampling for the township to determine the extent of bacterial contamination. Participated in the data analysis and interpretation associated with the sampling.

Geologist, Confidential Client, Springfield, Ohio. Collection of soil and groundwater samples and installation of monitoring wells to determine the extent of various contaminants.

Geologist, Various Investigations, Various Clients. Responsibilities include tasks associated with removal/closure of UST sites including excavation/removal oversight, sample collection, soil borings and monitoring well installation, development, surveying and sampling.

Professional Activities

Health and Safety Training as Required by 29 CFR 1910.120
Hazardous Site Supervisor Training as Required by 29 CFR 1910.120
Certified in Nuclear Density Gauge Operation
Certified ACI Concrete Field Testing Technician – Grade 1
Certified Site Assessor – Wisconsin Department of Commerce

RESUME

Education

B.A., 2003, Psychology/Sociology, California State University-Hayward

Graduate Studies, 2004-2005, Teaching, California State University-Hayward

Professional Registration

Asbestos Inspector, Wisconsin, No. AII-123176

Certified Site Assessor, Wisconsin, No. 1068516

Areas of Specialization

Environmental Compliance

Subsurface Investigation

Soil Technician

Soil and Groundwater Investigations

Daryl A. Lamb
Environmental Specialist

Summary of Experience

Mr. Lamb has more than 14 years of professional experience in environmental compliance, environmental site investigations and environmental remediation. He has developed a proven track record of developing and executing work assignments with professionalism and integrity, as a result he is a primary go-to environmental professional for a variety of environmental consulting activities throughout the Midwest region focusing on work in Illinois, Wisconsin, Michigan and Ohio. Additionally, his experience includes substantial assignments in California, Indiana, Iowa, Kansas, Kentucky, Minnesota, Pennsylvania, Oklahoma, Texas and Virginia. He has provided consulting services to major clients including ANR Pipeline Company, California Department of Transportation, Chrysler Group LLC, Home Depot, Johnson Controls, Inc., Santa Clara County California, Triton Energy Corporation and We Energies.

Relevant Project Experience

Environmental Compliance and Management - Mr. Lamb's experience includes management of environmental compliance matters, including:

- Review of cleanup goals and case closures with regulatory agencies concerning emergency response actions.
- Performance of chemical lab pack services involving identification of hazardous and other regulated materials, characterization, manifesting, shipment and recordkeeping.
- Responsible for environmental compliance with Hazardous Waste Disposal Program requirements involving waste identification, profiling, manifesting, packaging and disposal of hazardous wastes. Responsibilities included preparing detailed cost estimates, arranging contractors and subcontractors, cost tracking, preparing invoices and negotiating contract pricing/change orders.
- Responsible for initiating, maintaining and supervising safety precautions in compliance with OSHA regulations and state and local regulations.
- Trained personnel in sampling techniques, procedures and use of sampling and safety-related field equipment.
- Created, implemented and maintained inventory control of health and safety equipment.
- Performed compliance monitoring for soil-vapor and groundwater monitoring programs at multiple facilities involving an array of chemical constituents.

Environmental Investigations - Mr. Lamb's experience includes Phase I ESAs and subsurface environmental investigations at simple to complex sites. Project work included various aspects of site investigation and data collection, including:

- Geoprobe® and auger drilling investigation;
- Air and wash-rotary drilling;
- Soil and rock classification;
- Test pits;
- Soil sampling and screening;
- Monitoring well installation, development and sampling;
- Borehole and well abandonment; and
- Surficial mercury investigations.

Remedial Actions - Mr. Lamb has overseen emergency response cleanup actions that included highway spills and illegal drug laboratories and constructed, operated, maintained and repaired various soil and groundwater remediation systems. Specific experience included:

- Supervised and conducted the cleanup of illegal drug manufacturing laboratories, including the identification, packaging and shipping of hazardous materials and decontamination of property.
- Managed all phases of soil and groundwater remediation systems including UST removal, installation, operation and maintenance of air sparging/soil vapor extraction and groundwater treatment systems for petroleum and chlorinated VOC contamination. Presently administers multiple groundwater remediation systems at a large industrial facility.
- Operated several excavation dewatering systems designed to remove phase-separated hydrocarbons from the groundwater during the closure of a major Southern California oil production drilling site and tank farm. Mr. Lamb assisted in the chlorination and aeration of the produced water from the excavation. Water treatment was required to meet discharge standards of the local sewer district.
- Supervised remedial activities at a site in Michigan regulated under RCRA. Activities included excavation, removal and off-site disposal of non-hazardous-contaminated soil, including various waste materials from five discrete locations. Over 200,000 gallons of impacted excavation water were treated via activated carbon pressure filtration vessels prior to subsequent discharge into the local POTW.

Professional Activities

HAZWOPER 40-Hour and Supervisor Training
OSHA 10-Hour Construction Safety Training
MSHA Part 46 and Part 48 New Miner and Experienced Miner Training
Confined Space Entry
US DOT General Hazardous Material Training

APPENDIX C

Laboratory Certifications and Accreditations

STATE OF ILLINOIS

ENVIRONMENTAL PROTECTION AGENCY
NELAP - RECOGNIZED
ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

ENVIRONMENTAL CHEMISTRY CONSULTING SERVICES, INC.

2525 ADVANCE ROAD

MADISON, WI 53718

NELAP ACCREDITED

ACCREDITATION NUMBER #200062

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Primary Accrediting Authority: Louisiana Department of Environmental Quality

Celeste M. Crowley
Acting Manager
Environmental Laboratory Accreditation Program

Janet Cruse
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 003174
Expiration Date: 04/30/2014
Issued On: 05/09/2013

State of Illinois
Environmental Protection Agency

Certificate No.: 003174

Awards the Certificate of Approval

Environmental Chemistry Consulting Services, Inc.
2525 Advance Road
Madison, WI 53718

According to the Illinois Administrative Code, Title 35, Subtitle A, Chapter II, Part 186, ACCREDITATION OF LABORATORIES FOR DRINKING WATER, WASTEWATER AND HAZARDOUS WASTES ANALYSIS, the State of Illinois formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed below.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part 186 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part 186. Please contact the Illinois EPA Environmental Laboratory Accreditation Program (IL ELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Illinois is not an endorsement or a guarantee of validity of the data generated by the laboratory.

Hazardous and Solid Waste, Organic

8015B

Diesel range organics (DRO)		

8081A

4,4'-DDD	4,4'-DDE	4,4'-DDT
Aldrin	alpha-BHC	alpha-Chlordane
beta-BHC	Chlordane - not otherwise specified	delta-BHC
Dieldrin	Endosulfan I	Endosulfan II
Endosulfan sulfate	Endrin	Endrin aldehyde
Endrin ketone	gamma-BHC (Lindane)	gamma-Chlordane
Heptachlor	Heptachlor epoxide	Hexachlorobenzene
Methoxychlor	Mirex	PCNB
Toxaphene	trans-Nonachlor	

8082

PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260		

8095

1,3,5-Trinitrobenzene	1,3-Dinitrobenzene (1,3-DNB)	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
2,4,6-Trinitrotoluene	2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)
2-Amino-4,6-dinitrotoluene (2-Am-DNT)	2-Nitrotoluene	3,5-Dinitroaniline (3,5-DNA)
3-Nitrotoluene	4-Amino-2,6-dinitrotoluene (4-Am-DNT)	4-Nitrotoluene
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	Nitrobenzene	Nitroglycerine
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	Pentaerythritoltetranitrate (PETN)	

8151A

2,4,5-T	2,4,5-TP (Silvex)	2,4-D
2,4-DB	4-Nitrophenol	Acifluorfen
Bentazon	Chloramben	Dalapon
DCPA diacid	Dicamba	Dichloroprop
Dinoseb	MCPA	MCPP
Pentachlorophenol	Picloram	

8260B

1,1,1,2-Tetrachloroethane	1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane	1,1-Dichloroethane	1,1-Dichloroethene
1,1-Dichloropropene	1,2,3-Trichlorobenzene	1,2,3-Trichloropropane
1,2,4-Trichlorobenzene	1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane (DBCP)
1,2-Dibromoethane (EDB)	1,2-Dichlorobenzene	1,2-Dichloroethane
1,2-Dichloropropane	1,3,5-Trimethylbenzene	1,3-Dichlorobenzene

State of Illinois
Environmental Protection Agency

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2525 Advance Road
Madison, WI 53718

Hazardous and Solid Waste, Organic

8260B

1,4-Dichlorobenzene	1,4-Dioxane	1,3-Dichloropropane
2-Butanone (Methyl ethyl ketone, MEK)	2-Chlorotoluene	2,2-Dichloropropane
4-Chlorotoluene	4-Methyl-2-pentanone (Methyl isobutyl ketone, I	2-Hexanone
Benzene	Bromobenzene	Acetone
Bromodichloromethane	Bromoform	Bromochloromethane
Carbon disulfide	Carbon tetrachloride	Bromomethane
Chlorodibromomethane (Dibromochloromethan	Chloroethane	Chlorobenzene
Chloromethane	cis-1,2-Dichloroethene	Chloroform
Dibromomethane	Dichlorodifluoromethane	cis-1,3-Dichloropropene
Ethylbenzene	Hexachlorobutadiene	Dichloromethane (Methylene chloride)
Methyl ethyl ketone	Methyl isobutyl ketone	Isopropylbenzene
m-Xylene	Naphthalene	Methyl-t-butyl ether
n-Propylbenzene	o-Xylene	n-Butylbenzene
p-Xylene	sec-Butylbenzene	p-Isopropyltoluene
tert-Butylbenzene	Tetrachloroethene	Styrene
Toluene	trans-1,2-Dichloroethene	Tetrahydrofuran
Trichloroethene	Vinyl chloride	trans-1,3-Dichloropropene
		Xylenes (total)

8270C Mod_Farm Chemicals

Acetochlor	Alachlor	Atrazine
Atrazine Desethyl	Atrazine Desisopropyl	Butylate
Chlorpyrifos	Cyanazine	Diazinon
Dimethenamid	Dimethoate	EPTC
Ethalfluralin	Fonophos	Metolachlor
Metribuzin	Parathion ethyl	Parathion methyl
Pendimethalin	Phorate	Prometon
Propachlor	Propazine	Simazine
Terbufos	Triallate	Trifluralin

8270D

1-Methylnaphthalene	2-Methylnaphthalene	Acenaphthene
Acenaphthylene	Anthracene	Benzo(a)anthracene
Benzo(a)pyrene	Benzo(b)fluoranthene	Benzo(g,h,i)perlyene
Benzo(k)fluoranthene	Bromoxynil	Captan
Carbaryl (Sevin)	Carbofuran (Furaden)	Chrysene
Dibenz(a,h)anthracene	Dichlorovos	Dimethoate
Dinoseb	Disulfoton	Ethion
Fluoranthene	Fluorene	Indeno(1,2,3-cd) pyrene
Malathion	Methyl parathion	Naphthalene
Parathion	Pentachlorophenol	Phenanthrene
Phorate	Phosmet	Pyrene
Terbufos	Trifluralin	

8330A

1,3,5-Trinitrobenzene (1,3,5-TNB)	1,3-Dinitrobenzene (1,3-DNB)	2,4,6-Trinitrotoluene
2,4,6-Trinitrotoluene (2,4,6-TNT)	2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)
2-Amino-4,6-dinitrotoluene (2-Am-DNT)	2-Nitrotoluene (2-NT)	3-Nitrotoluene (3-NT)
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	4-Nitrotoluene (4-NT)	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)

State of Illinois
Environmental Protection Agency

Certificate No.: 003174

Awards the Certificate of Approval

Environmental Chemistry Consulting Services, Inc.
2525 Advance Road
Madison, WI 53718

Hazardous and Solid Waste, Organic	*8330A*	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
Nitrobenzene	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	
Wastewater, Inorganic		
USEPA350.1R2.0,1993		
Ammonia		
USEPA353.2R2.0,1993		
Nitrate	Nitrate-nitrite (as N)	Nitrite (as N)

QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures

Pace Analytical Services – Green Bay, WI
1241 Bellevue Street Suite 9
Green Bay, WI 54302
920-469-2436

CORPORATE APPROVAL

Steve A. Vanderboom — 5-6-2013
Steve A. Vanderboom
President/CEO
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Minneapolis, MN 55414 (612) 607-1700
Date

5-6-2013
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Corporate Director of Quality
1800 Elm Street, Suite 200
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Date

LOCAL APPROVAL

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Nils Melberg, Laboratory General Manager
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Date

Kate E. Grams — 4/4/13
Kate Grams, Laboratory Quality Manager
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Date

4/30/13
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Date

04/18/2013
Chad Rusch, Laboratory Technical Director
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Date

4/8/13
Christopher Haase, Laboratory Technical Director
920-469-2436
Date

Effective Date is the date of the last signature.

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Any printed documents in use within a Pace Analytical Services, Inc. laboratory have been reviewed and approved by the persons listed on the cover page. They can only be deemed official if proper signatures are present.

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1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

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

1.1. Introduction to PASI

1.1.1. Pace Analytical Services, Inc. (PASI) is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. PASI offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, industrial hygiene testing, explosives, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. PASI has implemented a consistent Quality System in each of its laboratories and service centers. In addition, the company utilizes an advanced data management system that is highly efficient and allows for flexible data reporting. Together, these systems ensure data reliability and superior on-time performance. This document defines the Quality System and QA/QC protocols.

1.1.2. Our goal is to combine our expertise in laboratory operations with customized solutions to meet the specific needs of our customers.

1.2. Statement of Purpose

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

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. PASI management is committed to maintaining the highest possible standard of service for our customers by following a documented quality system. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the PASI network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work. This daily focus on quality is applied with initial project planning, continued through all field and laboratory activities, and is ultimately included in the final report generation.

1.3.3. PASI management demonstrates its commitment to quality by providing the resources, including facilities, equipment, and personnel to ensure the adherence to these documented policies and procedures and to promote the continuous improvement of the quality system. All PASI personnel must comply with all current applicable state, federal, and industry standards, and are required to perform all tests in accordance with stated methods and customer requirements.

1.4. Core Values

1.4.1. **Integrity-** Pace personnel are required to abide by the PASI Code of Ethics and all Pace employees must go through Data Integrity/Ethics training upon initial orientation and as an annual refresher.

1.4.2. **Value Employees-** Pace management views employees as our most important asset and communicates to them the relevance and importance of their activities within their job functions and how they contribute to the achievement of the objectives of the quality management system.

1.4.3. **Know Our Customers-** Pace makes every effort to know our customers and address their sampling and analytical needs. More information on this item can be found in section 2.0.

1.4.4. **Honor Commitments-** Pace labs focus on making solid commitments with regards to quality, capacity, and agreed upon turnaround time to our customers.

1.4.5. **Flexible Response To Demand-** Pace labs are equipped with both the material and personnel resources to enable them to be responsive to the demands of customers when situations or projects need change.

1.4.6. **Pursue Opportunities-** Pace is committed to pursuing opportunities for the growth of the company by constantly exploring markets and areas where we can expand.

1.4.7. **Continuously Improve-** Pace has committed much time and effort into establishing a continuous improvement program where company personnel meet on a regular basis to share ideas in cost reduction, production improvement and standardization in order to develop best practices. This information, as well as company financial and production metrics, are tracked, evaluated, and shared with each Pace facility.

1.5. Code of Ethics

1.5.1. PASI's fundamental ethical principles are as follows:

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

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

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

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

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

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

1.5.4. Any Pace employee can contact corporate management to report an ethical concern by calling the anonymous hotline at 612-607-6431.

1.6. Standards of Conduct

1.6.1. Data Integrity

1.6.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at PASI are the cornerstones of the company. Lack of data integrity is an assault on our most basic values putting PASI and its employees at grave financial and legal risk and will not be tolerated. Therefore, employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

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

1.6.2. Confidentiality

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

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

1.6.3. Conflict of Interest

1.6.3.1. PASI employees must avoid situations that might involve a conflict of interest or could appear questionable to others. The employee must be careful in two general areas:

1.6.3.1.1. Participation in activities that conflict or appear to conflict with the employees' PASI responsibilities.

1.6.3.1.2. Offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally. This includes bribes, gifts, kickbacks, or illegal payments.

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

1.6.4. Compliance

1.6.4.1. All employees are required to read, understand, and comply with the various components of the standards listed in this document. As confirmation that they understand their responsibility, each employee is required to sign an acknowledgment form annually that then becomes part of the employee's permanent record. Employees will be held accountable for complying with the Quality Systems as summarized in the Quality Assurance Manual.

1.7. Laboratory Organization

1.7.1. The PASI Corporate Office centralizes company-wide accounting, business development, financial management, human resources development, information systems, marketing, quality,

safety, and training activities. PASI's Director of Quality is responsible for assisting the development, implementation and monitoring of quality programs for the company. See Attachment IIB for the Corporate Organizational structure.

1.7.2. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office.

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

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

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

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

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

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

1.7.8. Appropriate support groups are present in each laboratory. The actual organizational structure for PASI – Green Bay is listed in Attachment IIA. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its accrediting authorities and revise the organizational chart in the Quality Assurance Manual (QAM) within 30 days. For changes in Department Managers or Supervisors or other laboratory personnel, no notifications will be sent to the laboratory's accrediting agencies; changes to the organizational chart will be updated during or prior to the annual review process. Changes or additions in these key personnel will also be noted by additional signatures on the QAM, as applicable. In any case, the QAM will remain in effect until the next scheduled revision.

1.8. Laboratory Job Descriptions

1.8.1. Senior General Manager

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

1.8.2. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.3. Quality Manager

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Quality. They may also report to a Senior Quality Manager within the same facility;
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors Quality Assurance/Quality Control activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Quality office). The Quality Manager is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;

- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews and maintains records of proficiency testing results;
- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors correctives actions;
- Maintains the currency of the Quality Manual.

1.8.4. Quality Analyst

- Assists the SQM/QM in the performance of quality department responsibilities as delegated by the SQM/QM;
- Assists in monitoring QA/QC data;
- Assists in internal audits;
- Assists in maintaining training records;
- Assists in maintaining the document control system;

1.8.5. Technical Director

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

1.8.6. Administrative Business Manager

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

- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.7. Client Services Manager

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

1.8.8. Project Manager

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

1.8.9. Project Coordinator

- Responsible for preparation of project specifications and provides technical/project support;
- Coordinates project needs with other department sections and assists with proposal preparation;
- Prepares routine proposals and invoicing;
- Responsible for scanning, copying, assembling and binding final reports;
- Other duties include filing, maintaining forms, process outgoing mail, maintaining training database and data entry.

1.8.10. Department Manager/Supervisor

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

1.8.11. Group Supervisor/Leader

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

1.8.12. Laboratory Analyst

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

1.8.13. Laboratory Technician

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

1.8.14. Sample Management Personnel

- Signs for incoming samples and verifies the data entered on the Chain of custody forms;

- Enters the sample information into the Laboratory Information Management System (LIMS) for tracking and reporting;
- Stages samples according to EPA requirements;
- Assists Project Managers and Coordinators in filling bottle orders and sample shipments.

1.8.15. Systems Administrator or Systems Manager

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

1.8.16. Safety/Chemical Hygiene Officer

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

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

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

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through a web-based Learning Management System. After a new employee has been instructed in matters of human resources, they are given instructional materials for the LMS and a password for access.

1.9.2. A new hire training checklist is provided to the new employee that lists training items for the employee to work through either independently on LMS or with their supervisor or trainer. The training items that can be completed independently include:

- Reading through applicable Standard Operating Procedures;
- Reviewing the Quality Manual and Chemical Hygiene Plan;
- Core training modules such as quality control indicators, basic laboratory skills, etc.;
- Quality Systems training including traceability of measurements, method calibration, calibration verification, accuracy, precision and uncertainty of measurements, corrective actions, documentation, and root cause analysis;

- Data Integrity/Ethics training.

1.9.3. The new employee's Department Supervisor provides the employee with a basic understanding of the role of the laboratory within the structure of PASI and the basic elements of that individual's position. Supervised training uses the following techniques:

- Hands-on training
- Training checklists/worksheets
- Lectures and training sessions
- Method-specific training
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Proficiency testing programs.
- On-line courses

1.9.4. Group Supervisors/Leaders are responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's training file indicates what procedures an analyst or a technician is capable of performing, either independently or with supervision. The files also include documentation of continuing capability, which are fully detailed in Section 3.4. Training documentation files for each person are maintained by the Quality Office either in hardcopy format or within the LMS.

1.9.5. All procedures and training records are maintained and available for review during laboratory audits. These procedures are reviewed/updated periodically by laboratory management. Additional information can be found in SOP S-ALL-Q-020 **Training and Employee Orientation** or its equivalent revision or replacement.

1.10. Data Integrity System

1.10.1. The data integrity system at PASI provides assurances to management that a highly ethical approach is being applied to all planning, training and implementation of methods. Data integrity is crucial to the success of our company and Pace Analytical is committed to creating and maintaining a culture of quality throughout the organization. To accomplish this goal, PASI has implemented a data integrity system that encompasses the following four requirements:

1.10.1.1. A data integrity training program: standardized training is given to each new employee and a yearly refresher is presented to all employees. Key topics addressed by this training include:

1.10.1.1.1. Need for honesty and transparency in analytical reporting
1.10.1.1.2. Process for reporting data integrity issues
1.10.1.1.3. Specific examples of unethical behavior and improper practices
1.10.1.1.4. Documentation of non-conforming data that is still useful to the data user
1.10.1.1.5. Consequences and punishments for unethical behavior
1.10.1.1.6. Examples of monitoring devices used by management to review data and systems

1.10.1.2. Signed data integrity documentation for all employees: this includes a written quiz following the Ethics training session and written agreement to abide by the Code of Ethics and Standards of Conduct explained in the employee manual.

1.10.1.3. In-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.10.1.4. Documentation of any review or investigation into possible data integrity infractions. This documentation, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.10.2. PASI management makes every effort to ensure that personnel are free from any undue pressures that affect the quality of their work including commercial, financial, over scheduling, and working condition pressures.

1.10.3. Corporate management also provides all PASI facilities a mechanism for confidential reporting of data integrity issues that ensures confidentiality and a receptive environment in which all employees are comfortable discussing items of ethical concern. The anonymous message line is monitored by the Corporate Director of Quality who will ensure that all concerns are evaluated and, where necessary, brought to the attention of executive management and investigated. Any Pace employee can contact corporate management to report an ethical concern by calling the anonymous hotline at 612-607-6431.

1.11. Laboratory Safety

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

1.12. Security and Confidentiality

1.12.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by PASI staff. Keyless door lock combinations and computer access codes/logins are changed periodically. Posted signs direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors, including PASI staff from other facilities, must sign the Visitor's Logbook maintained by the receptionist. A staff member will accompany them during the duration of their stay on the premises unless the SGM/GM/AGM/OM, SQM/QM, or Technical Director specify otherwise. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out. The last staff member to leave their department for the day must ensure that all outside access points to that area are secure.

1.12.2. Additional security is provided where necessary, (e.g., specific secure areas for sample, data, and customer report storage), as requested by customers, or cases where national security is of concern. These areas are lockable within the facilities, or are securely offsite. Access is limited to specific individuals or their designees. Security of sample storage areas is the responsibility of the Sample Custodian. Security of samples and data during analysis and data reduction is the responsibility of Group Supervisors. Security of customer report archives is the responsibility of the Client Services Manager. These secure areas are locked whenever these individuals or their designees are not present in the facility.

1.12.3. Access to designated laboratory sample storage locations is limited to authorized personnel only. Provisions for lock and key access are provided. No samples are to be removed without proper authorization. If requested by customer or contract, samples are not to be removed from secure storage areas without filling out an associated internal chain of custody.

1.12.4. Standard business practices of confidentiality are applied to all documents and information regarding customer analyses. Specific protocols for handling confidential documents are described in PASI SOPs. Additional protocols for sample identification by internal laboratory identification numbers only are implemented as required under contract specific Quality Assurance Project Plans (QAPPs).

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

1.13. Communications

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

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

2.0. SAMPLE CUSTODY

2.1. Sampling Support

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

2.2. Field Services

2.2.1. Pace Analytical has a large Field Services Division which is based in their Minneapolis facility as well as limited field service capabilities in some of our other facilities. Field Services provides comprehensive nationwide service offerings including:

- Stack Testing
- Ambient Air
- CEM Certification Testing
- Air Quality Monitoring
- Onsite Analytical Services- FTIR and GC
- Real-time Process Diagnostic/Optimization Testing
- Wastewater, Groundwater and Drinking Water Monitoring
- Storm Water and Surface Water Monitoring
- Soil and Waste Sampling
- Mobile Laboratory Services

2.2.2. Field Services operates under the PASI Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in Standard Operating Procedures and Procedure Manuals.

2.3. Project Initiation

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

2.3.2. The laboratory maintains records of all such reviews, including discussions with customers. Routine analytical project documentation of quotes, notes, dates, initials, and/or recordings is maintained in a project folder by project management. Conditions for new and more complex contracts are determined by the SGM/GM/AGM/OM and sales representatives. Quality Management is consulted on technical requirements and operations staff provides input on volume capacities. Evidence of these reviews is maintained in the form of awarded Request for Proposals (RFPs), signed quotes or contracts, and a Customer Relationship Management (CRM) database. If a review identifies a potential mismatch between customer requirements and laboratory capabilities and/or capacities, Pace will specify its level of commitment by listing these exceptions to the requirements within the RFP, quote or contract.

2.3.3. Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-GB-C-012 **Review of Analytical Requests** or its equivalent revision or replacement.

2.4. Chain of Custody

2.4.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis. PASI has implemented Standard Operating Procedures to ensure that sample custody traceability and responsibility objectives are achieved for every project.

2.4.2. Field personnel or client representatives must complete a chain of custody for all samples that are received by the laboratory. The importance of completeness of COCs is stressed to the samplers and is critical to efficient sample receipt and to insure the requested methods are used to analyze the correct samples.

2.4.3. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.4.4. The sampler is responsible for providing the following information on the chain of custody form:

- Customer project name
- Project location or number
- Field sample number/identification
- Date and time sampled
- Sample matrix
- Preservative
- Requested analyses
- Sampler signature
- Relinquishing signature
- Date and time relinquished
- Sampler remarks as needed
- Custody Seal Number if present
- Regulatory Program Designation
- The state where the samples were collected to ensure all applicable state requirements are met
- Turnaround time requested
- Purchase order number

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

2.4.6. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.5. Sample Acceptance Policy

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

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

2.5.3. All samples must:

- Have unique customer identification that is clearly marked on durable waterproof labels affixed to the sample containers that match the chain of custody.
- Have clear documentation on the chain of custody related to the location of the sampling site with the time and date of sample collection.
- Have the sampler's name and signature.
- Have all requested analyses clearly designated on the COC.
- Have clear documentation of any special analytical or data reporting requirements.
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be correctly preserved unless the method allows for laboratory preservation.
- Be received within holding time. Any samples with hold times that are exceeded will not be processed without prior customer approval.
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval.
- Be received within appropriate temperature ranges - not frozen but ≤6°C (See Note 1), unless program requirements or customer contractual obligations mandate otherwise (see Note 2). The cooler temperature is recorded directly on the COC and the SCUR. Samples that are delivered to the laboratory immediately after collection are considered acceptable if there is evidence that the chilling process has been started. For example, by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data associated with any deviations from the above sample acceptance policy requirements will be appropriately qualified.

Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be read and recorded to

±0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ±0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤6°C limit

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

Note 3: Biological Tissue Samples should be received frozen at ≤0°C or on ice ≤6°C.

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

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

2.5.5. Samples for drinking water analysis that are improperly preserved, or are received past holding time, are rejected at the time of receipt, with the exception of VOA samples that are tested for pH at the time of analysis.

2.5.6. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.6. Sample Log-in

2.6.1. After sample inspection, all sample information on the chain of custody is entered into the Laboratory Information Management System (LIMS). This permanent record documents receipt of all sample containers including:

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

2.6.2. All samples received are logged into the LIMS within one working day of receipt. Sample login may be delayed due to customer clarification of analysis needed, corrective actions for sample receipt non-conformance, or other unusual circumstances. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 08:00 as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

2.6.3. The Laboratory Information Management System (Epic Pro) automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of BB-XXXXX-YYY. The BB represents the laboratory identification within Pace's laboratory network. The 5 digit "X" number represents the project number followed by a 3 digit sample number. The project number is a sequential number that is assigned as a new project is created. The sample number corresponds to the number of samples submitted by the client. In addition to the unique sample ID, there is a sample container ID that consists of the sample number, the container type (ex. BP1U), and bottle 1 of Y, where Y represents the total number of containers of that particular type. Together the sample LIMs number and sample container ID number create a unique barcode encryption that can be linked to the sample analysis requested by the client. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the customer's field identification; it will be a permanent reference number for all future interactions.

2.6.4. Current division codes are noted below. These division codes are used primarily for accounting purposes and LIMS sample identifications. More division codes may be added without updating this document.

10 = Minnesota/Montana
92 = Asheville and Charlotte
60 = Kansas
50 = Indianapolis
12 = Virginia/Duluth MN
51 = Columbus
75 = Dallas
35 = Florida
20 = Gulf Coast
30 = Pittsburgh
40 = Green Bay
17 = Pace Life Sciences
65 = Schenectady, NY
36 = South Florida

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

2.6.6. Samples with hold times that are near expiration date/time may be sent directly to the laboratory for analysis at the discretion of the Project Manager and/or SGM/GM/AGM/OM.

2.6.7. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.7. Sample Storage

2.7.1. **Storage Conditions**

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

2.7.1.2. Storage blanks, consisting of two 40mL aliquots of reagent water, are stored with volatile samples and are used to measure cross-contamination acquired during storage. If applicable, laboratories must have documented procedures and criteria for evaluating storage blanks, appropriate to the types of samples being stored.

2.7.1.3. Additional information can be found in S-GB-Q-028 **Monitoring Temperature Controlled Units.**

2.7.2. **Temperature Monitoring**

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

2.7.2.2. The temperature of each refrigerated storage area is maintained at ≤6°C unless state or program requirements differ. The temperature of each freezer storage area is maintained at <-10°C unless state or program requirements differ. The temperature of each storage area is checked and documented each day of use (each calendar day). If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

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

Additional information can be found in S-GB-Q-028 **Monitoring Temperature Controlled Units.**

2.7.3. **Hazardous Materials**

2.7.3.1. Pure product or potentially heavily contaminated samples must be tagged as "hazardous" or "lab pack" and stored separately from other samples.

2.7.4. **Foreign/Quarantined Soils**

2.7.4.1. Depending on the soil disposal practices of the laboratory, foreign soils and soils from USDA regulated areas are adequately segregated to enable proper sample disposal. The USDA requires these samples to be incinerated or sterilized by an approved treatment procedure. Additional information regarding USDA regulations and sample handling can be found in applicable local laboratory SOPs.

2.7.4.2. Additional information on sample storage can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement and in S-GB-W-001 **Waste Handling and Management**.

2.8. Sample Protection

2.8.1. PASI laboratory facilities are operated under controlled access protocols to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted at all times.

2.8.2. Samples are removed from storage areas by designated personnel and returned to the storage areas, if necessary, immediately after the required sample quantity has been taken.

2.8.3. Upon customer request, additional and more rigorous chain of custody protocols for samples and data can be implemented. For example, some projects may require internal chain-of-custody protocols.

2.8.4. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.9. Subcontracting Analytical Services

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

2.9.2. Prior to subcontracting samples to a laboratory outside Pace Analytical, the potential sub-contract laboratory will be pre-qualified by verifying that the subcontractor meets the following criteria:

- All certifications required for the proposed subcontract are in effect,
- Sufficient professional liability and other required insurance coverage is in effect, and
- Is not involved in legal action by any federal, state, or local government agency for data integrity issues and has not been convicted in such investigation at any time during the past 5 years.

2.9.3. The contact and preliminary arrangements are made between the PASI Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract laboratory agreement include:

- Method of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Laboratory certification requirement
- Price per analysis
- Turn-around time requirements

2.9.4. Chain-of-custody forms are generated for samples requiring subcontracting to other laboratories. Sample receiving personnel re-package the samples for shipment, create a transfer chain of custody form and record the following information:

- Pace Analytical Laboratory Number
- Matrix
- Requested analysis
- Special instructions regarding turnaround, required detection or reporting limits, or any unusual information known about the samples or analytical procedure.
- Signature in "Relinquished By"

2.9.5. All subcontracted sample data reports are sent to the PASI Project Manager. Pace will provide a copy of the subcontractor's report to the client when requested.

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

2.9.7. Additional information can be found in S-GB-C-009 **Subcontracting Samples** or its equivalent revision or replacement.

2.10. Sample Retention and Disposal

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

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

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

2.10.4. Additional information can be found in S-GB-W-001 **Waste Handling and Management** and S-GB-C-010 **Sample Management** or their equivalent revisions or replacements.

3.0. ANALYTICAL CAPABILITIES

3.1. Analytical Method Sources

3.1.1. PASI laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. The latest valid editions of methodologies are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 is a representative listing of general analytical protocol references. PASI discloses in writing to its customers and regulatory agencies any instances in which modified methods are being used in the analysis of samples.

3.1.2. In the event of a customer-specific need, instrumentation constraint or regulatory requirement, PASI laboratories reserve the right to use valid versions of methods that may not be the most recent edition available.

3.2. Analytical Method Documentation

3.2.1. The primary form of PASI laboratory documentation of analytical methods is the Standard Operating Procedure (SOP). SOPs contain pertinent information as to what steps are required by an analyst to successfully perform a procedure. The required contents for the SOPs are specified in the company-wide SOP for Preparation of SOPs (S-ALL-Q-001).

3.2.2. The SOPs may be supplemented by other training materials that further detail how methods are specifically performed. This training material will undergo periodic, documented review along with the other Quality System documentation.

3.3. Analytical Method Validation

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

3.4. Demonstration of Capability (DOC)

3.4.1. Analysts complete an initial demonstration of capability (IDOC) study prior to performing a method or when there is a change in instrument type, personnel, or test method, or at any time that a method has not been performed by the laboratory or analyst in a 12-month period. The mean recovery and standard deviation of each analyte, taken from 4 replicates of a quality control standard is calculated and compared to method criteria (if available) or established laboratory criteria for evaluation of acceptance. Each laboratory maintains copies of all demonstrations of capability, including those that fail acceptance criteria and corresponding raw data for future reference and must

document the acceptance criteria prior to the analysis of the DOC. Demonstrations of capability are verified on an annual basis.

3.4.2. For Continuing Demonstrations of Capability, the laboratories may use Performance Testing (PT) samples in lieu of the 4-replicate approach listed above. For methods or procedures that do not lend themselves to the "4-replicate" approach, the demonstration of capability requirements will be specified in the applicable SOP. Drinking Water DOCs must be done at or below the MCL.

3.4.3. Additional information can be found in SOP S-ALL-Q-020 **Training and Employee Orientation** or its equivalent revision or replacement.

3.5. Regulatory and Method Compliance

3.5.1. PASI understands that expectations of our customers commonly include the assumption that laboratory data will satisfy specific regulatory requirements. Therefore PASI attempts to ascertain, prior to beginning a project, what applicable regulatory jurisdiction, agency, or protocols apply to that project. This information is also required on the chain of custody submitted with samples.

3.5.2. PASI makes every effort to detect regulatory or project plan inconsistencies, based upon information from the customer, and communicate them immediately to the customer in order to aid in the decision making process. PASI will not be liable if the customer chooses not to follow PASI recommendations.

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

4.0. QUALITY CONTROL PROCEDURES

Quality control data is analyzed and where they are found to be outside pre-defined criteria, planned action is taken to correct the problem in order to prevent incorrect results from being reported. Quality control samples are to be processed in the same manner as client samples.

4.1. Method Blank

4.1.1. A method blank is used to evaluate contamination in the preparation/analysis system and is processed through all preparation and analytical steps with its associated samples.

4.1.2. A method blank is processed at a minimum frequency of one per preparation batch In the case of a method that has no separate preparation step, a method blank is processed with no more than 20 samples of a specific matrix performed by the same analyst, using the same method, standards, and reagents.

4.1.3. The method blank consists of a matrix similar to the associated samples that is known to be free of analytes of interest. Method blanks are not applicable for certain analyses, such as pH, conductivity, flash point and temperature

4.1.4. Method blanks are not applicable for certain analyses, such as pH, conductivity, flash point and temperature.

4.1.5. Each method blank is evaluated for contamination. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the reporting limit and is greater than 1/10 of the amount of that analyte found in any associated sample. Some labs, due to client requirements, etc., may have to evaluate their method blanks down to ½ the reporting limit as opposed to the reporting limit itself. Corrective actions for blank contamination may include the re-preparation and re-analysis of all samples (where possible) and quality control samples. Data qualifiers must be applied to results that are considered affected by contamination in a method blank.

4.1.6. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.2. Laboratory Control Sample

4.2.1. The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis.

4.2.2. An LCS is processed at a minimum frequency of one per preparation batch. In the case of a method that has no separate preparation step, an LCS will be processed with no more than 20 samples of a specific matrix performed by the same analyst, using the same method, standards, and reagents.

4.2.3. The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes.

4.2.4. The LCS contains **all** analytes specified by a specific method or by the customer or regulatory agency, which may include full list of target compounds, with certain exceptions. These exceptions may include analyzing only specific Aroclors when PCB analysis is requested or not spiking with all EPA Appendix IX compounds when a full Appendix IX list of compounds is requested. However,

the lab must ensure that all target components in its scope of accreditation are included in the spike mixture for the LCS over a two (2) year period. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

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

4.2.5. The LCS is evaluated against the method default or laboratory-derived acceptance criteria. For those methods that require laboratory-derived limits, method default control limits may be used until the laboratory has a minimum of 20, but preferably greater than 30, data points from which to derive internal acceptance criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any associated sample containing an 'out-of-control' compound must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier. When the acceptance criteria for the LCS are exceeded high, and there are associated samples that are non-detects, then those non-detects can be reported with data qualifiers, or when the acceptance criteria are exceeded low, those associated sample results may be reported if they exceed the maximum regulatory limit/decision level with data qualifiers.

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

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

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

4.2.8. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

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

4.3.1. A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch unless the MS is actually used as the LCS.

4.3.2. A **Matrix Spike/Matrix Spike Duplicate** (MS/MSD) set is processed at a frequency specified in a particular method or as determined by a specific customer request. This frequency will be specified in the applicable method SOP or customer QAPP. In the absence of such requirements, an MS/MSD set is routinely analyzed once per every 20 samples per matrix per method.

4.3.3. The MS and MSD consist of the sample matrix that is then spiked with known concentrations of target analytes. Laboratory personnel spike customer samples that are specifically designated as MS/MSD samples or, when no designated samples are present in a batch, randomly select samples to spike that have adequate sample volume or weight. Spiked samples are prepared and analyzed in the same manner as the original samples and are selected from different customers if possible.

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

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

4.3.6. A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method.

4.3.7. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.4. Sample Duplicate

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

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

4.4.3. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.5. Surrogates

4.5.1. Surrogates are compounds that reflect the chemistry of target analytes and are typically added to samples for organic analyses to monitor the effect of the sample matrix on compound recovery.

4.5.2. Surrogates are added to each customer sample (for organics), method blank, LCS, MS, and calibration standard prior to extraction or analysis. The surrogates are evaluated against the method or laboratory derived acceptance criteria or against project-specific acceptance criteria specified by the client, if applicable. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error. An exception to this would be samples that have high surrogate values but no reportable hits for target compounds. These samples would be reported, with a qualifier, because the implied high bias would not affect the final results. For methods with multiple surrogates, documentation regarding acceptance and associated compounds will be found in the individual method SOPs.

4.5.3. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.6. Internal Standards

4.6.1. Internal Standards are method-specific analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, sample, and calibration standard at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. At a minimum, the laboratory will follow method specific guidelines for the treatment of internal standard recoveries as they are related to the reporting of data.

4.6.2. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.7. Field Blanks

4.7.1. Field blanks are blanks prepared at the sampling site in order to monitor for contamination that may be present in the environment where samples are collected. These field quality control samples are often referenced as field blanks, rinsate blanks, or equipment blanks. The laboratory analyzes these field blanks as normal samples and informs the customer if there are any target compounds detected above the reporting limits.

4.8. Trip Blanks

4.8.1. Trip blanks are blanks that originate from the laboratory as part of the sampling event and are used to monitor for contamination of samples during transport. These blanks accompany the empty sample containers to the field and then accompany the collected samples back to the laboratory. These blanks are routinely analyzed for volatile methods where ambient background contamination is likely to occur.

4.9. Limit of Detection (LOD)

4.9.1. PASI laboratories are required to use a documented procedure to determine a limit of detection for each analyte of concern in each matrix reported. All sample processing steps of the preparation and analytical methods are included in this determination including any clean ups. For any test that does not have a valid LOD, sample results below the limit of quantitation (LOQ) cannot be reported.

4.9.2. The LOD is initially established for the compounds of interest for each method in a clean matrix with no target analytes present and no interferences at a concentration that would impact the results. The LOD is then determined every time there is a change in the test method that affects how the test is performed or when there has been a change in the instrument that affects the sensitivity. If required by customer, method or accreditation body, the LOD will be re-established annually for all applicable methods.

4.9.3. Unless otherwise noted, the method used by PASI laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. Where required by regulatory program or customer, the above referenced procedure will be followed.

4.9.4. Where specifically stated in the published method, LODs or MDLs will be performed at the listed frequency.

4.9.5. The validity of the LOD must be shown by detection (a value above zero) of the analytes in a QC sample in each quality system matrix. The QC sample must contain the analyte at no more than 3X the LOD for a single analyte test and 4X the LOD for multiple analyte tests. This verification must be performed on each instrument used for sample analysis and reporting of data. The validity of the LOD must be verified as part of the LOD determination process. This verification must be done prior to the use of the LOD for sample analysis.

4.9.6. An LOD study is not required for any analyte for which spiking solutions or quality control samples are not available such as temperature.

4.9.7. The LOD, if required, shall be verified annually for each quality system matrix, technology and analyte. In lieu of performing full LOD (MDL) studies annually, the laboratory can verify the LOD (MDL) on an annual basis, providing this verification is fully documented and does not contradict other customer or program requirements that the laboratory must follow. The requirements of this verification are:

- The spike concentration of the verification must be no more than 3X times the LOD for single analyte tests and 4X the LOD for multiple analyte tests.
- The laboratory must verify the LOD on each instrument used for the reporting of sample data.
- The laboratory must be able to identify all target analytes in the verification standard (distinguishable from noise).

4.9.8. Additional information can be found in SOP S-GB-Q-020 **Determination of LOD and LOQ** or its equivalent revision or replacement.

4.10. Limit of Quantitation (LOQ)

4.10.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For PASI laboratories, this LOQ is referred to as the RL, or Reporting Limit. This RL is based on the lowest

calibration standard concentration that is used in each initial calibration. Results below this level are not allowed to be reported without qualification since the results would not be substantiated by a calibration standard. For methods with a determined LOD, results can be reported out below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

4.10.2. The LOQ must be higher than the LOD.

4.10.3. To verify the LOQ, the laboratory will prepare a sample in the same matrix used for the LCS. The sample will be spiked with each target analyte at a concentration equivalent to the RL or 2X the RL. This sample must undergo the routine sample preparation procedure including any routine sample cleanup steps. The sample is then analyzed and the recovery of each target analyte determined. The recovery for each target analyte must meet the laboratories current control limits for an LCS. The annual LOQ verification is not required if the LOD was determined or verified annually on that instrument.

4.10.4. Additional information can be found in SOP S-GB-Q-020 **Determination of LOD and LOQ** or its equivalent revision or replacement.

4.11. Estimate of Analytical Uncertainty

4.11.1. PASI laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, PASI laboratories base this estimation on the recovery data obtained from the Laboratory Control Spikes. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-GB-Q-010 **Estimation of Measurement Uncertainty** or its equivalent revision or replacement.

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

4.12. Proficiency Testing (PT) Studies

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

4.12.2. The laboratory initiates an investigation whenever PT results are deemed 'unacceptable' by the PT provider. All findings and corrective actions taken are reported to the SQM/QM or their designee. A corrective action plan is initiated and this report is sent to the appropriate state accreditation agencies for their review. Additional PTs will be analyzed and reported as needed for certification purposes.

4.12.3. PT samples are treated as typical customer samples, utilizing the same staff, methods, equipment, facilities, and frequency of analysis. PT samples are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

4.12.4. Comparison of analytical results with anyone participating in the same PT study is prohibited prior to the close of the study.

4.12.5. Additional information can be found in SOP S-GB-Q-021 **Proficiency Testing Program** or its equivalent revision or replacement.

4.13. Rounding and Significant Figures

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

4.13.2. Data is compared to the reporting limits and MDLs to determine if qualifiers are needed before the rounding step occurs.

4.13.3. **Rounding:** PASI-Green Bay follows the odd / even guidelines for rounding numbers:

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

4.13.4. **Significant Digits**

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

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

4.14. Retention Time Windows

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

4.14.2. One process for the production of retention time windows: Make 3 injections of all single component or multi-component analytes over a 72-hour period. Record the retention time in minutes for each analyte and surrogate to 3 decimal places. Calculate the mean and standard deviation of the three absolute retention times for each target analyte and surrogate. For multi-component analytes, choose 3-5 major peaks and calculate the mean and standard deviation for each of the peaks. If the standard deviation of the retention times of a target analyte is 0.000, the lab may use a default standard deviation of 0.01. The width of the retention time window for each analyte and surrogate and major peak in a multi-component analyte is defined as +/- 3 times the standard deviation of the mean absolute retention time established during that 72-hour period or 0.03 minutes, whichever is greater.

4.14.3. The center of the retention time window is established for each analyte and surrogate by using the absolute retention times from the CCV at the beginning of the analytical shift. For samples run with an initial calibration, use the retention time of the mid-point standard of the initial calibration curve.

4.14.4. For more information, please reference the local facility's analytical SOPs.

5.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

5.1. Document Management

5.1.1. Additional information can be found in SOP S-GB-Q-029 **Document Control and Management** or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

5.1.2. Pace Analytical Services, Inc. has an established procedure for managing documents that are part of the quality system. The list of managed documents includes, but is not limited to, Standard Operating Procedures (both technical and non-technical), Quality Assurance Manuals, quality policy statements, training documents, work-processing documents, charts, posters, memoranda, notices, forms, software, and any other procedures, tables, plans, etc. that have a direct bearing on the quality system (including applicable data records and non-technical documents).

5.1.3. A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. This establishes that there are no invalid or obsolete documents in use in the facility. All documents are reviewed periodically and revised if necessary. Obsolete documents are systematically discarded or archived for audit or knowledge preservation purposes.

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

5.1.5. SOPs, specifically, are available to all laboratory staff via the Learning Management System (LMS) which is a secure repository that is accessed through an internet portal. As a local alternative to the hard copy system of controlled documents, secured electronic copies of controlled documents may be maintained on the laboratory's local server. These document files must be read-only for all personnel except the Quality Department and system administrator. Other requirements for this system are as follows:

- Electronic documents must be readily accessible to all facility employees.
- Electronic documents (i.e. pdf's) must be locked from printing. All hardcopy SOPs must be obtained from the Quality Department.

5.1.6. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for PASI. The base QAM template is distributed by the Corporate Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template and submit those modifications to the Corporate Director of Quality for review. Once approved and signed by both the CEO and the Director of Quality; the SGM/GM/AGM/OM, the SQM/QM, and any Technical Directors sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis by all of the PASI SQMs/QMs and revised accordingly by the Director of Quality.

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

5.1.7.1. SOPs fall into two categories: company-wide documents and facility specific documents. Company-wide SOPs start with the prefix S-ALL- and local SOPs start with the individual facility prefix.

5.1.7.2. The purpose of the company-wide SOPs is to establish policies and procedure that are common and applicable to all PASI facilities. Company-wide SOPs are document-controlled by the corporate quality office and signed copies are distributed to all of the SQMs/QMs. The local management personnel sign the company-wide SOPs. The SQM/QM is then in charge of distribution to employees, external customers, or regulatory agencies and maintaining a distribution list of controlled document copies.

5.1.7.3. Local PASI facilities are responsible for developing facility-specific SOPs applicable to their respective facility. The local facility develops these facility-specific SOPs based on the corporate-wide SOP template. This template is written to incorporate a set of minimum method requirements and PASI best practice requirements. The local facilities may add to or modify the corporate-wide SOP template provided there are no contradictions to the minimum method or best practice requirements. Facility-specific SOPs are controlled by the applicable SQM/QM according to the corporate document management policies.

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

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

5.1.7.6. Additional information can be found in SOP S-GB-Q-017 **Preparation of SOPs** or its equivalent revision or replacement.

5.1.8. **Other Documentation**

5.1.8.1. Insert additional procedures and protocols relevant for any other controlled documents employed within the laboratory.

5.2. Document Change Control

5.2.1. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After revisions are approved, a revision number is assigned and the previous version of the document is officially retired. Copies may be kept for audit or knowledge preservation purposes.

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

5.3. Management of Change

5.3.1. The process for documenting necessary changes within the laboratory network are not typically handled using the corrective or preventive action system as outlined in section 9.0. Management of Change is a proactive approach to dealing with change to minimize the potential negative impact of systematic change in the laboratory and to ensure that each change has a positive desired outcome. This process will primarily be used for the implementation of large scale projects and information system changes as a means to apply consistent systems or procedures within the laboratory network. The request for change is submitted by the initiator and subsequently assigned to an individual or team for development and planning. The final completion of the process culminates in final approval and verification that the procedure was effectively implemented. Additional information can be found in SOP S-GB-Q-016 **Management of Change** or its equivalent revision or replacement.

6.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

Each PASI facility is equipped with sufficient instrumentation and support equipment to perform the relevant analytical testing or field procedures performed by each facility. Support equipment includes chemical standards, thermometers, balances, disposable and mechanical pipettes, etc. This section details some of the procedures necessary to maintain traceability and to perform proper calibration of instrumentation and support equipment. See Attachment III for a list of equipment currently used at the Green Bay PASI facility.

6.1. Standards and Traceability

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

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

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

6.1.4. All prepared standard or reagent containers include the PASI identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials. This ensures traceability back to the standard preparation logbook.

6.1.5. For containers that are too small to accommodate labels that list all of the above information associated with a standard, the minimum required information will be PASI standard ID, concentration, and expiration date. This assures that no standard will be used past its assigned expiration date.

6.1.6. If a second source standard is required to verify an existing calibration or spiking standard, this standard must be obtained from a different manufacturer or from a different lot unless client specific QAPP requirements state otherwise.

6.1.7. Additional information concerning standards and reagent traceability can be found in the SOP S-GB-Q-026 **Standard and Reagent Management and Traceability** or its equivalent revision or replacement.

6.2. General Analytical Instrument Calibration Procedures (Organic and Inorganic)

6.2.1. All support equipment and instrumentation are calibrated or checked before use to ensure proper functioning and verify that the laboratory's requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards

traceable to recognized national standards or reference standards whose values have been statistically validated.

6.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in the narrative. Any specific method requirement for number and type of calibration standards supersedes the general requirement. Instrument and method specific calibration criteria are explained within the specific analytical standard operating procedures for each facility.

6.2.3. Results from all calibration standards analyzed must be included in constructing the calibration curve with the following exceptions:

6.2.3.1. The lowest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The reporting limit must be adjusted to the lowest concentration included in the calibration curve;

6.2.3.2. The highest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done an individual analyte basis. The upper limit of quantitation must be adjusted to the highest concentration included in the calibration curve;

6.2.3.3. Multiple points from either the high end or the low end of the calibration curve may be excluded as long as the remaining points are contiguous in nature and the minimum number of levels remains as established by method or standard operating procedure. The reporting limit or quantitation range, whichever is appropriate, must be adjusted accordingly;

6.2.3.4. Results from a concentration level between the lowest and highest calibration levels can only be excluded from an initial calibration curve for a documentable and acceptable cause with approval from the responsible department supervisor and the local SQM/QM or their designee. An acceptable cause is defined as an obvious sample introduction issue that resulted in no response or a documented response that is less than the lowest standard used in the ICAL. A suspected incorrectly prepared standard is not considered to be an acceptable cause. The results for all analytes are to be excluded and the point must be replaced by re-analysis. Re-analysis of this interior standard must occur within the same 12-hour tune time period for GC/MS methodologies and within 8 hours of the initial analysis of that standard for non-GC/MS methodologies. All samples analyzed prior to the re-analyzed calibration curve point must be re-analyzed after the calibration curve is completed and re-processed against the final calibration curve.

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

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

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

6.2.7. **General Organic Calibration Procedures**

6.2.7.1. Calibration standards are prepared at a minimum of five concentrations for organic analyses (unless otherwise stipulated in the method).

6.2.7.2. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Curves that do not meet the appropriate criteria require corrective action that may include re-running the initial calibration curve. Rounding to meet initial calibration criteria is not allowed, that is, 15.3 cannot be rounded down to meet a $\leq$ 15% RSD requirement. This also applies to linear and non-linear fit requirements. All initial calibrations are verified with an initial calibration verification standard (ICV) obtained from a second manufacturer or second lot from the same manufacturer if that lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

6.2.7.3. The calibration curve is periodically verified by the analysis of a mid-level continuing calibration verification (CCV) standard during the course of sample analysis. This standard is from the same source as the initial calibration unless otherwise specified in the source method to be from an alternate source material. Rounding to meet continuing calibration criteria is not allowed. Continuing calibration verification is performed at the beginning and end of each analytical batch except if an internal standard is used, then only one verification at the beginning of the batch is needed, whenever it is expected that the analytical system may be out of calibration, if the time period for calibration has expired, or for analytical systems that have specific calibration verification requirements. This verification standard must meet acceptance criteria in order for sample analysis to proceed.

6.2.7.4. In the event that the CCV does not meet the acceptance criteria, a second CCV may be injected as part of the diagnostic evaluation and corrective action investigation. If the second CCV is acceptable, the analytical sequence may be continued. If both CCVs fail, the analytical sequence is terminated and corrective action is initiated. Sample analysis cannot begin until after documented corrective action has been completed and either two consecutive passing CCVs have been analyzed or the instrument has successfully passed a new initial calibration. All samples analyzed since the last compliant CCV are re-analyzed for methodologies utilizing external calibration.

6.2.7.5. When instruments are operating unattended, autosamplers may be programmed to inject consecutive CCVs as a preventative measure against CCV failure with no corrective action. In this case, both CCVs must be evaluated to determine potential impact to the results. A summary of the decision tree and necessary documentation are listed below:

- If both CCVs meet the acceptance criteria, the analytical sequence is allowed to continue without corrective action. The 12 hour clock begins with the injection of the second CCV.
- If the first CCV does not meet the acceptance criteria and the second CCV is acceptable, the analytical sequence is continued and the results are reported.
- If the first CCV meets the acceptance criteria and the second CCV is out of control, the samples after the out of control CCV must be re-analyzed in a compliant analytical sequence.
- If both CCVs are out of control, all samples since the last acceptable CCV must be re-analyzed in a compliant analytical sequence.

6.2.7.6. Some analytical methods require that samples be bracketed by passing CCVs analyzed both before and after the samples. This is specific to each method but, as a general rule, all external calibration methods require bracketing CCVs. Most internal standard calibrations do not require bracketing CCVs.

6.2.7.7. Some analytical methods require verification based on a time interval; some methods require a frequency based on an injection interval. The type and frequency of the calibration verifications is dependent on both the analytical method and possibly on the quality program associated with the samples. The type and frequency of calibration verification will be documented in the method specific SOP employed by each laboratory.

6.2.8. General Inorganic Calibration Procedures

6.2.8.1. The instrument is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. A calibration blank is also included. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Rounding to meet initial calibration criteria is not allowed. This also applies to linear and non-linear fit requirements. The number of calibration standards used depends on the specific method criteria or customer project requirements, although normally a minimum of three standards is used.

6.2.8.2. The ICP and ICP/MS can be standardized with a zero point and a single point calibration if:

- Prior to analysis, the zero point and the single point calibration are analyzed and a linear range has been established,
- Zero point and single point calibration standards are analyzed with each batch
- A standard corresponding to the LOQ is analyzed with the batch and meets the established acceptance criteria
- The linearity is verified at the frequency established by the method or manufacturer.

6.2.8.3. All initial calibrations are verified with an initial calibration verification standard (ICV) obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

6.2.8.4. During the course of analysis, the calibration curve is periodically verified by the analysis of calibration verification standards (CCV). A calibration verification standard is analyzed within each analytical batch at method/program specific intervals to verify that the initial calibration is still valid. The CCV is also analyzed at the end of the analytical batch.

6.2.8.5. A calibration blank is also run with each calibration verification standard to verify the cleanliness of the system. All reported results must be bracketed by acceptable CCVs. Instrument and method specific calibration acceptance criteria are explained within the specific analytical standard operating procedures for each facility.

6.2.8.6. Interference check standards are also analyzed per method requirements and must meet acceptance criteria for metals analyses.

6.3. Support Equipment Calibration Procedures

6.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until repaired. The laboratory maintains records to demonstrate the correction factors applied to working thermometers.

6.3.2. On each day the equipment is used, balances, ovens, refrigerators (those used to keep samples and standards at required temperatures), freezers, and water baths are checked in the expected use range with NIST traceable references in order to ensure the equipment meets laboratory specifications and these checks are documented appropriately.

6.3.3. **Analytical Balances**

6.3.3.1. Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Calibration weights are ASTM Class 1 or other class weights that have been calibrated against a NIST standard weight and are re-certified every 5 years at a minimum against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the Quality department.

6.3.4. **Thermometers**

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

6.3.4.2. Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures. Each thermometer is individually numbered and assigned a correction factor based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

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

6.3.5. **pH/Electrometers**

6.3.5.1. The meter is calibrated before use each day, using fresh buffer solutions. Additional information regarding pH/Electrometers can be found in SOP S-GB-I-071 *Measurement of pH in Water, Soil and Waste* or its equivalent revision or replacement.

6.3.6. **Spectrophotometers**

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

6.3.7. **Mechanical Volumetric Dispensing Devices**

6.3.7.1. Mechanical volumetric dispensing devices including bottle top dispensers (those that are critical in measuring a required amount of reagent), pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis. Glass microliter syringes are checked for accuracy prior to initial use.

6.3.7.2. Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-GB-Q-030 **Support Equipment** or its equivalent revision or replacement.

6.4. Instrument/Equipment Maintenance

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

6.4.2. The Operations Manager and/or department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

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

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

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

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

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

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

6.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily.

7.0. CONTROL OF DATA

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

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

7.1. Analytical Results Processing

7.1.1. When analytical, field, or product testing data is generated, it is either recorded in a bound laboratory logbook (e.g., Run log or Instrument log) or copies of computer-generated printouts that are appropriately labeled and filed. These logbooks and other laboratory records are kept in accordance with each facility's Standard Operating Procedure for documentation storage and archival. If the laboratory chooses to minimize or eliminate its paper usage, these records can be kept on electronic media. In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues.

7.1.2. The primary analyst is responsible for initial data reduction and review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets and LIMS fields.

7.1.3. The primary analyst then compiles the initial data package for verification. This compilation must include sufficient documentation for data review. It may include standard calibrations, chromatograms, manual integration documentation, electronic printouts, chain of custody forms, and logbook copies.

7.1.4. Some agencies or customers require different levels of data reporting. For these special levels, the primary analyst may need to compile additional project information, such as initial calibration data or extensive spectral data, before the data package proceeds to the verification step.

7.2. Data Verification

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

7.2.2. Analysts performing the analysis and subsequent data reduction have primary responsibility for quality of the data produced. The primary analyst initiates the data verification process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. Data review checklists, either hardcopy or electronic, are used to document the data review process. The primary analyst is responsible for the initial input of the data into the LIMS. The primary analyst and reviewer must be clearly identified on all applicable data review checklists.

7.2.3. The completed data package is then sent to a designated qualified reviewer (this cannot be the primary analyst). The following criteria have been established to qualify someone as a data reviewer. To perform secondary data review, the reviewer must:

7.2.3.1. Have a current Demonstration of Capability (DOC) study on file and have an SOP acknowledgement form on file for the method/procedure being reviewed; or, See Note

7.2.3.2. Have a DOC on file for a similar method/technology (i.e., GC/MS) and have an SOP acknowledgment form on file for the method/procedure being reviewed; or, See Note

7.2.3.3. Supervise or manage a Department and have an SOP acknowledgment form on file for the method/procedure being reviewed; or,

7.2.3.4. Have significant background in the department/methods being reviewed through education or experience and have an SOP acknowledgment form on file for the method/procedure being reviewed.

7.2.4. **Note:** Secondary reviewer status must be approved personally by the SQM/QM or SGM/GM/AGM/OM in the event that this person has no prior experience on the specific method or general technology.

7.2.5. This reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer validates the data entered into the LIMS and documents approval of manual integrations.

7.2.6. Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data (or designating the review of data electronically). The Operations or Project Manager examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution.

7.2.7. Additional information regarding data review procedures can be found in SOP S-ALL-Q-037 **Data Review** or its equivalent revision or replacement, as well as in SOP S-GB-Q-024 **Manual Integration** or its equivalent revision or replacement.

7.2.8. The Data Checker program will process validated data for a given project against a set of pre-determined requirements and known chemistry relationships. The program creates a report that includes a series of warnings and errors for any requirement or condition determined to be suspect or incorrect. These warnings and error counts are displayed on the "Project Inquiry by Client" screen and on the final Data Checker reports. For projects that have any number of warnings or errors, the Data Checker report will provide a message that identifies the source and condition of the error or warning.

7.2.9. Some reports and/or data packages may be reviewed by the QM or SQM or designee based on program requirements (e.g., DoD) or client requirements. In this case a thorough review for completeness and accuracy may include a compilation of raw data and QC summaries in addition to the final report to produce a full deliverable package. See SOP S-GB-Q-022 (or equivalent replacement), **Audits and Inspections**, for full Quality department final report and raw data review requirements.

7.3. Data Reporting

7.3.1. Data for each analytical fraction pertaining to a particular PASI project number are delivered to the Project Manager for assembly into the final report. All points mentioned during technical and QC reviews are included in a case narrative if there is potential for data to be impacted.

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

7.3.2.1. A title which designates the report as “Final Report”, “Laboratory Results”, “Certificate of Results”, etc.;

7.3.2.2. Name and address of laboratory (or subcontracted laboratories, if used);

7.3.2.3. Phone number and name of laboratory contact to where questions can be referred;

7.3.2.4. A unique identification number for the report. The pages of the report shall be numbered and a total number of pages shall be indicated;

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

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

7.3.2.7. Identification of any sample that did not meet acceptable sampling requirements of the relevant governing agency, such as improper sample containers, holding times missed, sample temperature, etc.;

7.3.2.8. Date and time of collection of samples, date of sample receipt by the laboratory, dates of sample preparation and analysis, and times of sample preparation and analysis when the holding time for either is 72 hours or less;

7.3.2.9. Identification of the test methods used;

7.3.2.10. Identification of sampling procedures if sampling was conducted by the laboratory;

7.3.2.11. Deviations from, additions to, or exclusions from the test methods. These can include failed quality control parameters, deviations caused by the matrix of the sample, etc., and can be shown as a case narrative or as defined footnotes to the analytical data;

7.3.2.12. Identification of whether calculations were performed on a dry or wet-weight basis;

7.3.2.13. Reporting limits used;

7.3.2.14. Final results or measurements, supported by appropriate chromatograms, charts, tables, spectra, etc.;

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

7.3.2.16. Date report was issued;

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

7.3.2.18. If necessary, a statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory;

7.3.2.19. Identification of all test results provided by a subcontracted laboratory or other outside source;

7.3.2.20. Identification of results obtained outside of quantitation levels.

In addition to the requirements listed above, final reports shall also contain the following items when necessary for the interpretation of results:

7.3.2.21. Deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions;

7.3.2.22. Where relevant, a statement of compliance/non-compliance with requirements and/or specifications (e.g., the TNI standard);

7.3.2.23. Where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit;

7.3.2.24. Where appropriate and needed, opinions and interpretations, which may include opinions on the compliance/non-compliance of the results with requirements, fulfillment of contractual requirements, recommendations on how to use the results, and guidance to be used for improvement;

7.3.3. Additional items may be required per Client QAPPs or different state regulations.

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

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

7.3.6. The following positions are the only approved signatories for PASI final reports:

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

7.4. Data Security

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

7.5. Data Archiving

7.5.1. All records compiled by PASI are maintained legible and retrievable and stored secured in a suitable environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. These records may include, but are not limited to, customer data reports, calibration and maintenance of equipment, raw data from instrumentation, quality control documents, observations, calculations, and logbooks. These records are retained in order to provide for possible historical reconstruction including sampling, receipt, preparation, analysis, and personnel involved. TNI-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the SQM/QM or a designated Data Archivist.

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

7.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained by the acquiring entity for a minimum of five years. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

7.6. Data Disposal

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

8.0. QUALITY SYSTEM AUDITS AND REVIEWS

8.1. Internal Audits

8.1.1. Responsibilities

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

8.1.2. Scope and Frequency of Internal Audits

8.1.2.1. The complete internal audit process consists of the following four sections:

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

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

8.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible. Examples of system-wide elements that can be audited include:

- Quality Systems documents, such as Standard Operating Procedures, training documents, Quality Assurance Manual, and all applicable addenda
- Data records and non-technical documents
- Personnel and training files.
- General laboratory safety protocols.
- Chemical handling practices, such as labeling of reagents, solutions, and standards as well as all associated documentation.
- Documentation concerning equipment and instrumentation, calibration/maintenance records, operating manuals.
- Sample receipt and management practices.
- Analytical documentation, including any discrepancies and corrective actions.

- General procedures for data security, review, documentation, reporting, and archiving.
- Data integrity issues such as proper manual integrations.

8.1.2.4. When the operations of a specific department are evaluated, a number of additional functions are reviewed including:

- Detection limit studies
- Internal chain of custody documentation
- Documentation of standard preparations
- Quality Control limits and Control charts

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

8.1.2.6. A representative number of data audits are completed annually. Findings from these data audits are handled in the same manner as those from other internal and external audits.

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

8.1.3. Internal Audit Reports and Corrective Action Plans

8.1.3.1. Additional information can be found in SOP S-GB-Q-022 **Internal and External Audits** or its equivalent revision or replacement.

8.1.3.2. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the SQM/QM writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

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

8.1.3.4. Once completed, the internal audit report is issued jointly to the SGM/GM/AGM/OM and the manager(s)/supervisor(s) of the audited operation at a minimum. The responsible manager(s)/supervisor(s) responds within 14 days with a proposed plan to correct all of the deficiencies cited in the audit report. The SQM/QM may grant additional time for responses to large or complex deficiencies (not to exceed 30 days). Each response must include timetables for completion of all proposed corrective actions.

8.1.3.5. The SQM/QM reviews the audit responses. If the response is accepted, the SQM/QM uses the action plan and timetable as a guideline for verifying completion of the corrective action(s). If the SQM/QM determines that the audit response does not adequately address the correction of cited deficiencies, the response will be returned for modification.

8.1.3.6. To complete the audit process, the SQM/QM performs a re-examination of the areas where deficiencies were found to verify that all proposed corrective actions have been implemented. An audit deficiency is considered closed once implementation of the necessary corrective action has been audited and verified. This is usually within 60-90 days after implementation. If corrective action cannot be verified, the associated deficiency remains open until that action is completed.

8.2. External Audits

8.2.1. PASI laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

8.2.2. Audit teams external to the company review the laboratory to assess the effectiveness of systems and degree of technical expertise. The SQM/QM and other QA staff host the audit team and assist in facilitation of the audit process. Generally, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. In some cases, items of concern are discussed during a debriefing convened at the end of the on-site review process.

8.2.3. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM. The SGM/GM/AGM/OM provides the necessary resources for staff to develop and implement the corrective action plans. The SQM/QM collates this information and provides a written response to the audit team. The response contains the corrective action plan and expected completion dates for each element of the plan. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

8.3. Quarterly Quality Reports

8.3.1. The SQM/QM is responsible for preparing a quarterly report to management summarizing the effectiveness of the laboratory Quality Systems. This status report will include:

- Overview of quality activities for the quarter
- Certification status
- Proficiency Testing study results
- SOP revision activities
- Internal audit (method/system) findings
- Manual integration audit findings (Mintminer)
- Raw Data and Final Report review findings
- MDL activities
- Other significant Quality System items

8.3.2. The Corporate Director of Quality utilizes the information from each laboratory to make decisions impacting the quality program compliance of the company as a whole. Each

SGM/GM/AGM/OM utilizes the quarterly report information to make decisions impacting Quality Systems and operational systems at a local level.

8.3.3. Additional information can be found in SOP S-ALL-Q-014 **Quality System Review** or its equivalent revision or replacement.

8.4. Annual Managerial Review

8.4.1. A managerial review of Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements.

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

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

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

8.5. Customer Service Reviews

8.5.1. As part of the annual managerial review listed previously, the sales staff is responsible for reporting on customer feedback, including complaints. The acquisition of this information is completed by performing surveys.

8.5.2. The sales staff continually receives customer feedback, both positive and negative, and reports this feedback to the laboratory management in order for them to evaluate and improve their management system, testing activities and customer service.

8.5.3. In addition, the labs must be willing to cooperate with customers or their representatives to clarify customer requests and to monitor the laboratory's performance in relation to the work being performed for the customers. This cooperation may include providing the customer reasonable access to relevant areas of the lab for the witnessing of tests being performed; or the preparation of samples or data deliverables to be used for verification purposes.

8.5.4. Customer service is an important aspect to Pace's overall objective of providing a quality product. Good communication should be provided to the customer's throughout projects. The lab should inform the customer of any delay or major deviations in the performance of analytical tests.

9.0. CORRECTIVE ACTION

Additional information can be found in SOP S-GB-Q-027 **Corrective and Preventive Actions** or its equivalent revision or replacement.

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

9.1. Corrective Action Documentation

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

- Internal Laboratory Non-Conformance Trends
- PE/PT Sample Results
- Internal and External Audits
- Data or Records Review (including non-technical records)
- Client Complaints
- Client Inquiries
- Holding Time violations

9.1.2. Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g., matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

9.1.3. The person who discovers the deficiency or non-conformance initiates the corrective action documentation on the Non-Conformance Corrective/ Preventive Action report and/or LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

9.1.4. In the event that the laboratory is unable to determine the cause, laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within LabTrack or on the Corrective/Preventive Action Report.

9.1.5. After all the documentation is completed, the routing of the Corrective/Preventive Action Report and /or LabTrack will continue from the person initiating the corrective action, to their immediate supervisor or the applicable Project Manager and finally to the SQM/QM, if applicable, who may be responsible for final review and signoff of corrective/preventive actions.

9.1.6. In the event that analytical testing or results do not conform to documented laboratory policies or procedures, customer requirements, or standard specifications, the laboratory shall investigate the significance of the non-conformance and document appropriate corrective actions. The proper level of laboratory management will review any departure from these requirements for technical suitability. These departures are permitted only with the approval of the SGM/GM/AGM/OM or the SQM/QM. Where necessary, Project Management will notify the customer of the situation and will advise of any ramifications to data quality (with the possibility of work being recalled). The procedures for handling non-conforming work are detailed in SOP S-GB-Q-027 **Corrective and Preventive Actions** or its equivalent revision or replacement.

9.2. Corrective Action Completion

9.2.1. Internal Laboratory Non-Conformance Trends

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

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting error

9.2.2. PE/PT Sample Results

9.2.2.1. Any PT result assessed as “not acceptable” requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

9.2.3. **Internal and External Audits**

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

9.2.4. **Data Review**

9.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data reviews (e.g., by the SQM/QM), errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

9.2.5. **Client Complaints**

9.2.5.1. Project Managers are responsible for issuing corrective action forms, when warranted, for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor for investigation. After potential corrective actions have been determined, the Project Manager reviews the corrective action form to ensure all customer needs or concerns are being adequately addressed.

9.2.6. **Client Inquiries**

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

9.2.7. **Holding Time Violations**

9.2.7.1. In the event that a holding time has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the SQM/QM must be made aware of all holding time violations.

9.2.7.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the hold time excursion and the ultimate resolution is then documented and included in the customer project file. The SQM/QM includes a list of all missed holding times in their Quarterly Report to the corporate quality office.

9.3. Preventive Action Documentation

9.3.1. Pace laboratories can take advantage of several available information sources in order to identify needed improvements in all of their systems including technical, managerial, and quality. These sources may include:

- Management Continuous Improvement Plan (CIP) metrics which are used by all production departments within Pace. When groups compare performance across the company, ways to improve systems may be discovered. These improvements can be made within a department or laboratory-wide.

- Annual managerial reviews- part of this TNI-required and NVLAP-required review is to look at all processes and procedures used by the laboratory over the past year and to determine ways to improve these processes in the future.
- Quality systems reviews- any frequent checks of quality systems (monthly logbook reviews, etc.) can uncover issues that can be corrected or adjusted before they become a larger issue.

9.3.2. When improvement opportunities are identified or if preventive action is required, the laboratory can develop, implement, and monitor preventive action plans.

10.0. GLOSSARY

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

3P Program	The Pace Analytical continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all PASI labs.
Acceptance Criteria	TNI and DoD- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI and DoD- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
Accrediting Authority	DoD- The Territorial, State or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.
Accrediting (or Accreditation) Body	DoD- Authoritative body that performs accreditation.
Accuracy	TNI and DoD- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI and DoD- 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	DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- The evaluation process used to measure the performance or effectiveness of a system and its elements against specific criteria. Note: In this standard (DoD), assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	DoD- A process in which a sample is converted to free atoms.

Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. DoD- A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.
Batch	TNI and DoD- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. South Carolina- same definition as TNI except 24 hours should be changed to 8 hours.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Blind Sample	DoD- A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.
Calibration	TNI and DoD- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. DoD- The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	DoD- A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration. DoD- A substance or reference material used to calibrate an instrument.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. DoD- A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.
Chain of Custody	DoD- An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of custody Form (COC)	TNI and DoD- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	DoD- Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Confirmation	TNI and DoD- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures.
Conformance	DoD- An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.

Congener	DoD- A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a CVS in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Data Audit	DoD- A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e. that they meet specified acceptance criteria).
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.

Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form. DoD- 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	DoD- 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.
Demonstration of Capability	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate acceptable accuracy.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate is 1%.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form.
Document Control	DoD- 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.
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	DoD- The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	DoD- A solvent used to carry the components of a mixture through a stationary phase.
Elute	DoD- To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	DoD- A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	DoD- The process of measuring or collecting environmental data.

Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.
False Positive	DoD- An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition. Note: For DoD, the finding must be linked to a specific requirement.
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.

Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The time elapsed from the time of sampling to the time of extraction or analysis, or from extraction to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	DoD- One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP-AES that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.

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

Laboratory Duplicate	DoD- Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
LabTrack	Database used by Pace Analytical to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A training database used by Pace Analytical to train their employees. This system is a self-paced system which is capable of tracking all employee training requirements and documentation.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. DoD- The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate is 1%.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.
Management	DoD- Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	DoD- System to establish policy and objectives and to achieve those objectives.
Manager (however named)	DoD- The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI and DoD- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.

Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. DoD- A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI and DoD- 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	TNI and DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI and DoD- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	DoD- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	DoD- 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.
MintMiner	Program used by Pace Analytical to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.

Negative Control	DoD- Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	DoD- An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Performance Audit	DoD- The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	DoD- Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI and DoD- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI- Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. DoD- Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing	TNI and DoD- A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Proficiency Testing Program	TNI and DoD- 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 Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria. DoD- A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.
Protocol	TNI and DoD- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client. DoD- An integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	DoD- A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. DoD- The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of the users.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. DoD- 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.
Quality Manual	TNI and DoD- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions are to be used for purposes of batch and quality control requirements: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard. The quantitation range lies within the calibration range.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. DoD- Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank)	DoD- A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. DoD- A 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 Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location. DoD- A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.
Reference Toxicant	DoD- The toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e. statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A client-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (or otherwise named)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	DoD- Denotes a mandatory specification; often designated by the term "shall".
Retention Time	DoD- The time between sample injection and the appearance of a solute peak at the detector.
Sample	DoD- Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by Pace Analytical sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).

Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selective Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. DoD- The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.
Sensitivity	TNI and DoD- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	DoD- Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	DoD- Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio	DoD- 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.
Spike	DoD- A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI and DoD- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.

Standard (Chemical)	DoD- Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	DoD- A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. DoD- 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 Reference Material (SRM)	DoD- A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Supervisor	DoD- The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes specifically named by a client (also called project-specific analytes).
Technical Director	DoD- Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	DoD- A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.

Test Method	DoD- An adoption of a scientific technique for performing a specific measurement as documented in a laboratory SOP or as published by a recognized authority.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. DoD- The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	DoD- A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty Measurement	The parameter associated with the result of a measurement that characterized the dispersion of the values that could be reasonably attributed to the measurand (i.e. the concentration of an analyte).
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification	TNI and DoD- Confirmation by examination and objective 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 adjustment, to repair, 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 instrument's individual record.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).
Work Cell	DoD- A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented.

11.0. REFERENCES

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12.0. REVISIONS

The PASI Corporate Quality Office files both a paper copy and electronic version of a Microsoft Word document with tracked changes detailing all revisions made to the previous version of the Quality Assurance Manual. This document is available upon request. All revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual 16.0	Section 2.6.5: added VM/Duluth. Sections 2.7.1.3 and 2.7.2.2: added SOT references. Section 4.1.2: added parenthetical phrase directing the reader to the glossary section. Section 4.1.3: added language from old section 4.1.4 and deleted language in order to match current practices. Section 4.1.4: .reworded for clarity. Also added last sentence in red text to allow labs to insert additional method blank requirements. Sections 4.1.7, 4.2.9, 4.4.4, and 6.2.7.8: revised wording per updated Ohio VAP requirements. Sections 4.5.2 and 4.6.1: added 'calibration standard' to list of QC items that require the addition of surrogates and internals. Also added red letter text for additional lab-specific information. Section 4.10.3: fixed LOQ verification language to match TNI standard (V1M4, section 1.5.2.2.c). Old section 4.12.2: deleted. Covered in reference in current section 4.12.5. Section 6.2.3: moved language that had been in the 'organic calibration only' section to this general calibration section. The language in this section applies to both organic and inorganic tests. Section 6.2.7.3: added clarification statement regarding the calibration verification standard. Section 6.3.7.1: reworded for clarity and added red letter text for calibration of micro-liter syringes. Section 7.2.5: added language specifying secondary reviewer documents approval of manual integrations. Section 7.2.7: added reference to the Manual Integration SOP. Section 7.2.8: added new red-letter text language to match Data Checker SOP. Section 7.2.9: added new red-letter text language to comply with DoD QSM 4.2. Section 8.3.1: deleted items in order to match current SOP S-ALL-Q-014. Added red-letter text to the following sections for Ohio VAP labs: 2.5.2.1, 4.5.2.1, 4.6.3, and 7.6.2. Attachment VI: added red letter text under title to satisfy AZ state requirement. Attachment VIII, Analyte Chart: changed holding times expressed as '6 Months' to '180 Days' to match actual practice as defined by LIMS acodes. Attachment VIII, Analyte Chart: added explanation under the header to explain the holding times expressed in the chart.	05Feb2013

ATTACHMENT I- QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

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

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

PERCENT DIFFERENCE (%D)

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

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

PERCENT DRIFT

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

RELATIVE PERCENT DIFFERENCE (RPD)

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

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

CORRELATION COEFFICIENT (R)

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

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

ATTACHMENT I- QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

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

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

AVERAGE ($\overline{X}$)

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

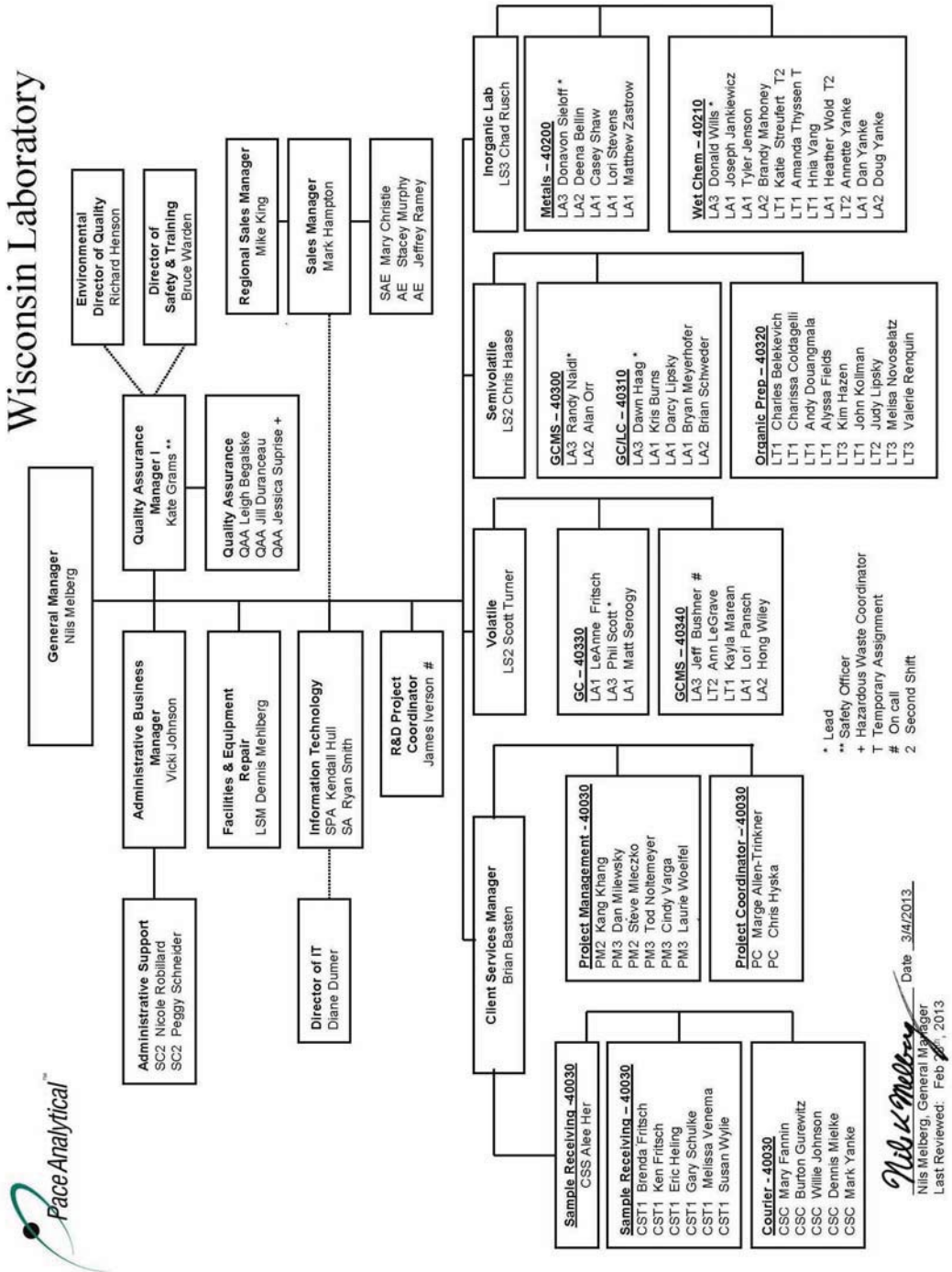
where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

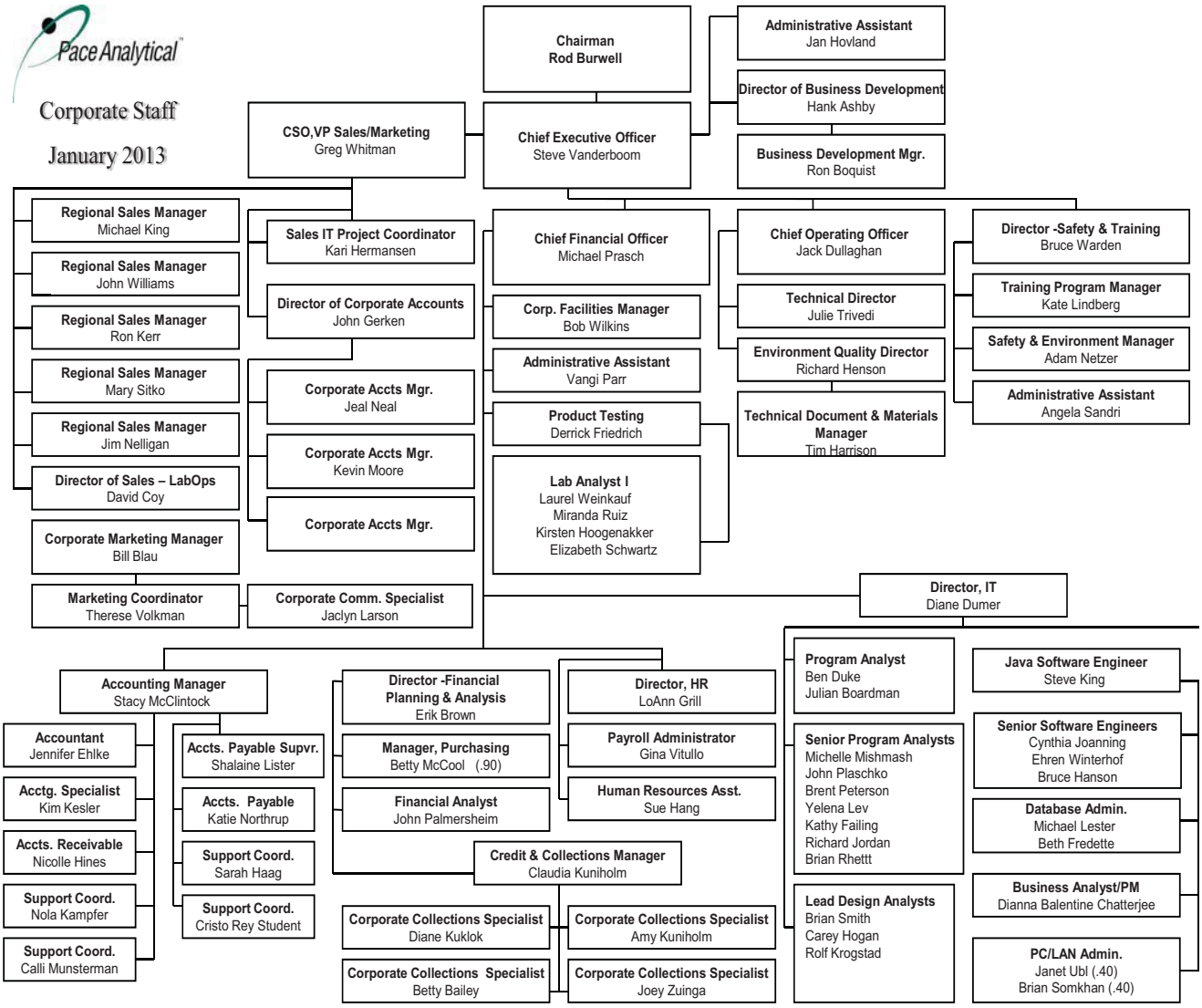
$$RSD = \frac{S}{\overline{X}} * 100$$

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

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



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



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

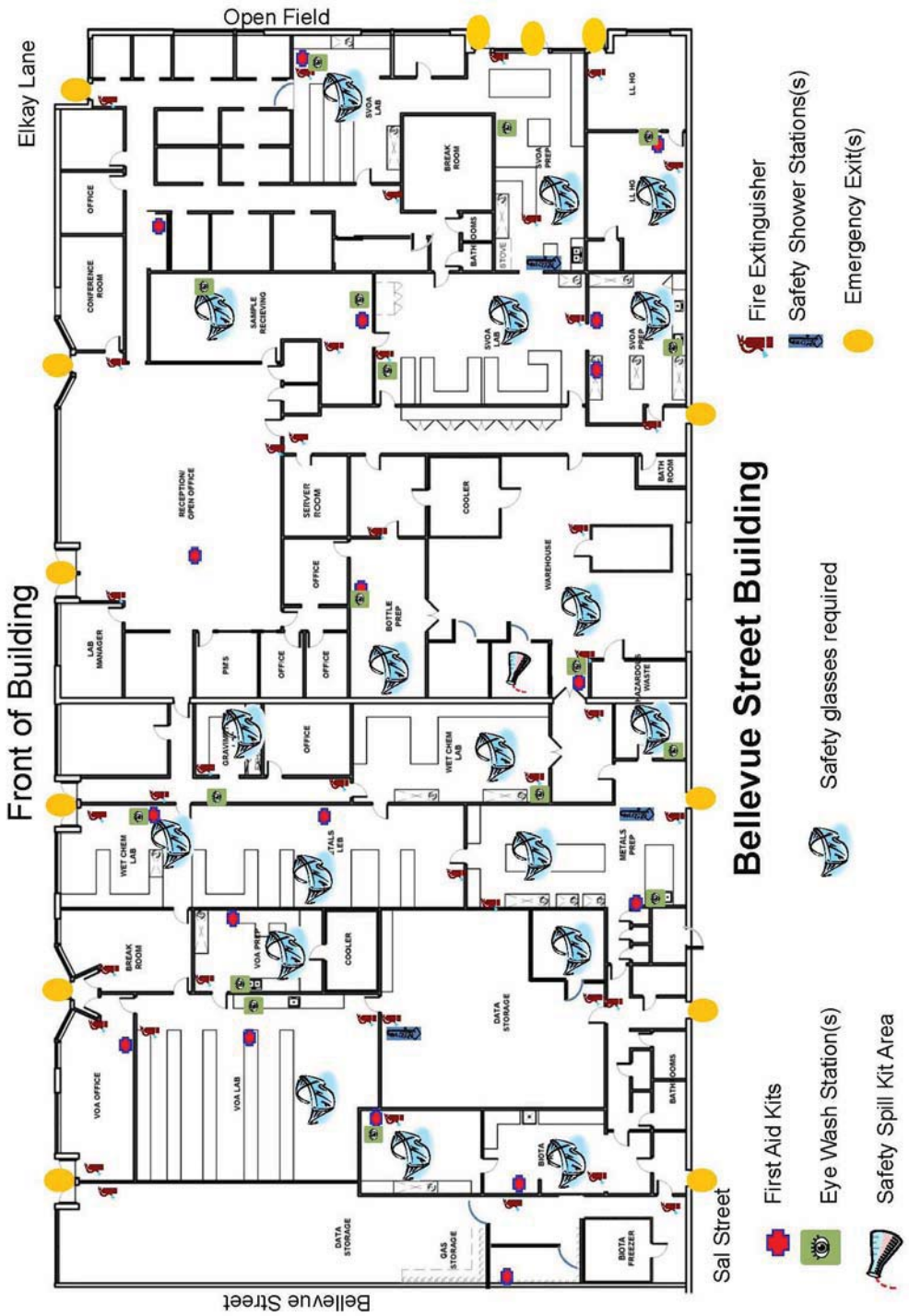
DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
QA	Hot Wire Anemometer	40HWA02	Control Company	4330	-	Air Flow Velocity
SVOA	GC	40GCS1	Hewlett-Packard	5890	FID	WI-DRO/8015
SVOA	GC	40GCS3	Hewlett-Packard	5890	FID	WI-DRO/8015
SVOA	GC	40GCS4	Hewlett-Packard	5890	FID	WI-DRO/8015SCREEN
SVOA	GC	40GCS5	Hewlett-Packard	5890	FID	WI-DRO/8015 ALCOHOL
SVOA	GC	40GCS6	Hewlett-Packard	5890	ECD/ECD	8081
SVOA	GC	40GCS7	Hewlett-Packard	6890	ECD/ECD	
SVOA	GC	40GCS8	Hewlett-Packard	6890	ECD/ECD	8081
SVOA	GC	40GCS9	Hewlett-Packard	6890	ECD/ECD	8082
SVOA	GC	40GCSA	Hewlett-Packard	5890	ECD/ECD	Screener
SVOA	GC	40GCSB	Hewlett-Packard	6890	ECD/ECD	8082
SVOA	GC	40GCSC	Hewlett-Packard	6890	ECD/ECD	8082
SVOA	GC	40GCSE	Hewlett-Packard	5890	ECD/ECD	8082
SVOA	GC	40GCSF	Hewlett-Packard	7890	FID	WI-DRO/8015
SVOA	GC/MS	40MSS1	Hewlett-Packard	5890	HP 5972	8270C/625
SVOA	GC/MS	40MSS2	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS3	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS4	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS5	Hewlett-Packard	5890	HP 5972	8270 SCREEN
SVOA	GC/MS	40MSS6	Hewlett-Packard	5890	HP 5972	8270C
SVOA	GC/MS	40MSS7	Hewlett-Packard	7890A	HP 5975	8270C
SVOA	Separatory Funnel Extractor	40SFE1	Lab Line	-	-	SW846 3510C
SVOA	Separatory Funnel Extractor	40SFE2	Lab Line	-	-	SW846 3510C
SVOA	Separatory Funnel Extractor	40SFE3	Lab Line	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC1	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC2	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC3	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC4	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC5	Zymark	-	-	SW846 3510C
SVOA	Sonicator	40SON3	Lab Line	9334	-	
SVOA	Precision Water Bath	40WB01	Precision	-	-	
SVOA	Water Baths	40WB02	-	-	-	
SVOA	Tube Heaters	-	Kontes	-	-	
SVOA	Accuprep MPS GPC Cleanup System (John)	40GPC2	J2 Scientific	-	-	SW846 3640A
SVOA	Accuprep MPS GPC Cleanup System (Jane)	40GPC1	J2 Scientific	-	-	SW846 3640A
SVOA	Mars Xtraction Microwave System	40MIC1	CEM	907501	-	SW846 3546
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX1	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX2	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX3	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX4	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX5	Gerhardt Soxtherm	SE-416	-	SW846 3540C

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX6	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX7	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX8	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Sonifier Cell Disruptors with Horns	Sonicator #2	-	-	-	SW846 3550B
SVOA	Sonifier Cell Disruptors with Horns	40SON1	Misonix	3000		SW846 3550B
SVOA	Sonifier Cell Disruptors with Horns	40SON2	Misonix	S-4000		SW846 3550B
SVOA	Environ Shaker	40SKR1	LabLine	3527	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT1	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT2	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT3	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT4	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT5	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT6	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT7	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT8	-	-	-	-
SVOA	Extraction Mixer	40EXT1	-	-	-	-
SVOA	Extraction Mixer	40EXT2	-	-	-	-
SVOA	Extraction Mixer	40EXT3	-	-	-	-
SVOA	Extraction Mixer	40EXT4	-	-	-	-
SVOA	Extraction Mixer	40EXT5	-	-	-	-
VOA	GC/MS	40MSV1	Hewlett-Packard	5890	HP 5972	SW846 8260B/5030
VOA	GC/MS	40MSV2	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV3	Agilent	6850	Agilent 5975	SW846 8260B/624/5030B
VOA	GC/MS	40MSV5	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV7	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV8	Agilent	6850	Agilent 5975B	SW846 8260B/624/5030B
VOA	GC/MS	40MSVA	Hewlett-Packard	7890	Agilent 5975C	SW846 8260B/624/5030B
VOA	GC/MS	40MSVB	Hewlett-Packard	7890A	Agilent 5975C	SW846 8260B/624/5030B
VOA	GC	40GCV1	Hewlett-Packard	5890	Agilent 5975	SW846 8021/WI MOD GRO
VOA	GC	40GCV2	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
VOA	GC	40GCV3	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV4	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV5	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV8	Hewlett-Packard	5890	FID	SW846 8015 - MEE
VOA	GC	40SCREEN1	Hewlett-Packard	5890	FID	Screener
VOA	GC	40SCREEN2	Hewlett-Packard	5890	FID	Screener
VOA	SNOOP	40SCREEN3	Research Engineers	-	-	Screener
VOA	Sonicator	40SON4	LabLine	9314	-	VOA Prep
VOA	Sonicator	40SON5	LabLine	9333	-	VOA Prep
WET	Apollo	40WTA5	Tekmar/Dohrmann	Apollo 9000	-	EPA 9060
WET	Fusion	40WTA7	Teledyne	14-9600-100	-	EPA 9060, SM 5310C
WET	Analytik Jena	40WTAA	Analytik Jenna	Multi EA 4000	-	EPA 9060, SM 5310C
WET	SmartChem	40WTA9	Westco Scientific	Smartchem 200		
WET	Oxygen Meter	40WET1	YSI	5000	YSI 5010	SM 5210B--01, SM 4500-O G
WET	BOD AUTOEZ	40WETE	Thermo/Orion	10060020	-	SM 5210B
WET	Flashpoint Instrument A	40WET4	Precision	-	-	EPA 1010A
WET	Flashpoint Instrument B	40WET5	Fisher	-	-	EPA 1010A
WET	Turbidimeter	40WET6	Hach	2100P	-	EPA 180.1, SM2130B
WET	Conductivity Meter	40WET7	Accument	30	-	EPA 120.1
WET	pH Meter	40WET8	Orion	720A	-	pH Meter
WET	EH Meter	40WET9	Accument	AB15	-	EH Meter
WET	Radiometer	40WET0	Titration	TIM840		Alkalinity - 2320B
WET	pH meter	40WETB	Symphony	SB20	-	1311, 1312, ASTM D3987
WET	pH Meter	40WETC	Orion	720A	-	pH Meter
WET	pH Meter	40WETD	Corning	320	-	pH Meter
WET	MICRO DIST Rapid Distillation System	40MD1	Lachat	-	-	350.1, 9012A
WET	MICRO DIST Rapid Distillation System	40MD2	Lachat	-	-	350.1, 9012A
WET	MICRO DIST Rapid Distillation System	40MD3	Lachat	-	-	350.1, 9012A
WET	Block Digestion System	40HB01	Westco Scientific	40/20	-	365.4, 351.2
WET	Block Digestion System	40HB02	Tecator	2040	-	365.4, 351.2
WET	Hot Block Digestion System	40HB03	Environmental Express	SC100	-	6010/6020
WET	COD Reactor	40COD1	Hach	45600	-	410.4
WET	COD Reactor	40COD2	Bioscience INC	100 003	-	410.4
WET	Direct Reading Spectrophotometer	40WTA1	Hach	DR 2000	-	EPA 410.4, SM3500Cr-B, Hach 8146, Walkley Black TOC
WET	Quik Chem 8000	40WTA2	Lachat	8000	-	350.1, 365.4, 9012A, 310.2, 351.2, 353.2
WET	Ion Chromatograph	40WTA3	Dionex	DX-120	-	EPA 300.0, 9056A
WET	Ion Chromatograph	40WTA4	Dionex	DX-120	-	EPA 300.0, 9056A

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
WET	Ion Chromatograph	40WTAB	Thermo Scientific	ICS-110	-	EPA 300.0, 9056A
WET	Quik Chem 8500 Series 2	40WTAC	Lachat	8500 Series 2	-	EPA 353.2, 350.1
WET	Hellige Colony Counter	-	Hellige	-	-	
WET	Steril-Quick Autoclave	-	National	704-9000-D	-	
WET	Stereomaster Stereoscope	-	Fisher	FW 99-20-1385	-	
WET	Sonicator	40SON6	Branson	8210	-	
WET	TCLP Tumbler	40TBL1	Environmental Express	-	-	1311, 1312, ASTM D3987
WET	TCLP Tumbler	40TBL2	Environmental Express	-	-	1311, 1312, ASTM D3987
WET	Reaction Vessel Tumbler	40TBL3	Reaction Vessel	-	-	1311, 1312, ASTM D3987
WET	Centrefudge	40CENT1	International Equipment Co	H260	-	
WET	Buret	40BUR1	KIMAX	253	-	
WET	Buret	40BUR2	Pyrex	2103	-	
MET	Hot Water Bath	40WB05	Thermo Fisher Scientific	2841	-	
MET	ICP	40ICP2	Thermo	ICAP 6500	-	
MET	Chiller	40ICP2	Neslab/Thermo	Selex 900	-	6010
MET	ICPMS	40ICM2	Thermo	X Series 2	-	6020
MET	Chiller	40ICM2	Neslab/Thermo	Merlin M75	-	6020
MET	UPS	40ICM2	Toshiba	1600EP		
MET	Vacuum Pump	40ICM2	Edwards	EM28		
MET	ICPMS	40ICM3	Thermo	X Series 2	-	6020
MET	Chiller	40ICM3	Neslab/Thermo	2500	-	6020
MET	UPS	40ICM3	Toshiba	1600EP		
MET	Vacuum Pump	40ICM3	Edwards	EM28		
MET	Quick Trace Mercury Analyzer	40HG2	Cetac	M-7500		7470/7471
MET	DMA-80 Direct Mercury Analyzer	40HG4	Milestone	DMA-80	-	7473
MET	Hot Block Metals Digestion System	40HB04	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	40HB06	Environmental Express	SC154	-	6010/6020
MET	Hot Block Metals Digestion System	40HB07	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	40HB08	Environmental Express	SC196	-	6010/6020
MET	Low-level Mercury Analyzer	40LHG1	Tekran	2500	2500	1631E
MET	Low Level Mercury Analyzer	40LHG4	Analytik Jena	Mercur	-	1631E
MET	Low Level Mercury Analyzer	40LHG5	Cetac	M-8000	-	1631E

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



ATTACHMENT V- LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
MANUALS					
Quality Manual	14.1	Quality Manual	1-Oct-04	7-Oct-11	Quality
CHP/Safety	8.0	Chemical Hygiene Plan/Safety Manual	18-Jul-11	1-Jun-12	Safety
SAMPLE MAN					
S-ALL-C-002	03	Bottle Order Database	4-Oct-06	5-Oct-12	Sample Management
S-ALL-C-005	03	Operation of PacePort Customer Feedback Form	27-Nov-07	16-Nov-12	Sample Management
S-GB-C-001	04	Procedure to Preserve Samples for Volatile Organic Analysis of Solid Matrices by Method 5035	12-Apr-05	5-Oct-12	Sample Management
S-GB-C-002	04	Extruding Sample from 25 g EnCores for Volatile and Semivolatile Analyses	12-Apr-05	5-Oct-12	Sample Management
S-GB-C-005	04	Maintenance of Ice Chests and Shipping Containers	1-Jun-05	7-Dec-11	Sample Management
S-GB-C-007	03	Laboratory Tracking of Samples	6-Jun-05	27-Sep-11	Sample Management
S-GB-C-008	03	Measurement of Percent Moisture in Soils and Solids	19-Dec-06	5-Oct-12	Sample Management
S-GB-C-009	02	Subcontracting Samples		8-Oct-12	Sample Management
S-GB-C-010	04	Sample Management	12-Jul-04	10-Dec-12	Sample Management
S-GB-C-011	01	Bottle Preparation	4-Oct-06	5-Oct-12	Sample Management
S-GB-C-012	01	Review of Analytical Requests	23-Dec-08	26-Sep-12	Sample Management
METALS					
S-GB-M-001	02	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry by 1631	1-Dec-06	6-Jul-11	Metals
S-GB-M-004	04	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy (7470A/7471A_B)	28-Aug-06	19-Dec-12	Metals
S-GB-M-005	05	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010C	23-Sep-08	19-Dec-12	Metals
S-GB-M-006	04	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020A	25-Sep-08	5-Dec-11	Metals
S-GB-M-007	02	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020	13-May-10	5-Dec-11	Metals
S-GB-M-008	02	Cleaning Metals Glassware	5-May-05	21-Sep-12	Metals
S-GB-M-009	02	Hardness by Calculation	27-Oct-05	28-Sep-11	Metals
S-GB-M-011	02	The Determination of Mercury in Biological Samples by Cold Vapor Atomic Absorption Spectroscopy (245.6)	28-Dec-06	5-Dec-11	Metals
S-GB-M-013	01	Soil Fraction Preparation for Lead Analysis	21-Mar-06	6-Jul-11	Metals
S-GB-M-014	03	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010B	28-Feb-07	19-Dec-12	Metals
S-GB-M-015	02	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry (1631E)- Analytik Jena	16-Aug-11	19-Dec-12	Metals
S-GB-M-016	00	Determination of Mercury in Solids by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry	21-Mar-12	21-Mar-12	Metals

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-M-017	00	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy - CETAC M-7500 (7470A/7471A_B)	3-Aug-11	8-Aug-11	Metals
MICRO					
S-GB-MB-001	00	Fecal Coliform Determination Using the Membrane Filter Technique	13-Sep-05	5-Oct-12	Wetchem
S-GB-MB-002	00	Heterotropic Plate Count	17-Nov-05	5-Oct-12	Wetchem
S-GB-MB-003	00	Colisure Presence/Absence Test for Detection and Identification of Coliform Bacteria and *Escherichia coli* in Drinking Waters	28-Nov-05	5-Oct-12	Wetchem
WET CHEM					
S-GB-I-001	08	Total Sulfide, Iodometric Titration	26-Jan-05	16-Nov-12	Wetchem
S-GB-I-002	06	Flash Point (Pensky-Martens Closed Cup Method For Ignitability	27-Jan-05	3-Dec-12	Wetchem
S-GB-I-009	04	Ion Chromatography- Sulfate, Nitrate, Flouride (EPA 300.0)	7-Feb-05	28-Sep-12	Wetchem
S-GB-I-010	03	Wet Chemistry Glassware Cleaning	5-Feb-05	21-Sep-12	Wetchem
S-GB-I-013	03	Free Liquids (Paint Filter)	13-Jul-05	21-Sep-12	Wetchem
S-GB-I-015	03	Oxidation - Reduction Potential (Eh) Measurement	29-Jul-05	28-Sep-12	Wetchem
S-GB-I-016	03	Specific Gravity	25-Aug-05	28-Sep-12	Wetchem
S-GB-I-017	03	Ferrous Iron	30-Aug-05	28-Sep-12	Wetchem
S-GB-I-020	04	Color Determination in Aqueous Samples	15-Sep-05	28-Sep-12	Wetchem
S-GB-I-027	03	Dissolved Oxygen	1-Mar-06	28-Sep-12	Wetchem
S-GB-I-030	03	Turbidity (Nephelometric)	10-Apr-06	3-Dec-12	Wetchem
S-GB-I-037	04	The Determination of Total Organic Carbon Using the Walkley-Black Procedure	8-Aug-05	21-Sep-12	Wetchem
S-GB-I-044	03	Biochemical Oxygen Demand	15-Feb-07	28-Sep-12	Wetchem
S-GB-I-045	04	Chromium, Hexavalent-Colorimetric	15-Feb-07	3-Dec-12	Wetchem
S-GB-I-047	03	Total Kjeldahl Nitrogen using Block Digestion and Analyzed by Lachat 8000 Flow Injection following EPA Method 351.2	17-Feb-07	7-Oct-11	Wetchem
S-GB-I-048	02	Ammonia using Micro-Distillation and Analyzed by Lachat 8000 Flow Injection following EPA Method 350.1	17-Feb-07	26-Aug-11	Wetchem
S-GB-I-051	02	Nitrate and Nitrite Analyzed by Lachat 8000 Flow Injection	20-Feb-07	26-Aug-11	Wetchem
S-GB-I-052	04	Chemical Oxygen Demand, Colorimetric, Manual (Chemetric Vials)	21-Feb-07	28-Sep-12	Wetchem
S-GB-I-053	01	Acid Volatile Sulfide / Simultaneously Extracted Metals	4-Apr-07	16-Nov-09	Wetchem
S-GB-I-059	04	The Determination of Total Organic Carbon Using the Apollo 9000 Instrument	21-May-09	28-Sep-12	Wetchem
S-GB-I-061	02	Total Phosphorus using Block Digestion and Analyzed by SmartChem	1-Jun-09	30-Oct-12	Wetchem
S-GB-I-063	02	The Determination of Total Organic Carbon Using the Teledyne Tekmar Fusion Instrument	28-Dec-09	28-Sep-12	Wetchem
S-GB-I-064	01	Cyanide by SmartChem	18-Jan-10	28-Sep-12	Wetchem

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-I-066	01	Measurement of Specific Conductance in Water	12-Dec-06	7-Dec-12	Wetchem
S-GB-I-067	01	TCLP - Toxicity Characteristic Leaching Procedure, SPLP - Synthetic Precipitation Leaching Procedure and ASTM - ASTM D 3987-85	5-Jul-10	28-Sep-12	Wetchem
S-GB-I-068	01	Measurement of Volatile Solids and Solids in Water	24-Aug-07	3-Dec-12	Wetchem
S-GB-I-069	01	Alkalinity by Titration using the Radiometer TIM840 - SM 2320B	17-Dec-10	24-Aug-11	Wetchem
S-GB-I-070	01	Alkalinity by SmartChem (EPA 310.2)	19-Apr-11	24-Sep-12	Wetchem
S-GB-I-071	01	Measurement of pH in Water, Soil, and Waste	12-Dec-06	28-Sep-12	Wetchem
S-GB-I-072	01	Acidity by Titration using the Radiometer TIM840 - SM 2310B_305.1	1-May-12	16-Nov-12	Wetchem
S-GB-I-073	00	The Determination of Total Organic Carbon Using the EA4000 Instrument (EPA 9060A)	20-Sep-12	28-Sep-12	Wetchem
GAS SECTION					
S-GB-O-005	04	Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO	29-Apr-05	16-Nov-12	Gas
S-GB-O-006	04	Modified Method for Determination of Gasoline Range Organics	27-Apr-05	19-Nov-12	Gas
S-GB-O-008	05	Total Petroleum Hydrocarbons - Gasoline by Gas Chromatography Using Flame-ionization Detectors	15-Jun-05	10-Dec-12	Gas
S-GB-O-009	04	Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors	27-Apr-05	3-Dec-12	Gas
S-GB-O-010	04	Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics	29-Apr-05	30-Oct-12	Gas
S-GB-O-017	04	Analysis of Dissolved Methane, Ethane, and Ethene in Ground Water by Static Headspace and Gas Chromatography	27-Apr-05	3-Dec-12	Gas
SVOA SECTION					
S-ALL-O-038	01	Processing Tentatively Identified Compounds (TICs) for GC/MS	9-Jun-11	3-Dec-12	QA
S-GB-O-015	04	Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics	11-May-05	28-Sep-11	SVOA
S-GB-O-018	03	Determination of DRO Sample Weight and Methylene Chloride Addition	13-Apr-05	21-Sep-12	SVOA
S-GB-O-019	04	WI Modified Method for Determination of Diesel Range Organics	13-Apr-05	13-Jul-11	SVOA
S-GB-O-023	05	Total Petroleum Hydrocarbons - Diesel	25-May-05	7-Jul-11	SVOA
S-GB-O-025	03	Alcohols & Glycols by Direct Injection GC/FID	14-Dec-05	1-May-12	SVOA
S-GB-O-026	06	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by 8082	23-May-06	1-May-12	SVOA
S-GB-O-027	05	Analysis of Organochlorine Pesticides by Gas Chromatography by 8081	23-May-06	13-Jul-11	SVOA
S-GB-O-028	04	Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes	24-May-06	21-Sep-12	SVOA

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-O-031	03	Extraction of Biological Samples for Organochlorine Pesticides/PCBs	20-Jan-05	21-Sep-12	SVOA
S-GB-O-032	03	Gel Permeation Chromatography	26-Jan-05	21-Sep-12	SVOA
S-GB-O-033	02	Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis	8-Feb-05	21-Sep-12	SVOA
S-GB-O-034	03	Sulfuric Acid Cleanup	3-Feb-05	7-Jul-11	SVOA
S-GB-O-036	03	Florisil Cleanup for PCB and Toxaphene Samples	26-Jan-05	7-Jul-11	SVOA
S-GB-O-037	03	Florisil Cartridge Cleanup for Organochlorine Pesticide Samples	26-Jan-05	28-Sep-11	SVOA
S-GB-O-038	04	Silica Gel Cleanup for Organic Analysis	3-Feb-05	21-Sep-12	SVOA
S-GB-O-039	03	Copper Cleanup for the Removal of Sulfur from PCB and Toxaphene Samples	23-Aug-05	7-Jul-11	SVOA
S-GB-O-040	04	Extraction of Wipes and Oil for PCB Analysis	11-Jul-08	1-May-12	SVOA
S-GB-O-041	04	Extraction of PCBs Using the Automated Soxhlet 3541	23-Aug-07	1-May-12	SVOA
S-GB-O-043	03	Extraction of Toxaphene Using the Automated Soxhlet 3541	3-May-07	21-Sep-12	SVOA
S-GB-O-044	02	Determination of Low Level PAHs by GC/MS-SIM in Solid and Biological Matrices:8270SIMPAH	5-Feb-05	13-Jul-11	SVOA
S-GB-O-045	05	Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons, Base/Neutral/Acids, and Total Petroleum Hydrocarbons in Solid Matrices by 3546	21-Sep-07	6-Feb-13	SVOA
S-GB-O-047	03	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography following 8082A	22-Sep-08	21-Sep-12	SVOA
S-GB-O-048	02	Analysis of Fox River Polychlorinated Biphenyls (PCBs) by Gas Chromatography	4-Jun-09	13-Jul-11	SVOA
S-GB-O-049	04	Determination of Semi-Volatile Organics by GC/MS (8270)	28-Nov-06	26-Sep-12	SVOA
S-GB-O-050	02	Determination of Semi-Volatile Organics by gc/ms (Selective Ion Monitoring)	28-Nov-06	3-May-12	SVOA
S-GB-O-052	01	Extraction of PCBs In Tissue Using the Automated Soxhlet	11-Nov-09	1-May-12	SVOA
S-GB-O-053	02	Separatory Funnel Extraction by 3510C	16-Nov-06	3-May-12	SVOA
S-GB-O-054	01	Ultrasonic Extraction by 3550B	16-Nov-06	1-May-12	SVOA
S-GB-O-055	01	WI Modified Method for Determination of Extended Range Diesel Range Organics	12-Jul-11	1-May-12	SVOA
S-GB-O-057	01	Analysis of Toxaphene by Gas Chromotography Using St.John's River Waste Management Department (SJRWMD) Protocol	10-Jul-10	26-Sep-12	SVOA
S-GB-O-058	01	Analysis of Toxaphene by Gas Chromotography Using Hercules 97 and Total Area Under the Curve (TAUC)	7-Jul-10	26-Sep-12	SVOA
S-GB-O-060	00	Extraction of TPH-Diesel Using the Automated Soxhlet_3541	7-Jul-11	7-Jul-11	SVOA
S-GB-O-062	00	SVOA Sample Screening	6-Feb-13	6-Feb-13	SVOA
VOA SECTION					
S-GB-O-001	06	Sample Screening Volatile Organics Prior to Preparation	24-Aug-05	14-Nov-11	VOA

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-O-012	04	Cleaning of Syringes Used in the Analysis of Volatile Organics	12-Apr-05	17-Oct-12	VOA
S-GB-O-056	05	Determination of Volatile Organics by GC/MS 8260	14-Jul-06	30-Dec-11	
IT					
S-GB-IT-001	00	System Security and Integrity	26-Jan-05	28-Sep-11	IT
S-GB-IT-002	00	Server Back-up	4-Jun-09	30-Oct-12	IT
QA SYSTEM					
S-ALL-Q-003	07	Document Numbering	12-Apr-04	29-Nov-12	QA
S-ALL-Q-007	03	EPIC Pro: Acode Validation	15-Feb-05	24-Aug-11	QA
S-ALL-Q-008	02	EPIC Pro: Acode Addition/Modification	7-Jun-10	13-Jul-11	QA
S-ALL-Q-009	05	Laboratory Documentation	22-Dec-04	29-Nov-12	QA
S-ALL-Q-014	02	Quality system Review	5-May-04	12-Sep-12	QA
S-ALL-Q-015	00	Review of Laboratory Management System	12-Sep-12	12-Sep-12	QA
S-ALL-Q-020	05	Orientation and Training Procedures	30-Jun-05	5-Oct-12	QA
S-ALL-Q-022	03	3P Program Continuous Process Improvement	19-May-05	12-Sep-12	QA
S-ALL-Q-028	02	Use and Operation of Lab Track System	22-May-07	5-Oct-12	QA
S-ALL-Q-029	03	MintMiner Data File Review	20-Nov-07	19-Dec-12	QA
S-ALL-Q-030	04	Operation of Data Checker for Epic Pro	5-Aug-10	21-Mar-12	QA
S-ALL-Q-034	01	Anonymous Hotline Procedure	5-Aug-10	24-Aug-11	QA
S-ALL-Q-035	01	Data Recall	31-Jan-11	21-Sep-12	QA
S-ALL-Q-037	01	Data Review Process	9-Feb-11	11-Apr-11	QA
S-GB-Q-001	03	Employee Master Signature Log	10-Jan-06	14-Nov-11	QA
S-GB-Q-002	03	Training Record Files Maintained by the QAO	10-Jan-06	14-Nov-11	QA
S-GB-Q-003	02	Data Reduction, Validation, and Reporting	5-Jan-09	7-Oct-11	QA
S-GB-Q-004	03	Laboratory Notebooks and Logbooks	17-Jan-06	14-Nov-11	QA
S-GB-Q-005	03	Precision and Accuracy Measurement and Evaluation	23-Mar-06	14-Nov-11	QA
S-GB-Q-006	03	Data Archiving	27-Apr-06	14-Nov-11	QA
S-GB-Q-007	04	Method of Syringe Technique	9-May-06	14-Nov-11	QA
S-GB-Q-008	04	Preventative, routine, and non-routine maintenance	9-May-06	30-Dec-11	QA
S-GB-Q-009	04	Common Laboratory Calculations and Statistical Evaluation of Data	11-May-06	29-Nov-12	QA
S-GB-Q-010	04	Estimation of Measurement Uncertainty	15-Feb-07	25-Jul-12	QA
S-GB-Q-011	01	Method Validation and Modification Studies	7-Jun-10	25-Jul-12	QA
S-GB-Q-012	02	Purchasing of Laboratory Supplies	3-Apr-06	30-Oct-12	QA
S-GB-Q-013	02	Receipt and Storage of Laboratory Supplies	21-Mar-06	21-Sep-12	QA
S-GB-Q-016	01	Management of Change	11-May-11	25-Jul-12	QA
S-GB-Q-017	01	Preparation of SOPs	5-May-04	21-Sep-12	QA
S-GB-Q-018	01	Evaluation and Qualification of Vendors	10-Apr-08	25-Jul-12	QA
S-GB-Q-019	01	Software Validation	9-Jun-09	25-Jul-12	QA
S-GB-Q-020	01	Determination of LOD and LOQ	21-Feb-05	25-Jul-12	QA
S-GB-Q-021	01	Proficiency Testing Program	15-Feb-07	21-Sep-12	QA
S-GB-Q-022	01	Internal and External Audits	30-Dec-04	26-Sep-12	QA
S-GB-Q-023	01	MCL Violation Reporting	11-May-09	26-Sep-12	QA
S-GB-Q-024	01	Manual Integration	15-Mar-05	29-Nov-12	QA
S-GB-Q-025	01	Sample Homogenization and Sub-Sampling	16-Nov-06	29-Nov-12	QA

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-Q-026	01	Standard and Reagent Management and Traceability	9-Feb-11	29-Nov-12	QA
S-GB-Q-027	00	Corrective Action / Preventative Action Process	17-Aug-05	21-Mar-12	QA
S-GB-Q-028	01	Monitoring Storage Units	20-Apr-05	10-Oct-12	QA
S-GB-Q-029	00	Document Management	30-Dec-04	30-Dec-11	QA
S-GB-Q-030	00	Support Equipment	17-Jan-05	30-Dec-11	QA
S-GB-Q-031	00	Control Charting and Trend Analysis	9-Feb-11	21-Mar-12	QA
Safety					
S-ALL-S-001	04	Hazard Assessment	1-Jan-05	5-Oct-12	Safety
S-GB-S-001	05	Regulated Soil Handling	5-Jan-06	12-Sep-12	Safety
S-GB-S-002	03	Control of Hazardous Energy Program - Lockout/Tagout	10-Jan-06	21-Sep-12	Safety
S-GB-S-004	03	Rescue Alert System Operation	16-Nov-05	21-Sep-12	Safety
S-GB-S-007	00	Label Destruction	3-Sep-10	14-Nov-11	Safety
S-GB-S-008	00	Air Quality Monitoring and Fume Hood Monitoring	4-Dec-12	10-Dec-12	Safety
Waste					
S-GB-W-001	01	Waste Handling and Management	29-Jul-05	10-Dec-12	Waste
S-GB-W-002	00	Waste Management Training Requirements	11-Feb-09	21-Sep-12	Safety
Equipment					
S-GB-E-001	02	Use and Maintenance of the NANOpure Infinity Water Purification System	28-Mar-06	27-Sep-11	Equipment
S-GB-E-002	02	Operation of Waste Disposal Equipment	21-Sep-07	27-Sep-11	Equipment
Training					
S-ALL-T-002	04	Learn Center	25-Apr-11	16-Nov-12	Training
Laboratory					
S-GB-L-001	06	Biological Tissue, Plant, and Synthetic Material Preparation	20-Jan-05	9-Sep-11	Laboratory
S-GB-L-002	03	Small Rodent Handling and Homogenization	20-Jan-05	28-Sep-12	Laboratory
S-GB-L-003	04	The Determination of Lipids in Tissues, Fats, and Plants	20-Jan-05	9-Sep-11	Laboratory
S-GB-L-004	03	Determination of Percent Solids in Tissue Samples	3-Feb-05	9-Sep-11	Laboratory
S-GB-L-005	01	Reagent Water Quality	10-Jan-08	28-Sep-11	Laboratory
S-GB-L-007	00	Cleaning of Equipment Used in the Process of Homogenizing Biological Tissue, Plant, and Synthetic Materials.	19-Jul-11	9-Sep-11	Laboratory

ATTACHMENT VI- LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE) SCOPE AND APPLICATION CERTIFICATES ARE MAINTAINED AND FILED IN THE LOCAL QUALITY DEPARTMENT

Green Bay Laboratory

Accrediting Authority	Program Category	Accrediting Agency	Certification #	Expiration Date
Florida (NELAP)	Biological Tissue	Dept of Health, Bureau of Laboratories	E87948	6/30/2013
Florida (NELAP)	Hazardous Waste - Solid	Dept of Health, Bureau of Laboratories	E87948	6/30/2013
Florida (NELAP)	Waste Water	Dept of Health, Bureau of Laboratories	E87948	6/30/2013
Georgia	Waste Water - NELAP stipulation	Environmental Protection Divison	E87948	6/30/2013
Georgia	Waste Water - NELAP stipulation	Environmental Protection Divison	E87948	6/30/2013
Illinois (NELAP)	Hazardous Waste - Solid	Illinois EPA	200050	8/3/2013
Illinois (NELAP)	Waste Water	Illinois EPA	200050	8/3/2013
Kentucky	UST	Environmental and Public Protection Cabinet	82	8/26/2013
Louisiana (NELAP)	Hazardous Waste - Solid	Department of Environmental Quality	4168	6/30/2013
Louisiana (NELAP)	Waste Water	Department of Environmental Quality	4168	6/30/2013
Louisiana (NELAP)	Biological Tissue	Department of Environmental Quality	4168	6/30/2013
Minnesota	Hazardous Waste	Dept of Health	055-999-334	12/31/2013
Minnesota	Waste Water	Dept of Health	055-999-344	12/31/2013
Minnesota	UST	Department of Health	055-999-344	12/31/2013
New York (NELAP)	Solid - Hazardous Waste	Dept of Health	11887	4/1/2013
New York (NELAP)	Solid - Hazardous Waste	Dept of Health	11888	4/1/2013
New York (NELAP)	Waste Water	Dept of Health	11888	4/1/2013
New York (NELAP)	Waste Water	Dept of Health	11887	4/1/2013
North Dakota	Hazardous Waste	Dept of Health Chemistry Division	R-150	6/30/2013
North Dakota	Waste Water	Dept of Health Chemistry Division	R-150	6/30/2013
South Carolina	Hazardous Waste	Dept of Health & Environmental Control	83006001	8/31/2013
South Carolina	Waste Water	Dept of Health & Environmental Control	83006001	8/31/2013
US Dept of Agriculture	Foreign Soil Permit	Dept of Agriculture	S-76505	4/1/2014
Wisconsin	Drinking Water	Dept of Natural Resources	405132750	8/31/2013
Wisconsin	Hazardous Waste	Dept of Natural Resources	405132750	8/31/2013
Wisconsin	Waste Water	Dept of Natural Resources	405132750	8/31/2013
Wisconsin	Drinking Water	Dept of Agriculture, Trade & Consumer Protection	105-444	12/31/2013

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

CHAIN-OF-CUSTODY / Analytical Request Document

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

Pace Analytical™ www.pacelabs.com

Page: of

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

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

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

REGULATORY AGENCY
☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER
☐ UST ☐ RCRA ☐ OTHER

Site Location
STATE:

Section D Required Client Information

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

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

ITEM #
MATRIX CODE (see valid codes to left)
SAMPLE TYPE (G=GRAB C=COMP)
COLLECTED: COMPOSITE START DATE / TIME; COMPOSITE END/GRAB DATE / TIME
SAMPLE TEMP AT COLLECTION
OF CONTAINERS
Preservatives: Unpreserved, H_2SO_4, HNO_3, HCl, NaOH, $Na_2S_2O_3$, Methanol, Other
Analysis Test (Y/N)
Requested Analysis Filtered (Y/N)
Residual Chlorine (Y/N)
Pace Project No./ Lab I.D.

1 2 3 4 5 6 7 8 9 10 11 12

ADDITIONAL COMMENTS
RELINQUISHED BY / AFFILIATION — DATE — TIME
ACCEPTED BY / AFFILIATION — DATE — TIME
SAMPLE CONDITIONS: Temp in °C; Received on Ice (Y/N); Custody Sealed Cooler (Y/N); Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE
PRINT Name of SAMPLER:
SIGNATURE of SAMPLER:
DATE Signed (MM/DD/YY):

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

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

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acidity	SM2310B	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Actinides	HASL-300	Water		pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid		None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Alkylated PAHs		Water		$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid		$\leq 10^oC$	1 Year/40 Days
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid		None	180 days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	$\leq 6^oC$; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	$\leq 6^oC$	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	$\leq 6^oC$	28 days
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	$\leq 6^oC$	14 Days
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	$\leq 6^oC$; $Na_2S_2O_3$	24 Hours

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Base/Neutrals and Acids	8270	Solid	8oz Glass	≤ 6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Biomarkers		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6°C	48 hours
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	14 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	48 Hours
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil		
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 6°C	6 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 6°C	6 Hours
Coliform, Total and Escherichla (E. coli)	SM9223B	Water	100mL Plastic	≤ 10°C	48 Hours after collection; results from samples analyzed 30-48 Hours after collection must be qualified as analyzed >30 hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	24 Hours
Condensable Particulate	EPA 202	Air	Solutions	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Emissions					
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present-applies to SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6°C	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	30/45 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Ferrous Iron	SN3500Fe-D	Water	Glass	None	Immediate
Flashpoint/Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics-Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics-Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6°C	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	≤ 6°C; packed jars with no headspace	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	≤ 6°C in MeOH	21 Days
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤ 6°C	14/7 Days if extracts stored ≤ 6°C or 14/14 Days if extracts stored at ≤ -10°C
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Heterotrophic Plate Count (MPC)	SM9215B	Water	100mL Plastic	≤ 6°C	24 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/SM3500Cr-C,D	Water	Plastic/Glass	≤ 6°C	24 Hours
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6°C	24 Hours after extraction
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Lipids	Pace Lipids	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid			
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	≤ 6°C	28 days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	≤ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
Methanol, Ethanol	8015 modified	Water	40mL vials	≤ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	≤ 6°C	14 Days
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	48 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass		Pest: 7/40 Days; PCB: 1 Year/1 Year
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	≤ -10°C	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; ≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6°C	1 Year/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/1 Year
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Oil Range Organics- ORO					
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	14 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; ≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid			
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days
Saturated Hydrocarbons		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days	≤ 6°C; pH<2 1:1 HCl (optional)

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				unpreserved	
Saturated Hydrocarbons		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/ 9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤ 6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black	Solid	Glass	≤ 6°C	14 Days
Total Organic Halogen (TOX)	SM5320/9020/9021	Water	Glass; no headspace	≤ 6°C	14 Days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HCl	180 days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C;	7/28 Days

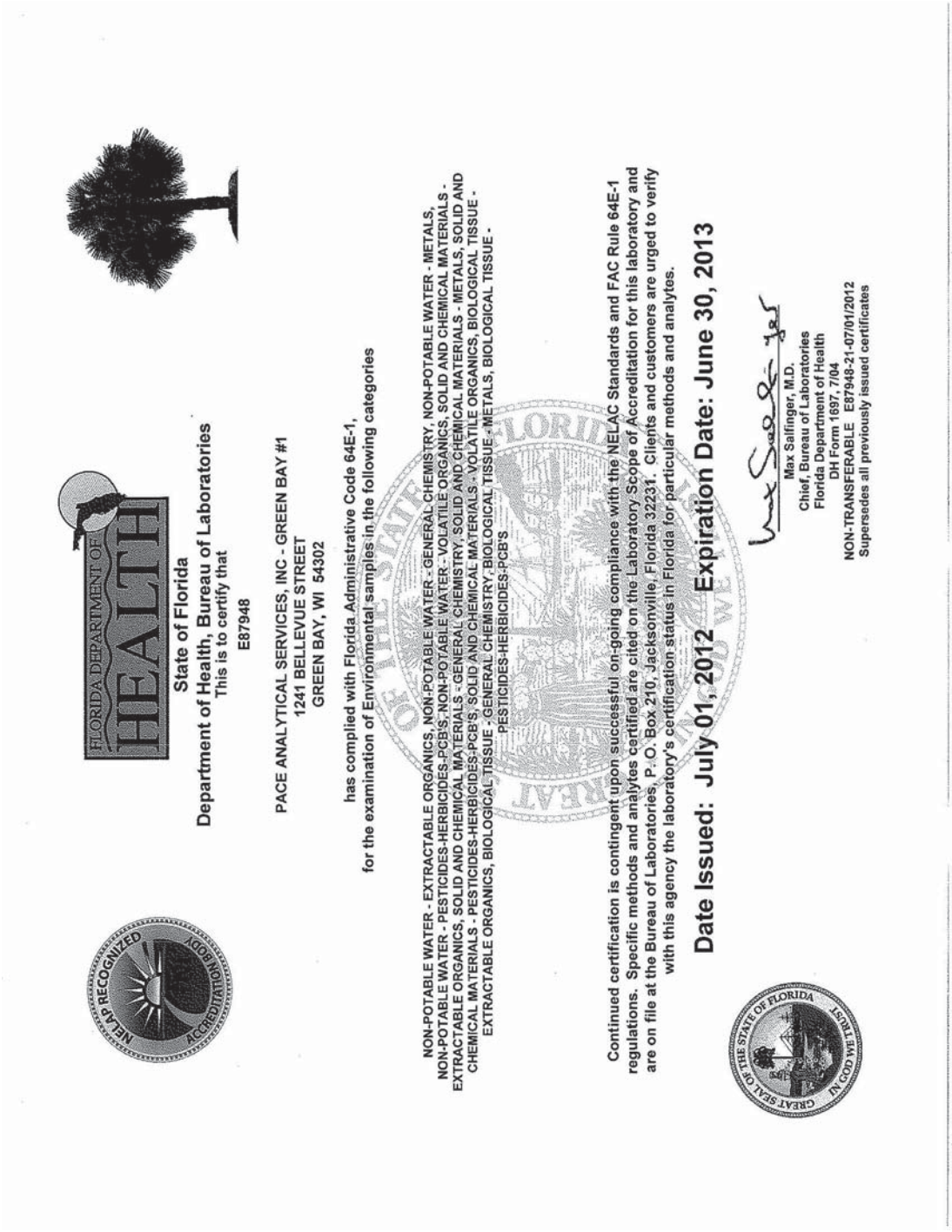
Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Hydrocarbons (aliphatic and aromatic)				packed jars with no headspace	
Volatiles	TO-14	Air	Summa Canister	None	30 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	48 Hours
Volatiles	TO-15	Air	Summa Canister	None	30 Days
Volatiles	8260	Solid	5035 vial kit	See note 1	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6°C; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days

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

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

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

ATTACHMENT IX- NELAC CERTIFICATE



FLORIDA DEPARTMENT OF HEALTH

State of Florida
Department of Health, Bureau of Laboratories
This is to certify that
E87948

PACE ANALYTICAL SERVICES, INC - GREEN BAY #1
1241 BELLEVUE STREET
GREEN BAY, WI 54302

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS, BIOLOGICAL TISSUE - EXTRACTABLE ORGANICS, BIOLOGICAL TISSUE - GENERAL CHEMISTRY, BIOLOGICAL TISSUE - METALS, BIOLOGICAL TISSUE - PESTICIDES-HERBICIDES-PCB'S

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P.O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2012 Expiration Date: June 30, 2013

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E87948-21-07/01/2012
Supersedes all previously issued certificates

NELAP RECOGNIZED ACCREDITATION BODY

GREAT SEAL OF THE STATE OF FLORIDA IN GOD WE TRUST

Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	3/19/2012
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2,4-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3,5-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2012** **Expiration Date: 6/30/2013**

Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Hexanone	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/1/2005

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2012** **Expiration Date: 6/30/2013**

Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	4/1/2005
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 625	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acetone	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetophenone	EPA 625	Extractable Organics	NELAP	4/1/2005
Acetophenone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acidity, as CaCO3	EPA 305.1	General Chemistry	NELAP	4/1/2005
Acidity, as CaCO3	SM 2310 B (4a)	General Chemistry	NELAP	5/22/2007
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	5/15/2009
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 624	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009

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Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	4/1/2005
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6010	Metals	NELAP	4/1/2005
Aluminum	EPA 6020	Metals	NELAP	4/1/2005
Ammonia as N	EPA 350.1	General Chemistry	NELAP	4/1/2005
Aniline	EPA 625	Extractable Organics	NELAP	4/1/2005
Aniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Antimony	EPA 6010	Metals	NELAP	4/1/2005
Antimony	EPA 6020	Metals	NELAP	4/1/2005
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6010	Metals	NELAP	4/1/2005
Arsenic	EPA 6020	Metals	NELAP	4/1/2005
Barium	EPA 6010	Metals	NELAP	4/1/2005
Barium	EPA 6020	Metals	NELAP	4/1/2005
Benzene	EPA 602	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Benzidine	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzoic acid	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Beryllium	EPA 6010	Metals	NELAP	4/1/2005
Beryllium	EPA 6020	Metals	NELAP	4/1/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	4/1/2005
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/1/2005
Boron	EPA 6010	Metals	NELAP	4/1/2005
Boron	EPA 6020	Metals	NELAP	4/1/2005
Bromide	EPA 300.0	General Chemistry	NELAP	4/1/2005
Bromide	EPA 9056	General Chemistry	NELAP	4/1/2005
Bromobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 624	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Cadmium	EPA 6010	Metals	NELAP	4/1/2005
Cadmium	EPA 6020	Metals	NELAP	4/1/2005
Calcium	EPA 6010	Metals	NELAP	4/1/2005
Calcium	EPA 6020	Metals	NELAP	4/1/2005

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbazole	EPA 8270	Extractable Organics	NELAP	4/1/2005
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	4/1/2005
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	4/1/2005
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chloride	EPA 300.0	General Chemistry	NELAP	4/1/2005
Chloride	EPA 9056	General Chemistry	NELAP	4/1/2005
Chlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 624	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chromium	EPA 6010	Metals	NELAP	4/1/2005
Chromium	EPA 6020	Metals	NELAP	4/1/2005
Chromium VI	SM 3500-Cr B (20th/21st Ed.)/UV-VIS	General Chemistry	NELAP	5/30/2007
Chrysene	EPA 625	Extractable Organics	NELAP	4/1/2005
Chrysene	EPA 8270	Extractable Organics	NELAP	4/1/2005
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Cobalt	EPA 6010	Metals	NELAP	4/1/2005
Cobalt	EPA 6020	Metals	NELAP	4/1/2005
Conductivity	EPA 120.1	General Chemistry	NELAP	4/1/2005
Copper	EPA 6010	Metals	NELAP	4/1/2005
Copper	EPA 6020	Metals	NELAP	4/1/2005
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibromomethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/1/2005
Diethyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Ethanol	EPA 8015	Volatile Organics	NELAP	5/15/2009
Ethanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 602	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 625	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluoride	EPA 300.0	General Chemistry	NELAP	4/1/2005
Fluoride	EPA 9056	General Chemistry	NELAP	4/1/2005
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	5/15/2009
Gasoline range organics (GRO)	WI-GRO	Volatile Organics	NELAP	5/15/2009
Hardness	SM 2340 B	General Chemistry	NELAP	4/1/2005
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/1/2005
Ignitability	EPA 1010	General Chemistry	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Iron	EPA 6010	Metals	NELAP	4/1/2005
Iron	EPA 6020	Metals	NELAP	4/1/2005
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isophorone	EPA 625	Extractable Organics	NELAP	4/1/2005
Isophorone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Isopropyl alcohol (2-Propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	4/1/2005
Lead	EPA 6010	Metals	NELAP	4/1/2005
Lead	EPA 6020	Metals	NELAP	4/1/2005
Lithium	EPA 6020	Metals	NELAP	5/15/2009
m/p-Xylenes	EPA 8021	Volatile Organics	NELAP	5/15/2009
m/p-Xylenes	EPA 8260	Volatile Organics	NELAP	5/15/2009
Magnesium	EPA 6010	Metals	NELAP	4/1/2005
Magnesium	EPA 6020	Metals	NELAP	4/1/2005
Manganese	EPA 6010	Metals	NELAP	4/1/2005
Manganese	EPA 6020	Metals	NELAP	4/1/2005

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Mercury	EPA 1631	Metals	NELAP	5/30/2007
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7470	Metals	NELAP	4/1/2005
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methanol	EPA 8015	Volatile Organics	NELAP	5/15/2009
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	5/15/2009
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6010	Metals	NELAP	4/1/2005
Molybdenum	EPA 6020	Metals	NELAP	4/1/2005
Naphthalene	EPA 625	Extractable Organics	NELAP	4/1/2005
Naphthalene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Nickel	EPA 6010	Metals	NELAP	4/1/2005
Nickel	EPA 6020	Metals	NELAP	4/1/2005
Nitrate	EPA 9056	General Chemistry	NELAP	4/1/2005
Nitrate as N	EPA 300.0	General Chemistry	NELAP	4/1/2005
Nitrite	EPA 9056	General Chemistry	NELAP	4/1/2005
Nitrite as N	EPA 300.0	General Chemistry	NELAP	4/1/2005
Nitrobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Propanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	4/1/2005
o-Xylene	EPA 8021	Volatile Organics	NELAP	5/15/2009
o-Xylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
pH	EPA 9040	General Chemistry	NELAP	4/1/2005
pH	SM 4500-H+-B	General Chemistry	NELAP	5/22/2007
Phenanthrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 625	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	4/1/2005
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Potassium	EPA 6010	Metals	NELAP	4/1/2005
Potassium	EPA 6020	Metals	NELAP	4/1/2005
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 625	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	5/30/2007
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	5/30/2007
Residue-total	SM 2540 B	General Chemistry	NELAP	5/30/2007
Residue-volatile	EPA 160.4	General Chemistry	NELAP	4/1/2005
Residue-volatile	SM 2540 E	General Chemistry	NELAP	5/30/2007
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Selenium	EPA 6010	Metals	NELAP	4/1/2005
Selenium	EPA 6020	Metals	NELAP	4/1/2005
Silicon	EPA 6020	Metals	NELAP	3/19/2012
Silver	EPA 6010	Metals	NELAP	4/1/2005
Silver	EPA 6020	Metals	NELAP	4/1/2005
Sodium	EPA 6010	Metals	NELAP	4/1/2005

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Sodium	EPA 6020	Metals	NELAP	4/1/2005
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Styrene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Sulfate	EPA 300.0	General Chemistry	NELAP	4/1/2005
Sulfate	EPA 9056	General Chemistry	NELAP	4/1/2005
Sulfide	SM 4500-S F (19th/20th/21st Ed.)/TITR	General Chemistry	NELAP	1/30/2008
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Thallium	EPA 6010	Metals	NELAP	4/1/2005
Thallium	EPA 6020	Metals	NELAP	4/1/2005
Tin	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toluene	EPA 602	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 624	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Total cyanide	EPA 335.4	General Chemistry	NELAP	4/1/2005
Total cyanide	EPA 9012	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	4/1/2005
Total organic carbon	EPA 9060	General Chemistry	NELAP	6/24/2011
Total organic carbon	SM 5310 C	General Chemistry	NELAP	6/24/2011
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	5/15/2009
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	5/15/2009
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Turbidity	SM 2130 B	General Chemistry	NELAP	4/1/2005
Uranium	EPA 6020	Metals	NELAP	3/19/2012
Vanadium	EPA 6010	Metals	NELAP	4/1/2005
Vanadium	EPA 6020	Metals	NELAP	4/1/2005
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 602	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 624	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Zinc	EPA 6010	Metals	NELAP	4/1/2005
Zinc	EPA 6020	Metals	NELAP	4/1/2005
Zirconium	EPA 6020	Metals	NELAP	3/19/2012

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State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	3/19/2012
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2,4-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3,5-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Hexanone	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	4/1/2005
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acetone	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetophenone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6010	Metals	NELAP	4/1/2005
Aluminum	EPA 6020	Metals	NELAP	4/1/2005
Ammonia as N	EPA 350.1	General Chemistry	NELAP	4/1/2005
Aniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Antimony	EPA 6010	Metals	NELAP	4/1/2005
Antimony	EPA 6020	Metals	NELAP	4/1/2005
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6010	Metals	NELAP	4/1/2005
Arsenic	EPA 6020	Metals	NELAP	4/1/2005
Barium	EPA 6010	Metals	NELAP	4/1/2005
Barium	EPA 6020	Metals	NELAP	4/1/2005
Benzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Benzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzoic acid	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Beryllium	EPA 6010	Metals	NELAP	4/1/2005
Beryllium	EPA 6020	Metals	NELAP	4/1/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/1/2005

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/1/2005
Boron	EPA 6010	Metals	NELAP	4/1/2005
Boron	EPA 6020	Metals	NELAP	4/1/2005
Bromobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Cadmium	EPA 6010	Metals	NELAP	4/1/2005
Cadmium	EPA 6020	Metals	NELAP	4/1/2005
Calcium	EPA 6010	Metals	NELAP	4/1/2005
Calcium	EPA 6020	Metals	NELAP	4/1/2005
Carbazole	EPA 8270	Extractable Organics	NELAP	4/1/2005
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chromium	EPA 6010	Metals	NELAP	4/1/2005
Chromium	EPA 6020	Metals	NELAP	4/1/2005
Chrysene	EPA 8270	Extractable Organics	NELAP	4/1/2005
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Cobalt	EPA 6010	Metals	NELAP	4/1/2005
Cobalt	EPA 6020	Metals	NELAP	4/1/2005
Copper	EPA 6010	Metals	NELAP	4/1/2005
Copper	EPA 6020	Metals	NELAP	4/1/2005
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	5/15/2009

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/1/2005
Diethyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Ethanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 8270	Extractable Organics	NELAP	4/1/2005
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	5/15/2009
Gasoline range organics (GRO)	WI-GRO	Extractable Organics	NELAP	5/15/2009
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/1/2005
Ignitability	EPA 1010	General Chemistry	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005

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State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Iron	EPA 6010	Metals	NELAP	4/1/2005
Iron	EPA 6020	Metals	NELAP	4/1/2005
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isophorone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Isopropyl alcohol (2-Propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	4/1/2005
Lead	EPA 6010	Metals	NELAP	4/1/2005
Lead	EPA 6020	Metals	NELAP	4/1/2005
Lithium	EPA 6020	Metals	NELAP	5/15/2009
Magnesium	EPA 6010	Metals	NELAP	4/1/2005
Magnesium	EPA 6020	Metals	NELAP	4/1/2005
Manganese	EPA 6010	Metals	NELAP	4/1/2005
Manganese	EPA 6020	Metals	NELAP	4/1/2005
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7471	Metals	NELAP	4/1/2005
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6010	Metals	NELAP	4/1/2005
Molybdenum	EPA 6020	Metals	NELAP	4/1/2005
Naphthalene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Nickel	EPA 6010	Metals	NELAP	4/1/2005
Nickel	EPA 6020	Metals	NELAP	4/1/2005
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Propanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	4/1/2005
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	4/1/2005
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Percent (%) moisture	ASTM D2974-87	General Chemistry	NELAP	5/15/2009
pH	EPA 9040	General Chemistry	NELAP	4/1/2005
pH	EPA 9045	General Chemistry	NELAP	4/1/2005
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phosphorus	EPA 6020	Metals	NELAP	3/19/2012
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	4/1/2005
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Potassium	EPA 6010	Metals	NELAP	4/1/2005
Potassium	EPA 6020	Metals	NELAP	4/1/2005
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 8270	Extractable Organics	NELAP	4/1/2005
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Selenium	EPA 6010	Metals	NELAP	4/1/2005
Selenium	EPA 6020	Metals	NELAP	4/1/2005
Silver	EPA 6010	Metals	NELAP	4/1/2005
Silver	EPA 6020	Metals	NELAP	4/1/2005
Sodium	EPA 6010	Metals	NELAP	4/1/2005
Sodium	EPA 6020	Metals	NELAP	4/1/2005
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Styrene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	4/1/2005
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Thallium	EPA 6010	Metals	NELAP	4/1/2005

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State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Thallium	EPA 6020	Metals	NELAP	4/1/2005
Tin	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toluene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Total cyanide	EPA 9012	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/1/2005
Total nitrogen	TKN + Total nitrate-nitrite	General Chemistry	NELAP	4/1/2005
Total organic carbon	EPA 9060	General Chemistry	NELAP	5/30/2007
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	4/1/2005
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Uranium	EPA 6020	Metals	NELAP	3/19/2012
Vanadium	EPA 6010	Metals	NELAP	4/1/2005
Vanadium	EPA 6020	Metals	NELAP	4/1/2005
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Zinc	EPA 6010	Metals	NELAP	4/1/2005
Zinc	EPA 6020	Metals	NELAP	4/1/2005

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State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	5/30/2007
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	5/30/2007
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Acenaphthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6020	Metals	NELAP	5/30/2007

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State Surgeon General

Laboratory Scope of Accreditation

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Antimony	EPA 6020	Metals	NELAP	5/30/2007
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6020	Metals	NELAP	5/30/2007
Barium	EPA 6020	Metals	NELAP	5/30/2007
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Beryllium	EPA 6020	Metals	NELAP	5/30/2007
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	5/30/2007
Boron	EPA 6020	Metals	NELAP	5/30/2007
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Cadmium	EPA 6020	Metals	NELAP	5/30/2007
Calcium	EPA 6020	Metals	NELAP	5/30/2007
Carbazole	EPA 8270	Extractable Organics	NELAP	5/30/2007
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chromium	EPA 6020	Metals	NELAP	5/30/2007
Chrysene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Cobalt	EPA 6020	Metals	NELAP	5/30/2007
Copper	EPA 6020	Metals	NELAP	5/30/2007
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Fluorene	EPA 8270	Extractable Organics	NELAP	5/30/2007
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	5/30/2007
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Iron	EPA 6020	Metals	NELAP	5/30/2007
Isophorone	EPA 8270	Extractable Organics	NELAP	5/30/2007
Lead	EPA 6020	Metals	NELAP	5/30/2007
Lithium	EPA 6020	Metals	NELAP	5/15/2009
Magnesium	EPA 6020	Metals	NELAP	5/30/2007
Manganese	EPA 6020	Metals	NELAP	5/30/2007
Mercury	EPA 245.6	General Chemistry	NELAP	5/30/2007
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7471	Metals	NELAP	5/30/2007
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6020	Metals	NELAP	5/30/2007
Naphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Nickel	EPA 6020	Metals	NELAP	5/30/2007
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2012** **Expiration Date: 6/30/2013**

Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	5/30/2007
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	5/30/2007
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Phenanthrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Phenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Potassium	EPA 6020	Metals	NELAP	5/30/2007
Pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Selenium	EPA 6020	Metals	NELAP	5/30/2007
Silver	EPA 6020	Metals	NELAP	5/30/2007
Sodium	EPA 6020	Metals	NELAP	5/30/2007
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Thallium	EPA 6020	Metals	NELAP	5/30/2007
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Vanadium	EPA 6020	Metals	NELAP	5/30/2007
Zinc	EPA 6020	Metals	NELAP	5/30/2007

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2012** **Expiration Date: 6/30/2013**

Pace Analytical Services, Inc.
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920 469-2436
Fax: 920 469-8827

STANDARD OPERATING PROCEDURE

DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) SPECTROSCOPY BY 6010C

REFERENCE METHODS: SW-846 METHOD 6010C WITH 3010A AND 3050B

LOCAL SOP NUMBER:	S-GB-M-005-REV.05
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-M-005-REV.04

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/19/12

Kate Grams, Laboratory Quality Manager — Date: 12/7/12

Chad Rusch, Department Manager — Date: 12/07/2012

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 This Standard Operating Procedure (SOP) documents the procedures used by PASI – Wisconsin to determine the concentration of specific metals in environmental water, wipe, paint chips, sludges, soils, and solid samples. The laboratory utilizes the ICP (Inductively Coupled Plasma – Optical Emission Spectrometer) and bases these documented procedures on those listed in EPA SW-846 Method 6010C. Sample preparation procedures are based on SW-846 Methods 3010A and 3050B.

2. SUMMARY OF METHOD

2.1 Samples are digested, excluding filtered groundwater, by heating with appropriate acids and oxidizing agents to solubilize the target elements. Portions of the digestates (or filtered, acidified groundwater samples) are pumped into a nebulizer to produce an aerosol. The aerosol is aspirated into the torch of an Inductively Coupled argon Plasma Optical Emission Spectrometer (ICP-OES) where it is evaporated and decomposed into atoms and ions. The plasma energy causes the target atoms to become excited and, during relaxation, emit characteristic light in the visible and/or ultraviolet emissions. Each element in the sample emits photons at a discrete wavelength(s), which are specific to that element. The light emissions are separated into wavelength and order by passing through a prism and onto an Echelle grating. The signal is then read and quantified by a Charge Injection Device (CID). The intensities of the wavelengths are proportional to the quantity of the target elements that is determined through a comparison to a known concentration (a calibration curve). The signals received from the CID are digitized and relayed to the instrument computer as an analytical signal.

2.2 Background correction may be required to compensate for spectral interferences. Background is measured adjacent to analyte lines at a wavelength selected to be free of spectral interference and which reflects the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a correction would actually degrade the analytical result.

3. SCOPE AND APPLICATION

3.1 This procedure may be used to determine concentrations of trace metals in water, wipe, paint chips, sludges, soils, and solid samples. A list of applicable compounds and Practical Reporting Limits (PRL) for aqueous, solid and TCLP/SPLP/ASTM samples are shown in Table 3.1

Table 3.1 – Elements and associated PRLs for water and leachates

	Element	Wavelength (nm)	PRL Aqueous (ug/L)	PRL Solid* (mg/kg)	PRL TCLP/SPLP/ASTM (mg/L)
Ag	Silver	328.0	10.0	1.0	0.250
Al	Aluminum	396.1	500.0	50.0	2.50
As	Arsenic	189.0	20.0	2.0	0.250
B	Boron	208.9	100.0	10.0	0.500
Ba	Barium	455.4	5.0	0.50	2.50
Be	Beryllium	313.0	4.0	0.40	0.250
Ca	Calcium	317.9	200.0	20	5.000
Cd	Cadmium	228.8	5.0	0.50	0.005
Co	Cobalt	228.6	5.0	0.50	0.250
Cr	Chromium	267.7	5.0	0.50	0.250
Cu	Copper	324.7	10.0	1.0	0.250
Fe	Iron	259.9	100.0	10.0	2.50
K	Potassium	766.4	200.0	20.0	5.000
Mg	Magnesium	279.0	200.0	20.0	5.000
Mn	Manganese	257.6	5.0	0.50	0.250
Mo	Molybdenum	202.0	20.0	2.0	0.250
Na	Sodium	589.5	1000.0	100.0	5.000
Ni	Nickel	231.6	10.0	1.0	0.250
Pb	Lead	220.3	7.5	1.0	0.0075
Sb	Antimony	206.8	20.0	2.0	0.250
Se	Selenium	196.0	20.0	2.0	0.250
Sn	Tin	189.9	50.0	5.0	0.250
Sr	Stronium	407.7	5.0	0.50	0.250
Ti	Titanium	334.9	5.0	0.50	0.250
Tl	Thallium	190.8	40.0	4.0	0.250
V	Vandaium	292.4	5.0	0.50	0.250
Zn	Zinc	206.2	40.0	4.0	0.250

* Based on standard weights/volumes (0.5g/50mL). Final RLs for individual samples may vary depending in actual weights/volumes and percent solids used. Soil includes soils, sludges, paint chips, wipes, and solids. (Unit for wipes are in Total ug.)

Soils are reported on a dry weight basis.

3.2 This procedure is restricted to use by, or under the supervision of, analysts experienced in the digestion of samples for metals analysis and analysis of digestates by ICP. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.3 This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

3.4 Current Method Detection Limits can be found in current LIMS or can be provided by the Quality Department.

4. INTERFERENCES

4.1 Spectral Interferences – Overlap of emission lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light can contribute to spectral interferences. These interferences can typically be minimized by careful selection of quantitation wavelengths, interelement corrections, and background correction.

4.2 Physical Interferences –Changes in sample viscosity, surface tension, or other effects associated with sample transport and nebulization can produce significant inaccuracies, especially in samples containing high concentrations of dissolved solids and acids. Dissolved solids may build up on the nebulizer tip, altering the sample flow rate and causing instrument drift. These effects can be minimized by sample dilution or use of a specially designed high-solids nebulizer. Use of internal standards helps in recognizing sample introduction issues.

4.3 Chemical Interferences – Molecular compound formation, ionization effects, and solute vaporization effects are typically not significant with ICP determinations. If observed, they can be minimized by careful selection of plasma and spectrometer operating parameters.

4.4 Memory Interferences – Sample deposition on the nebulizer tubing, spray chamber, and plasma torch can cause apparent sample carryover. Memory interferences can be minimized by flushing the system with rinse blanks between samples. If memory interference is suspected for a sample, the sample must be re-analyzed after a sufficient rinse period.

4.5 High Salt Concentrations – High salt concentrations in sample digestates can cause signal suppression and confuse interference tests. Use of internal standards helps in recognizing signal suppression

5. SAFETY

5.1 Standards and Reagents

5.2 The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound shall be treated as a potential health hazard. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Special care shall be taken when handling the high-concentration acids and oxidizing reagents used for sample digestion. All digestions must be conducted in a properly functioning fume hood. The use of personal protective equipment (gloves, lab coats and safety glasses) is required.

5.3 Samples

5.3.1 Although sample check-in should be notified of any hazardous samples, samples shall always be considered as “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.

5.4 Equipment

5.4.1 ICP Radio Frequency (RF) Generator – The RF generator used to induce the atomic plasma produces a high energy radio emission. The electrical requirements for this equipment are substantial. The RF generator must only be serviced by those trained specifically for service and repair of the instrument.

5.4.2 ICP RF Coil – The wound coil around the top of the torch produces a high energy, oscillating radio frequency field. The field is substantial and may interfere with surrounding electronics including implanted medical devices. Those individuals with such devices (i.e. pacemakers) must stay clear of this instrument while in operation.

6. DEFINITIONS

6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

6.2 Instrumental Detection Limit (IDL) – The concentration equivalent to a signal, due to the analyte, which is equal to the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day at the same wavelength.

6.3 Instrument Check Standard – A multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. This is the same as the Continuing Calibration Verification Standard (CCV) referred to in the calibration section of this SOP.

6.4 Spectral Interference Check Solution (SIC) – A solution of selected analytes of higher concentrations used to evaluate the procedural routine for correcting known inter-element spectral interferences. This is also known as the Inter-element Correction Solution (ICSA).

6.5 Linear Detection Range – Defined as the upper limit of quantitation for an analyte. The LDR is determined as the upper range limit of an observed signal that is no more than 10% below the level extrapolated from the lower standards.

6.6 Interference Check Sample - (ICSAB) – A solution containing both interfering and analyte elements of known concentration that can be used to verify background and inter-element correction factors.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Collection, Preservation, Storage and Hold Times

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil/ Solid/ Wipe/Paint Chip/Sludge	Pre-cleaned plastic or glass containers. Refer to Pace S-ALL-Q-021, *Sub-Sampling* SOP, for obtaining representative aliquots.	No Preservation	Refrigerated at ≤°C	Up to 6 months
Total Metals –Aqueous	Pre-cleaned plastic containers with HNO_3	Nitric Acid (HNO_3) to pH<2, preserved at time of collection	Room Temperature	Up to 6 months
Dissolved Metals – Aqueous(1)	Pre-cleaned plastic containers – Filter sample at time of collection through a 0.45µm membrane filter.	Nitric Acid (HNO_3) to pH<2, filtered then preserved at time of collection.	Room Temperature	Up to 6 months
TCLP/SPLP/ASTM	Pre-cleaned plastic containers.	Filtered then preserved at time of digestion. -Refrigerate at 4±2 °C until preservation. -Nitric Acid (HNO_3) to pH<2	Refrigerate at 4±2 °C	From Field Collection to Leach Extraction: 180 Days. From Digestion to Determinative Analysis: 180 Days. (Total Elapsed Time: 360 Days).

(1) If filtration cannot be performed in the field, the sample must be taken in a pre-cleaned, unpreserved plastic container and transported to the lab as soon as possible. On arrival at the lab, the sample must be filtered and then preserved to a pH <2. The sample must sit for **24 hours** prior to preparation. This is to ensure that the sample does not have a buffering effect that raises the pH to above <2. The data is qualified if sample preservation fails to bring pH down to ≤2. (Note: TCLP Extracts are not required to sit for 24 hours prior to digestion after the addition of nitric acid.)

7.2 Preservative Check

The pH of samples must be verified to be pH<2 and documented in the Sample Receiving or Metals pH logbook prior to aliquoting for analysis. If a sample has a pH>2, additional preservative must be added to return the sample to <2. Acid volume shall not to exceed 2% of the container capacity. Once adjusted, the sample must be allowed to sit for 24 hours prior to preparation. If the sample is not able to maintain a preservation of a pH of<2, then it must be qualified as such.

8. EQUIPMENT AND SUPPLIES

8.1 Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
iCAP System	Thermo Scientific	ICAP 6500	Serial Number: 20073913
Autosampler	Cetac	ASX-520	Serial Number: 080735A 520
Data System	iTEVA	2.0.039	
Reciculator	Neslab	ThermoFlex900	Serial Number: 107271037
Analytical Balance			

8.2 General Supplies

Item	Vendor (or equivalent)	Model / ID (or equivalent)	Catalog # (or equivalent)	Description
Argon – high purity grade	PraxAir			Bulk
Autosampler Vials	Fisher Scientific	15 mL Polypropylene Test Tubes	14-375-150	
Autosampler Pump Tubing	Analytical West		PT-2180PS	red/red 1.14mm ID – Carrier
			PT-2200SAS	1.30mm ID – Waste
			PT-2080PS-F	orange/orange 0.38mm ID – Internal Standard
Digestion Vials	Environmental Express		SC499	Pre-cleaned polypropylene 70-mL screw cap vials.
Watch Glass	Environmental Express			Polypropylene, to fit 70-mL digestion vial
Heating Block	Environmental Express			Polypropylene, to fit 70-mL digestion vial
Mechanical Pipettes and Tips	Eppendorf			Or equivalent
Filter paper	Whatman		541 Filter Paper	
pH Strips				
Y Connectors	Hewlett-Packard		G1820-65106	
Duo Torch / Duo Ceramic Cone	Thermo Fisher Scientific		8423-120-51241 / 84223-120-51261	
Spray Chamber/Spray Chamber adapter	Thermo Fisher Scientific		8423-120-51411 / 8423-120-51251	
SC-FAST Probe	ESI		SC-5037-3995-150	7” Teflon/Carbon Probe 1.0mmID
SC-FAST Waste Line	ESI		SC-0323-0002-215	Waste Line
SC-FAST Valve	ESI	F7	SC-0599-1024	F7 Valve Head
	ESI		ES-2501-PPF2	
	ESI		ES-2501-PPMZ	
Peri-Pump	ESI			Fluoropolymer union, barbed
Nebulizer Line	ESI		SC-0317-0250-30	
Nuts and Ferrules	ESI		SC-0599-0116-K	For high flow 1/16” tubing
Nuts and Ferrules	ESI		SC-0599-0108-W	For high flow 1/8” tubing
Vacuum Line	ESI		SC-0321-32-215	For SC-FAST high flow valve
Sample Loop	ESI		SC-0318-15	1.5mL sample loop
Nebulizer	ESI		ES-2040	Micro Flow PFA-St nebulizer

8.3 Glassware

Glassware	Vendor / Item # / Description
Volumetric Flasks	Class A
Graduated Cylinders	Class A

9. REAGENTS AND STANDARDS

9.1 Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Nano-pure Water	ASTM Type II, or equivalent (18mOhm or higher resistivity)	Nano-pure Water
Nitric Acid (HNO_3)	Trace metals grade, Fisher Insta-analyzed, or equivalent	J.T Baker Cat #9598-34 or equivalent
Hydrochloric Acid (HCl)	Trace metals grade, Fisher Insta-analyzed, or equivalent	Fisher Cat # A508 or equivalent
Hydrogen Peroxide (H_2O_2)	30% certified reagent grade; trace metals grade if available. Stabilized solutions come with Sodium added.	Fisher Cat # H325-4 or equivalent
Acidified Reagent Water	5mL Nitric Acid and 5mL Hydrochloric Acid per 100mL Nano-pure water	

9.2 Standards Definitions, Storage, and Sources

9.2.1 Definitions

Standard	Description	Comments
Initial Calibration Standard(s)	Standards prepared from single and/or multi-element standard(s) at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range for single point calibration.	Calibration standards can be made and used for 6 months unless a standard used to make them expires earlier.
Initial Calibration Verification Standard	Standard prepared near the midpoint of the calibration range and is used to verify the accuracy of the calibration.	Must be prepared from a source independent of CCV and Calibration Solutions.
Continuing Calibration Verification Standard	Standards prepared from single and/or multi-element standards at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range. This standard verifies the accuracy of the calibration curve.	May be prepared from the same source as the calibrations standards
Reporting Limit Verification Standard (RLVS)	Must be performed after calibration and prior to samples and at the end of the analytical sequence. The recovery must be between 70 and 130% to report sample data.	NELAC requirement, a client specific requirement for certain QAPPs
Inter-element correction solution (ICSA)	A solution prepared with high concentrations of interfering elements (Al, Ca, Fe, Mg...). This is run daily at a minimum to verify the inter-element correction factors.	Al, Ca, Fe, Mg
Inter-element correction solution (ICSAB)	A solution prepared with high concentrations of interfering elements and low level concentrations of the other target elements. This is analyzed daily and immediately following the ICSA solution.	Not a 6010C requirement but may be required on a client specific basis.
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard solution contains a non-target element (yttrium (Y)) at a concentration yielding an appropriate final concentration in the sample.	Y at 5ppm with Li. Added in-line to all samples and standards.
Single Element Standards	Stock standards purchased from venders containing one element. Used for checking IECs and may be used for checking linear ranges.	Must be 99.99% pure.
Spiking Standard	This solution contains all target analytes and should not be prepared from the same standards as the calibration standards.	

9.2.2 Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	▪ Concentrated reference solution purchased directly from approved vendor	▪ Manufacturer's recommended expiration date for unopened standards.	▪ Manufacturer's recommended storage conditions ▪ When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	▪ Reference solutions prepared by dilutions of the stock solution	▪ Should be prepared as needed or at least every 6 months. The intermediate or working standard can not exceed the expiration date recommended by the manufacturer for each component used.	▪ Store at room temperature

9.3 Standard Sources

9.3.1 Standards are prepared from commercially available multi-compound stock solutions. The sources of the stock solutions, formulas for preparing dilutions and working standards, and concentrations for all compounds are presented in tables within section 9.3. All intermediate and working standards are prepared using acidified Nano-pure water and used immediately or stored for no longer than 6 months.

9.3.2 Analytical Working Standard Preparations - Depending on the volume of each solution needed, the standards are brought to volume in class A volumetric flasks with Nano-pure water unless otherwise noted.

Table 9.3.1 – Calibration Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	2 ml	10 mL HNO3 / 10 mL HCl	200 ml	1.0 ppm
	Ba	100 ppm				1.0 ppm
	Be	100 ppm				1.0 ppm
	Cd	100 ppm				1.0 ppm
	Co	100 ppm				1.0 ppm
	Cr	100 ppm				1.0 ppm
	Cu	100 ppm				1.0 ppm
	Mn	100 ppm				1.0 ppm
	Ni	100 ppm				1.0 ppm
	Pb	100 ppm				1.0 ppm
	Se	100 ppm				1.0 ppm
	Sr	100 ppm				1.0 ppm
	Tl	100 ppm				1.0 ppm
	V	100 ppm				1.0 ppm
	Zn	100 ppm				1.0 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	4 ml			2.0 ppm
	Mo	100 ppm				2.0 ppm
	Sb	100 ppm				2.0 ppm
	Sn	100 ppm				2.0 ppm
	Ti	100 ppm				2.0 ppm
	Ag	50 ppm				1.0 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	10 ml			50 ppm
	Ca	1000 ppm				50 ppm
	Fe	1000 ppm				50 ppm
	K	1000 ppm				50 ppm
	Mg	1000 ppm				50 ppm
	Na	1000 ppm				50 ppm

Table 9.3.2 – CCV Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	5.0 ml	50 mL HNO3/ 50 mL HCl	1000 ml	0.5 ppm
	Ba	100 ppm				0.5 ppm
	Be	100 ppm				0.5 ppm
	Cd	100 ppm				0.5 ppm
	Co	100 ppm				0.5 ppm
	Cr	100 ppm				0.5 ppm
	Cu	100 ppm				0.5 ppm
	Mn	100 ppm				0.5 ppm
	Ni	100 ppm				0.5 ppm
	Pb	100 ppm				0.5 ppm
	Se	100 ppm				0.5 ppm
	Sr	100 ppm				0.5 ppm
	Tl	100 ppm				0.5 ppm
	V	100 ppm				0.5 ppm
	Zn	100 ppm				0.5 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	10.0 ml			1.0 ppm
	Mo	100 ppm				1.0 ppm
	Sb	100 ppm				1.0 ppm
	Sn	100 ppm				1.0 ppm
	Ti	100 ppm				1.0 ppm
	Ag	50 ppm				0.5 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	25.0 ml			25 ppm
	Ca	1000 ppm				25 ppm
	Fe	1000 ppm				25 ppm
	K	1000 ppm				25 ppm
	Mg	1000 ppm				25 ppm
	Na	1000 ppm				25 ppm

Table 9.3.3 – ICV (Second Source) Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
IV #1 (PA-STD-1B)	As	200 ppm	0.5 ml	10 mL HNO3/ 10 mL HCl	200 ml	0.5 ppm
	Ba	200 ppm				0.5 ppm
	Be	200 ppm				0.5 ppm
	Cd	200 ppm				0.5 ppm
	Co	200 ppm				0.5 ppm
	Cr	200 ppm				0.5 ppm
	Cu	200 ppm				0.5 ppm
	Mn	200 ppm				0.5 ppm
	Ni	200 ppm				0.5 ppm
	Pb	200 ppm				0.5 ppm
	Se	200 ppm				0.5 ppm
	Sr	200 ppm				0.5 ppm
	Tl	200 ppm				0.5 ppm
	V	200 ppm				0.5 ppm
	Zn	200 ppm				0.5 ppm
IV #2 (PA-STD-2B)	B	200 ppm	1.0 ml			1.0 ppm
	Mo	200 ppm				1.0 ppm
	Sb	200 ppm				1.0 ppm
	Sn	200 ppm				1.0 ppm
	Ti	200 ppm				1.0 ppm
	Ag	100 ppm				0.5 ppm
IV #3 (PA-STD-3B)	Al	2000 ppm	2.5 ml			25 ppm
	Ca	2000 ppm				25 ppm
	Fe	2000 ppm				25 ppm
	K	2000 ppm				25 ppm
	Mg	2000 ppm				25 ppm
	Na	2000 ppm				25 ppm

Table 9.3.4 – Spiking Standard (for LCS and MS/MSD) Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)	Spike Concentration *
IV #1 (PA-STD-1B)	As	200 ppm	25 ml	12 mL HNO3	200 ml	50 ppm	500 ppb
	Ba	200 ppm				50 ppm	500 ppb
	Be	200 ppm				50 ppm	500 ppb
	Cd	200 ppm				50 ppm	500 ppb
	Co	200 ppm				50 ppm	500 ppb
	Cr	200 ppm				50 ppm	500 ppb
	Cu	200 ppm				50 ppm	500 ppb
	Mn	200 ppm				50 ppm	500 ppb
	Ni	200 ppm				50 ppm	500 ppb
	Pb	200 ppm				50 ppm	500 ppb
	Se	200 ppm				50 ppm	500 ppb
	Sr	200 ppm				50 ppm	500 ppb
	Tl	200 ppm				50 ppm	500 ppb
	V	200 ppm				50 ppm	500 ppb
	Zn	200 ppm				50 ppm	500 ppb
IV #2 (PA-STD-2B)	B	200 ppm	25 ml			50 ppm	500 ppb
	Mo	200 ppm				50 ppm	500 ppb
	Sb	200 ppm				50 ppm	500 ppb
	Sn	200 ppm				50 ppm	500 ppb
	Ti	200 ppm				50 ppm	500 ppb
	Ag	100 ppm				25 ppm	250 ppb
IV #3 (PA-STD-3B)	Al	2000 ppm	25 ml			500 ppm	5000 ppb
	Ca	2000 ppm				500 ppm	5000 ppb
	Fe	2000 ppm				500 ppm	5000 ppb
	K	2000 ppm				500 ppm	5000 ppb
	Mg	2000 ppm				500 ppm	5000 ppb
	Na	2000 ppm				500 ppm	5000 ppb

*** Use 1.0 ml of spiking std to 50 ml**

Table 9.3.5 – Low Level Check Standard (CRDL) Intermediate

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)*	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppb)
4400-100118CS01	Ag	10 ppm	10 ml	5 mL HNO3 / 5 mL HCl	100 ml	1,000 ppb
	Al	500 ppm				50,000 ppb
	As	20 ppm				2,000 ppb
	B	100 ppm				10,000 ppb
	Ba	5 ppm				500 ppb
	Be	4 ppm				400 ppb
	Ca	200 ppm				20,000 ppb
	Cd	5 ppm				500 ppb
	Co	5 ppm				500 ppb
	Cr	5 ppm				500 ppb
	Cu	10 ppm				1,000 ppb
	Fe	100 ppm				10,000 ppb
	K	1000 ppm				1000,000 ppb
	Mg	200 ppm				20,000 ppb
	Mn	5 ppm				500 ppb
	Mo	20 ppm				2,000 ppb
	Na	1000 ppm				100,000 ppb
	Ni	10 ppm				1,000 ppb
	Pb	7.5 ppm				750 ppb
	Sb	20 ppm				2,000 ppb
	Se	20 ppm				2,000 ppb
	Sn	50 ppm				5,000 ppb
	Sr	5 ppm				500 ppb
	Ti	5 ppm				500 ppb
	Tl	40 ppm				4,000 ppb
	V	5 ppm				500 ppb
	Zn	40 ppm				4,000 ppb

Table 9.3.5b – Low Level Check Standard (CRDL) Working Solution

Low Level Check Standard Intermediate	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)*	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppb)
CRDL Intermediate (In Table 9.3.5)	Ag	1,000 ppb	2 ml	10 mL HNO3 / 10 mL HCl	200 ml	10 ppb
	Al	50,000 ppb				500 ppb
	As	2,000 ppb				20 ppb
	B	10,000 ppb				100 ppb
	Ba	500 ppb				5 ppb
	Be	400 ppb				4 ppb
	Ca	20,000 ppb				200 ppb
	Cd	500 ppb				5 ppb
	Co	500 ppb				5 ppb
	Cr	500 ppb				5 ppb
	Cu	1,000 ppb				10 ppb
	Fe	10,000 ppb				100 ppb
	K	100,000 ppb				1000 ppb
	Mg	20,000 ppb				200 ppb
	Mn	500 ppb				5 ppb
	Mo	2,000 ppb				20 ppb
	Na	100,000 ppb				1000 ppb
	Ni	1,000 ppb				10 ppb
	Pb	750 ppb				7.5 ppb
	Sb	2,000 ppb				20 ppb
	Se	2,000 ppb				20 ppb
	Sn	5,000 ppb				50 ppb
	Sr	500 ppb				5 ppb
	Ti	500 ppb				5 ppb
	Tl	4,000 ppb				40 ppb
	V	500 ppb				5 ppb
	Zn	4,000 ppb				40 ppb

Table 9.3.6– Inter-element Correction Solution (ICSA) Working Dilutions and Concentrations

Pace Standard #	Vendor	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (mg/L)	Stock Concentration (µg/mL)
Single Element Al	Ultra Scientific	ICP-113	10	200	Al	500	10000
Single Element Ca	Ultra Scientific	ICP-120	10	200	Ca	500	10000
Single Element Mg	Ultra Scientific	ICP-112	10	200	Mg	500	10000
Single Element Fe	Ultra Scientific	ICP-126	4	200	Fe	200	10000

Table 9.3.6b– Inter-element Correction Solution (ICSAB) Working Dilutions and Conc. – (As needed).

Pace Standard #	Vendor	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (µg/mL)	Stock Concentration (µg/mL)
Single Element Al	Ultra Scientific	ICP-113	9.5	200	Al	475	10000
Single Element Ca	Ultra Scientific	ICP-120	9.5	200	Ca	475	10000
Single Element Mg	Ultra Scientific	ICP-112	9.5	200	Mg	475	10000
Single Element Fe	Ultra Scientific	ICP-126	3.5	200	Fe	175	10000
IV #1 (PA-STD-1B)	Inorganic Ventures	PA-STD-1B	0.5	200	As	0.5	200 ppm
					Ba	0.5	200 ppm
					Be	0.5	200 ppm
					Cd	0.5	200 ppm
					Co	0.5	200 ppm
					Cr	0.5	200 ppm
					Cu	0.5	200 ppm
					Mn	0.5	200 ppm
					Ni	0.5	200 ppm
					Pb	0.5	200 ppm
					Se	0.5	200 ppm
					Sr	0.5	200 ppm
					Tl	0.5	200 ppm
					V	0.5	200 ppm
					Zn	0.5	200 ppm
IV #2 (PA-STD-2B)	Inorganic Ventures	PA-STD-2B	1.0	200	B	1.0	200 ppm
					Mo	1.0	200 ppm
					Sb	1.0	200 ppm
					Sn	1.0	200 ppm
					Ti	1.0	200 ppm
					Ag	0.5	100 ppm
IV #3 (PA-STD-3B)	Inorganic Ventures	PA-STD-3B	2.5	200	Al	25	2000 ppm
					Ca	25	2000 ppm
					Fe	25	2000 ppm
					K	25	2000 ppm
					Mg	25	2000 ppm
					Na	25	2000 ppm
Single Element Ni	Ultra Scientific	ICP-028	0.1	200	Ni	1.0	1000 ppm

Table 9.3.7– Internal Standard Solution Working Dilutions and Concentrations

Pace Standard #	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (mg/L)	Stock Concentration (mg/L)
IS	ICP69N-1 (Accustandard)	5	1000	Yttrium	5 mg/L	1000
	ICP-103-5 (Ultra Scientific)	100	1000	Lithium	1000 mg/L	10000

10. CALIBRATION

10.1 Profile

The iCAP 6500 ICP will automatically check a carbon reference line against ambient air conditions each time the plasma is ignited to maintain wavelength accuracy.

10.2 Daily Initial Calibration

Allow the instrument 30 minutes to warm up after igniting the torch. The ICP must be calibrated each time it is set up for analysis or every 24 hours according to the manufacturer's instructions. Calibration requires analysis of a Calibration Blank and at least one level of calibration solution. Calibration standards can be made and used for 6 months unless a standard used to make them expires earlier. It would then expire on that date. Instrument standardization date and time must be recorded in the raw data. See Table 9.3.1 for details on the preparation of the Initial Calibration Standard.

10.3 Calibration Verification

10.3.1 Initial Calibration Verification (ICV) / Second Source

To assess the accuracy relative to the purity of the calibration standards, a standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as **Secondary Source Verification** or, alternatively as **Initial Calibration Verification**. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. See the most current version of SOP S-GB-Q-026 *Standard and Reagent Management and Traceability* for more information of standards. This standard must be analyzed immediately after the calibration and before any batch QC or client samples. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{ICV} - \text{TrueValue}_{ICV}] / \text{TrueValue}_{ICV} * 100$$

See Table 9.3.3 for details on the preparation of this standard. See Table 10.1 for control criteria

10.3.2 Reporting Limit Verification Standard (CRDL)

With every Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) must also be analyzed and meet established acceptance criteria. The CRDL is analyzed prior to any samples being analyzed and at the end of the analytical sequence. Additional CRDLs may be analyzed throughout the analytical sequence at the analyst's discretion. The limits for the CRDL are +/- 30% of the true concentration. See Table 9.3.5 for details on the preparation of this standard.

10.3.3 ICSA (Interference Check Standard) Verification

With every Initial Calibration, the inter-element check solution standard must be run to verify the inter-element correction factors. At a minimum, the ICSA standard must be analyzed at the beginning of the run, following the initial calibration and evaluated against the criteria in Table 10.1. If client specific requirements dictate that the ICSA must be analyzed at both the beginning and end of the analysis, the ICSA may be analyzed immediately after the completion of those specific samples and is not required to be placed at the very end of the sequence. If client specific requirements dictate that the ICSAB be run, the ICSAB must be analyzed immediately following the ICSA and be held to the criteria in Table 10.1. See Table 9.3.6 and 9.3.6b for details on preparation of these standards.

10.3.4 Continuing Calibration Verification (CCV)

As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as **Continuing Calibration Verification**. The validity of the initial calibration is checked after every 10 samples and at the end of an analytical sequence by analyzing a midpoint calibration standard (CCV).

The values obtained from the analysis of the CCV are compared to the true values and a percent difference calculated according to the formula in Section 10.3.1. The percent difference must meet the method specified criteria in Table 10.1 for the analysis to proceed. See Table 9.3.2 on the preparation of this standard.

Table 10.1 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression for elements requiring multiple calibration levels	Correlation coefficient ≥ 0.998	If not met, remake standards and recalibrate. One point calibration is standard for the Wisconsin laboratory.
Initial Calibration Verification Standard (Second Source)	Immediately after each initial calibration	≤±10% of true value The % RSD between replicate integrations of an ICV std must be <5% (min of 2 integrations).	If 4 or more replicates are used for analysis the RSD must be ≤ 3%
Calibration Blank	Immediately after each initial calibration (ICB), every 10 samples (CCB), and at the end of the analytical sequence	≤ PRL	If criteria are not met, follow the corrective actions in 10.4.2. If the concentration in the blank is outside the control limit and <1/10 the concentration in the sample, the sample result may be reported.
CRDL Standard	Immediately after verification and at the end of the analytical sequence.	Each element tested must be at least between 70-130% of the PRL value.	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.4.
ICSA	Once per ICAL, immediately after verification	Results for non-interference elements = ND ± 2x RL Recovery of interfering elements must be 80-120% of true value	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.4.5
ICSAB	Once per ICAL, immediately after ICSA if requested by the client.	Results for non-interference elements = True Value ± 20% (or 2xRL, whichever is greater)	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.4.5
Continuing Calibration Verification	After every 10 samples and at the end of analytical sequence	≤±10% of true value The % RSD between replicate integrations of a CCV std must be <5% (min of 2 integrations).	If criteria not met, follow corrective actions in 10.4.6

10.4 Calibration Corrective Actions

10.4.1 Calibration Linearity Problems

10.4.1.1 Reanalyze the original calibration standards to determine instrument consistency.
10.4.1.2 Prepare and analyze a new calibration standard to determine preparation consistency / standard integrity.
10.4.1.3 Perform instrument maintenance, document in instrument log book
10.4.1.4 Recalibrate instrument according to procedures in Section 10.

10.4.2 Calibration Blank Problems

10.4.2.1 Reanalyze the original CB standard to determine instrument consistency.
10.4.2.2 Prepare and analyze a new CB standard to determine preparation consistency / standard integrity. Note: If you run two CB, only one may be used for reporting purposes.
10.4.2.3 Perform instrument maintenance, document in instrument log book
10.4.2.4 Recalibrate instrument according to procedures in Section 10.
10.4.2.5 Reanalyze any samples not bracketed by passing CBs.
10.4.2.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing CB that exceeded criteria may be reported if 10X concentration in the blank is less than the reported result.
Samples analyzed with a bracketing CB that exceeded criteria may be reported if the sample result is a non-detect.

10.4.3 Initial Calibration Verification (Secondary Source) Problems

10.4.3.1 Reanalyze the original ICV standard to determine instrument consistency.
10.4.3.2 Prepare and analyze a new ICV standard to determine preparation consistency / standard integrity.
10.4.3.3 Perform instrument maintenance, document in instrument log book
10.4.3.4 Recalibrate instrument according to procedures in Section 10.
10.4.3.5 Reanalyze any samples not bracketed by passing ICVs.
10.4.3.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing ICV that exceeded criteria due to an increase in response may be reported if the target analytes were not detected in the samples.

10.4.4 CRDL Standard Problems

10.4.4.1 Check CAL0 – Elevated counts resulting from contamination may cause the CRDL to fail low.
10.4.4.2 Reanalyze the original CRDL standard to determine instrument consistency.
10.4.4.3 Prepare and analyze a new CRDL standard to determine preparation consistency / standard integrity.
10.4.4.4 Perform instrument maintenance, document in instrument log book
10.4.4.5 Recalibrate instrument according to procedures in Section 10.

10.4.4.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with CRDL that is outside criteria may be footnoted or the CRDL for the reported sample adjusted.

10.4.5 ICSA/ICSAB Standard Problems

10.4.5.1 Reanalyze the original **ICSA/ICSAB** standard to determine instrument consistency.
10.4.5.2 Prepare and analyze a new **ICSA/ICSAB** standard to determine preparation consistency / standard integrity.
10.4.5.3 Adjust IEC and rerun **ICSA/ICSAB.** To update IECs, follow the instruction below.
10.4.5.4 The Inter-element Correction Factors (IEC's) are used to correct for false signals (positive and negative) produced when peaks overlap in the spectra.
10.4.5.5 To set up Initial IEC, run single element standards with only background correction points in place. The standards are run at the linear range.

The following calculation is done:

$$\frac{\text{Element result (ug/L)}}{\text{Interfering element result (ug/L)}} = \text{IEC}$$

10.4.5.6 This ratio is entered in the IEC table in the method file. Type the ratio under the k1 header in the appropriate row. Enter a negative before the ratio if it is a negative interference.
10.4.5.6.1 Perform instrument maintenance, document in instrument log book
10.4.5.6.2 Recalibrate instrument according to procedures in Section 10.

10.4.6 Continuing Verification Problems

10.4.6.1 Reanalyze the original CCV standard to determine instrument consistency.
10.4.6.2 Prepare and analyze a new CCV standard to determine preparation consistency / standard integrity.
10.4.6.3 Perform instrument maintenance, document in instrument log book
10.4.6.4 Recalibrate instrument according to procedures in Section 10.
10.4.6.5 Reanalyze any samples not bracketed by passing CCVs.
10.4.6.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing CCV that exceeded criteria due to an increase in response may be reported if the target analytes were not detected in the samples.

11. PROCEDURES

11.1 Aqueous Sample Preparation for Total or Dissolved Metals by 3010A – Hot Block Digestion

11.1.1 Groundwater samples for dissolved metals may be analyzed without digestion. All other water samples must be digested.

11.1.2 Aqueous samples for total or dissolved metals (if visual precipitation) may be prepared according to EPA 3010A. Samples for dissolved metals must be filtered prior to preservation. Once preserved, samples must be allowed to sit for 24 hours prior to preparation. TCLP/SPLP/ASTM extracts do not require to sit for 24 hours prior to digestion.

11.1.3 Verify that the hood is turned on. Turn on the Hot Block and set the temperature so that samples on the block maintain a temperature of 95 +/- 5ºC. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the sample temperature in the sample preparation logbook. The well position should be rotated on a per batch basis to ensure that the block temperature is verified across the block.

11.1.4 Place a 50mL well-mixed aliquot of each sample in digestion vials. Place 50 mL of Nano-pure water into digestion vials for each MB and LCS (one per 20 samples). Prepare the MS and MSD by placing 50mL aliquots of selected sample from the batch into digestion vials. One MS and one duplicate or MSD must be prepared for each analytical batch of 20 samples or less. Additionally, one MS must be prepared for each TCLP/SPLP/ASTM sample matrix digested in a digestion batch.

11.1.5 Spike the LCS, MS, and MSD with 1.0mL of the spiking solution.

11.1.6 Carefully add the digestion reagents to each sample, MB, LCS, MS, and MSD vial.

Digestion Program for all Aqueous Samples, EPA Method 3010A

Steps	Reagents[1]	Actions
1	5.0mL concentrated HNO_3, 2.5 mL HCl	Heat at 95 +/- 5ºC °C for 4 hours
2		Remove digestates from Hot Block and allow to cool in the hood.
3		Adjust the final volume of each vial to 50 mL by diluting with 18 MOhm Nano-Pure water. Place on screw cap and shake thoroughly.
4		Centrifuge, let settle, or filter sample only if there is concern that insoluble materials may clog the nebulizer. Filtering is liable to cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and pre-rinsed with dilute HNO_3. If filtration is required, filter all QC samples as well.

1. Acid ratios must be maintained to match method. Those specified are based on a 50mL sample volume

Note: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the samples and re-prep.

11.1.7 Record the lot numbers of all reagents and materials in the digestion logbook. This includes the lot numbers of the digestion tubes, filters or filter paper and any additional materials in contact with the sample matrices.

11.2 Soil/ Solid/Sludge/Paint Chip/Wipe Sample Preparation by 3050B

11.2.1 Soil, sludges, and solids are prepared according to Method 3050B. Wipes and Paint Chips are also prepared using 3050B.

11.2.2 Verify that the hood is turned on. Turn on the Hot Block and set the temperature to 95±5ºC. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the sample well ID and temperature in the sample logbook. This well measurement should be rotated on a per batch basis to ensure that the block temperature is consistent across the block.

11.2.3 To prepare Paint Chips for digestion, transfer paint chips into labeled drying tins. Dry samples at 103-105 degrees Celsius for 12-24 hours. Remove samples from oven and desiccate for 1 hour.

11.2.4 If possible, mix each sample thoroughly to homogenize. If homogenization is not feasible, remove the top layer of the sample from the container and discard. Remove a sample aliquot in the range of 0.5 – 0.599 g for analysis (0.5g dry sample). Weigh the aliquot to the nearest 0.01g and transfer to a digestion vial. Refer to Pace S-ALL-Q-021, *Sub-Sampling* SOP, for more details. For paint chips, Weigh out 0.5 gram of sample into a digestion tube. If the sample available is less than 0.5 gram, use the entire sample. For Wipes, digest the WHOLE wipe in the digestion vial. For sludges with % solids >10% and <50%, weigh out 2.5-5 grams.

11.2.5 Prepare the MB and LCS by adding 2.0 mL of Nano-pure water into digestion vials containing 0.500-0.504g of Teflon chips. The weight is documented as 0.500g so as to not bias the results. Prepare the duplicate, MS and MSD by placing aliquots of a selected sample from the batch into digestion vials as described for environmental samples. Spike the LCS, MS, and MSD with 1.0 mL spiking solution.

11.2.6 Carefully add the digestion reagents to each sample, MB, LCS, MS, and MSD vial and heat them with the Hot Block at 95±5ºC according to the following digestion program.

11.2.7 MS/MSDs are not performed on paint chips, because the sample concentration is frequently excessively high and dwarfs any spike. As a result an LCSD is performed for paint chip batches.

Digestion Program for Soil/ Solid samples by method 3050B

Steps	Reagents[1]	Actions
1	Nano-Pure Water	Add Appoximately 2.0 mLs of water to all vessels in the prep batch.
2	10.0 mL 1:1 HNO3	Cover with watch glass and heat without boiling for 2 hours. Remove from heat and cool.
3	2.5 mL 30% H_2O_2	Cover with watch glass and heat on Hot Block for 15 minutes or until effervescence subsides. Warming will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Remove and allow to cool.
4	2.5mL concentrated HCl	Reflux on Hot Block until reaction is complete (no brown fumes) without boiling. Allow samples to cool.
5		Dilute sample to 50-mL final volume with 18 MOhm Nano-Pure water. Place screw cap on vials, shake, allow sample to settle, centrifuge at 2000-25000 rpm for 10 minutes, OR and filter if necessary.

1. Acid ratios must be maintained to match method. Those specified are based on a 0.5g sample amount
2. In effect, this produces the 1:1 nitric acid (required by method 3050) in the actual digestion cup.

11.2.8 Record the lot numbers of all reagents and materials in the digestion logbook. This includes the lot numbers of the digestion tubes, filters or filter paper and any additional materials in contact with the sample matrices.

11.3 ICP System Preparation

11.3.1 Preventative Maintenance

Inspect the sample introduction system including the nebulizer, torch, injector tube and up-take tubing for salt deposits, dirt and debris that could restrict solution flow and affect instrument performance. Document all maintenance in ICP maintenance logbook.

See Attachment I for troubleshooting guide.

Operating Parameters

Set up and configure the ICP according to manufacturer's operating instructions using operating parameters shown in the following table.

Table 11.3.1 - Operating Parameter for ICP Systems

Operating Parameter	ICAP 6500 20073913
Argon Flow	80 L/min
Nebulizer Pressure	0.55 L/min
RF Output	1175 W
Pump Speed	15 rpm
Aux Gas Flow	0.5 L/min
Coolant Gas Flow	15 l/min

11.3.2 Calibrate ICP according to Section 10.

11.4 Sample Analysis

11.4.1 Batch Sequence

If using an autosampler, enter an autosampler sequence into the ICP data system per manufacturer's instuctions. Calibration blanks, standards, initial checks, and interlement correction standards shall be run before analysis of environmental samples. All samples, including calibration, checks, and other standards, can also be run manually.

Table 11.4.1 – Example analytical sequence after calibration of a blank and a standard.

Autosampler Position	Sample Description	Autosampler Position	Sample Description
1	ICV	27	Sample 15
2	ICB	28	Sample 16
3	CRDL	29	CCV
4	ICSA *	30	CCB
5	CCV	31	Sample 17
6	CCB	32	Sample 18
7	MB1	33	Sample 19
8	LCS1	34	Sample 20
9	Sample 1	35	MB2
10	Sample 2	36	LCS2
11	Sample 3	37	Sample 21
12	Sample 3 Matrix Spike	38	Sample 22
13	Sample 3 Matrix Spike Duplicate	39	Sample 23
14	Sample 4	40	Sample 24
15	Sample 5	41	CCV
16	Sample 6	42	CCB
17	CCV	43	Sample 25
18	CCB	44	Sample 26
19	Sample 7	45	Sample 27
20	Sample 8	46	Sample 28
21	Sample 9	47	Sample 28 Matrix Spike
22	Sample 10	48	Sample 28 Matrix Spike Duplicate
23	Sample 11	49	Sample 29
24	Sample 12	50	CRDL
25	Sample 13	51	CCV
26	Sample 14	52	CCB

* ICSAB will follow the ICSA if needed/requested by client.

Load Autosampler

11.4.2.1 Load the autosampler tray according to the vial position on the sequence table.

11.5 Sample Analysis

11.5.1 Analyze all standards, quality control samples, and environmental samples.

11.6 Data Reduction

11.6.1 **Quantitative Analysis –** The instrument produces results in ug/L. If the initial sample aliquot and final digestate volumes are the same, the ICP data system will calculate the concentration of each element directly. If the initial and final volumes are different, the actual values are posted into the LIMS system and calculations are performed in the LIMS.

11.6.1.1 Water Samples – Since initial sample aliquot and final digestate volumes are the same, the iCAP analyzer data system will calculate the concentration directly. No further calculations are necessary unless the sample was diluted.

11.6.1.2 Soil/Solid/Paint Chip/Sludge Samples -

$$\text{Final Result (mg/kg dry weight corrected)} = \frac{\text{Raw data result (ug/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{Sample weight (g)} * \text{Dry weight for soil/solid (decimal form)}}$$

11.6.1.3 Wipes

$$\text{Final Result (Total ug)} = \frac{\text{Raw data result (ug/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{1 wipe}}$$

11.6.2 Samples with analyte concentrations that exceed 90% of the upper linear range must be diluted and re-analyzed.

11.6.3 Any sample in which any element value is below the instrument detection limit or PRL must be reported as less than the PRL (or ND) for that specific element. Do not report data below the element PRL concentration unless it is qualified as an "estimated" result with a "J" or "B" data qualifier.

11.6.4 Metals Analytical Review and Reporting System (ADAMS) or LimsLink is used to transfer data from the instrument to the LIMS system.

12. QUALITY CONTROL

12.1 Refer to the most current version of the Pace Quality Manual Appendix I Quality Control Calculations and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

12.2 Instrument Quality Control

12.3 Refer to Table 10.1 for initial and continuing calibration criteria and corrective actions. This includes ICV, CCV, CB, RLVS, and ICSA standard checks.

12.4 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Nano-Pure water	One per 20 samples	1) Target analytes must be less than the RL.	1) Re-analyze blank to confirm failure. 2) Qualify results and / or re-extract associated samples. **Exceptions:** 1) If sample ND, report sample without qualification 2) If sample result >10x MB detects no qualification is needed. 3) If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Nano-Pure Water and Spiking solution (see table 9.3.4 for elements)	One per 20 samples	Laboratory Calculated Limits or default limits of 80-120% Should not exceed 80-120%	1) Reanalyze the LCS to verify failure 2) If LCS passes, review samples for potential problems 3) If problem persists, check spike solution 4) Re-digest and re-analyze samples where possible **Exceptions:** 1) If LCS rec > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Environmental Sample and Spiking solution (see table 9.3.4 for elements)	One per 20 samples. For leached samples one per sample matrix (soil, and wood chips would each require a separate matrix spike).	Laboratory Calculated Limits or default limits of 75-125% Should not exceed 75-125%	1) If LCS and MBs are acceptable, the MS may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate OR (alternative) Sample Dup	A duplicate sample OR MS/MSD pair must be analyzed per batch of 20 samples.	Laboratory Derived Limits for RPD or default limits of 20%. Recovery criteria should not exceed 75-125%.	1) Results not meeting criteria must be footnoted on client report.

12.5 Dilution Test – A dilution test is recommended but not required for method 6010C. A dilution test is run only at client request.

12.5.1 Perform a 5X dilution on one sample per preparation batch. Diluted and undiluted sample results should agree within 10% of each other if the sample contains analyte concentrations at least 50 times the PRL. If agreement between diluted and undiluted samples meeting the concentration requirements is >10%, then matrix effect should be suspected and samples footnoted appropriately.

12.6 Post-digestion Spike Test or Analyte Addition – Post digestion spikes are recommended but not required for method 6010C. A post spike is run only at client request.

12.6.1 Spike one digestate from a preparation batch to achieve the same analyte addition level as found in the LCS. The acceptable recovery range is 80-120%. If outside this range, matrix effects should be suspected and samples annotated.

12.7 **Internal Standards –** Yttrium is used as an internal standard. Yttrium is not calibrated for. Precision of the Yttrium reps are evaluated using < or = 5% RSD. Accuracy of the Yttrium average count levels are evaluated using 50-150% recovery as compared to the calibration blank. If either of these criteria is not meet, the sample or quality control standard must be rerun, possibly at a dilution.

12.8 **Multiple Instrument Integration RSD** – The RSD is required to be evaluated between multiple instrument integrations. The RSD must be less than or equal to 20% if the analyte concentration is greater than the reporting limit. If the RSD is greater than 20%, the laboratory must reanalyze the sample.

13. METHOD PERFORMANCE

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted annually per S-GB-Q-020, *Determination of LOD and LOQ* per instrument.

13.2 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures*.

13.3 **Linear Dynamic Range Study**

13.3.1 It is a Pace best practice to perform this linear dynamic range study determination once, keep the data, and then annually or semi-annually verify with a single high point. If additional range is to be considered, the laboratory must perform the study as indicated below.

13.3.2 A linear dynamic range study must be conducted at least annually. The study is conducted for each element by analyzing increasing concentrations (at least 3 levels) until the results generated exceed ±10% difference from the true value. The highest concentration within the 10% criteria is the maximum of the linear range for that element.

13.4 **Instrument Detection Limits (IDLs)** in ug/L are estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined at least quarterly.

13.5 **Lower limit of quantitation check sample (LLQC)**

The LLQC samples should be analyzed after establishing the PRLs, and on an as needed basis to demonstrate the desired the detection capability. The LLQC sample is carried through the entire preparation and analytical procedure. Acceptance criteria are ±30% of the true value.

13.6 Method Modifications

13.6.1 Method modifications for EPA method 6010C are as follows:

13.6.2 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.

13.6.3 All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.

13.6.4 Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories

13.6.5 When insufficient volume for the method specified matrix spike/matrix spike duplicate (MS/MSD) is not provided by the client, a laboratory control spike duplicate will be analyzed to demonstrate precision criteria. Laboratory batches will be qualified with the appropriate "M5" data qualifier. When performing this analysis on paint chip samples, a MS/MSD will not be completed on the samples due to high levels of elements present in the native sample.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management*.

14.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. REFERENCES

15.1 USEPA, SW-846, Method 6010C Revision 3, "Inductively Coupled Plasma Atomic Emission Spectrometry", February 2007.

15.2 USEPA, SW-846, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectrometry", December 1996.

15.3 USEPA, SW-846, Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils", December 1996.

16. TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS, APPENDICES, ETC.

16.1 **Attachment I** - ICP Troubleshooting Guide

16.2 **Attachment II** – ICP Flowchart

17. REVISIONS

Document Number	Reason for Change	Date
S-ALL-GB-M-005-REV.00	First Issue	22Sept2008
S-GB-M-005-REV.01	Update Signature Page	26May2009
S-GB-M-005-Rev.02	Updated Signature Page Table 3.1: Updated Pb PRL to 7.5ug/L Sections 9.2.1 and 10.2: Added that calibration solutions can be used until manufacturer's expiration date Section 11: Changed HNO3 to 5mL from 3mL.	28Apr2010
S-GB-M-005-Rev.03	Body of Document: Included Leach Information Corrected Formatting throughout document. Corrected Header information to 6010C from 6010B. Table 3.1: Updated Reporting Limits to match current. Table 3.1, Section 11.6.1.3: Changed unit for Wipe to Total ug. Section 7.1: Added TCLP/SPLP/ASTM Preservation Requirement and Holding Times. Section 7.2: Preservation Check: Changed maximum volume of acid to add as 2% from 5%. Table 9.3.6: Updated Vendor and Catalog number. Section 11.6.4: Updated program for data reduction to ADAMS from MARRS.	28Jun2011
S-GB-M-005-Rev.04	Table 3.1: Updated TCLP/SPLP/ASTM PRLs. Section 9 Tables: Updated formatting to merged cells when the same standard, acids, final volume, etc. are used for one complete standard. Section 9: Updated expiration dates of standards to 6 month or manufacturer expiration date, whichever is sooner. Added Table 9.3.5 CRDL Intermediate Standard. Section 13.4: Changed requirement for IDL Studies to quarterly from yearly. Section 13.6.4 and 13.6.5: Added method modifications. Throughout document: Updated instances of Reagent Water to Nano-Pure Water, updated RLVS to CRDL standard.	30Sep2011
S-GB-M-005-Rev.05	Section 4.2: Updated language on physical interferences. Section 4.5: Updated high salt interferences language. Table 9.3.2: Updated volume of standards used. Table .9.3.5: Updated K concentration in stock and working std. Table 9.3.6: Updated Ag concentration in stock and working std. Section 11.2.4: Updated digestion procedures.	07Dec2012

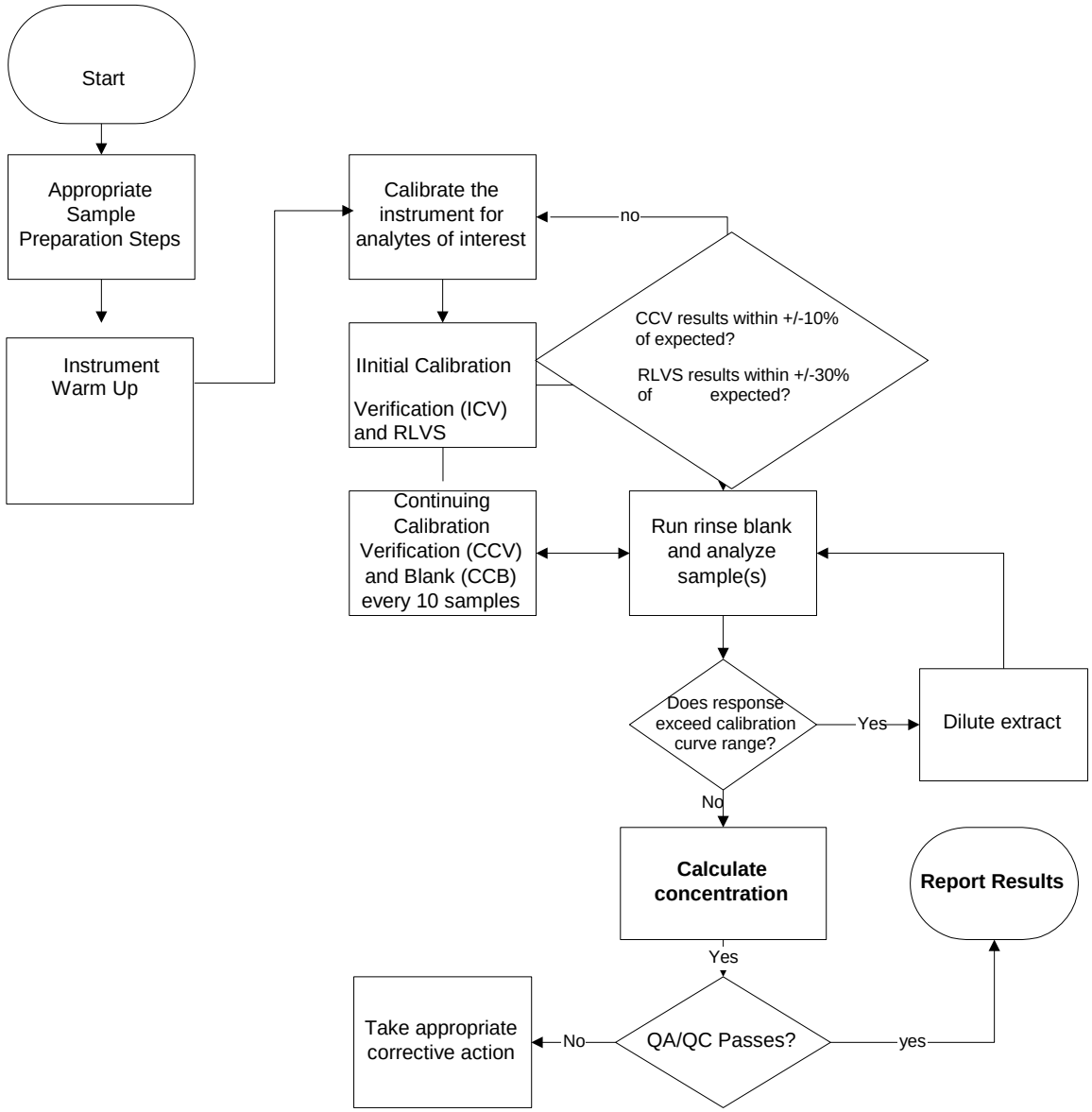
Attachment I

GENERAL ICP TROUBLESHOOTING GUIDE

Problem	Solution
Poor linearity in a dilution series of standards.	This may be as easy as not having spiked your standards correctly. Before you remake your standards, first examine the sample introduction area, making sure you're getting a consistent flow of sample through your pump tubing and that your connectors are not too tight. Then check your nebulizer and injector for visible build up. If build up is present, clean in a dilute nitric acid (HNO_3) solution for about 10-15 minutes. Also examine your torch, making sure it is also reasonably clean. Lastly, check the argon tank to determine that it is not running on empty. If everything is satisfactory, remake standards. If problems persist, try a second source of standards to determine if your initial set of standards has gone bad.
Bubbles are collecting in the back area of the nebulizer (glass type).	The reason why bubbles form in this back area is because the pump tubing is not inserted deeply enough into the cavity of the nebulizer. Cut the tip of the pump tubing to be inserted into the nebulizer at a roughly 45º angle, preferably with a razor blade to obtain a nice, clean cut. Dip the cut end into a wetting agent such as dilute Triton X solution, and start to insert into the nebulizer. Using moderate pressure, begin to work the pump tubing into the nebulizer while twisting the tubing. The twisting action causes the tubing to gradually decrease in diameter, making it easier to insert deeply into the cavity.
Hard instrument failure (software related). These are generally problems associated with the software communication with the instrument.	Exit out of the program. Shut down the PC that is linked to the instrument itself and restart. During the PC reboot process, locate the reset button(s) on the front and/or back of the instrument and depress them. Enter back into the program and continue your analysis. This generally will fix this type of problem, but if you encounter additional problems of this type, then a service call may be necessary for more advanced troubleshooting advice.
Peristaltic pump occasionally stutters or stops. This problem may be hard to notice since this is a gradual wearing of the belt that attaches to the pump motor and turns the rollers themselves.	Replace the belt if you have an available spare. If you do not have a replacement, a service call needs to be made. A service person will generally come out and install the belt but on occasion, based on your mechanical abilities, they may ship the part to you and have you perform the installation.

Attachment II

ICP Flowchart



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STANDARD OPERATING PROCEDURE

MEASUREMENT OF pH IN WATER, SOIL, AND WASTE

REFERENCE METHODS: SM 4500-H^+ AND SW-846 METHODS 9040B AND 9045C

LOCAL SOP NUMBER:	S-GB-I-071-REV.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S -GB-I-071-REV.00
SOP TEMPLATE NUMBER:	SOT-ALL-I-015-Rev.00

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 09/28/12

Kate Grams, Laboratory Quality Manager — Date: 9/26/12

Chad Rusch, Department Manager — Date: 09/27/2012

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

The purpose of this SOP is to provide a laboratory specific procedure to measure pH in water, soil, and waste samples while meeting the requirements of SM Method 4500-H^+ and SW846 Methods 9040B and 9045C.

2. Summary of Method

2.1. Water samples and wastes containing at least 20% water are measured electrometrically using a combination pH electrode with temperature compensation. The method for analyzing this type of sample is either SM Method 4500-H^+ or SW846 9040B and must be reported referencing one of these methods.

2.2. Soils and solids containing less than 20% water are mixed with reagent water and the pH of the resulting aqueous suspension is measured in the same manner as a water sample. The method for analyzing this type of sample is SW846 9045C and must be reported referencing this method.

3. Scope and Application

3.1. This method is applicable to water samples (including drinking water, surface water, groundwater, saline water, and domestic and industrial wastewaters), soils, sludges, and solid wastes.

3.2. Since pH measurement requires some water content, concentrated acids and bases and concentrated acids and bases mixed with inert substances cannot be measured.

3.3. This SOP was prepared for method-compliant use by all Pace Analytical environmental laboratories.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced with electrode measurements. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

4.2. Sodium error at pH levels greater than 10 can be reduced or eliminated by using a “low sodium error” electrode.

4.3. Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or washing with detergent, then rinsing with distilled water. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.

4.4. Sample temperature can cause variations in electrometric measurement of pH in two ways, by causing a change in electrode output and by actually altering the sample pH. Since both variation sources cannot be fully controlled, measurements should be made using a temperature compensating electrode and the analysts should report the sample temperature at the time of analysis along with the pH result. **Coatings of oily material or particulate matter can impair electrode response. The electrode should be thoroughly rinsed and cleaned between samples. Wiping of the electrode using an optical safe wipe may be required to remove oil or clay like soils.**

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

6. Definitions

Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Plastic container (125 mL).	None	If samples cannot be analyzed immediately, they must be stored at 4±2 °C	Samples should be analyzed as soon as possible following collection, preferably at the collection site.(1,2)
Soil	Plastic container (4-oz)	None	If samples cannot be analyzed immediately, they must be stored at 4±2 °C	Samples should be analyzed as soon as possible following collection, preferably at the collection site.(2)

(1) For aqueous samples analyzed and reported according to SM 4500-H^+, PASI observes a 15 minute hold time. Laboratories must qualify results as exceeding the recommended hold time for this ***field parameter*** when analysis occurs beyond this time frame.

(2) For samples analyzed and reported according to either SW-846 9040B or 9045C, PASI observes a 24 hour hold time. Laboratories must qualify results as exceeding the recommend hold time when analysis occurs beyond this time frame.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
pH Meter	Orion	Orion 720A	
Glass Combination Electrode with Automatic Temperature Compensation	Orion	9157BN	Orion Triode pH Electrode
Analytical Balance	Mettler	PJ 480	Capable of weighing to 0.01 g

8.2. General Supplies

Item	Vendor	Model / ID	Catalog #	Description
Disposable Beakers, 50-mL	Fisher		01-29107	
Magnetic Stirrer	Fisher	Corning		
Spin Bars	Fisher			
Wash Bottle	Fisher			
Graduated cylinders	Fisher			50-mL or 100-mL
Laboratory Wipe Tissues	Fisher		06-666A	Kimwipes

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	De-ionized water	ASTM Type II water
Electrode Filling Solution	Used for filling electrode	Orion p/n 90004

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, and second source verification.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Primary Calibration Buffer, pH 2.00 ± 0.02	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB96-1, or equivalent
Primary Calibration Buffer, pH 7.00 ± 0.01	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB107-4, or equivalent
Primary Calibration Buffer, pH 10.00 ± 0.02	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB115-4, or equivalent
Primary Calibration Buffer, pH 12.45 ± 0.01	This standard solution is used for daily calibration of the measurement system. Manufactured by Oakton – p/n 00654-12	MG Scientific p/n T-00654-12, or equivalent
Secondary Source Calibration Buffer, pH 4.00 ± 0.01	This standard solution is used to verify calibration before sample analysis.	Fisher p/n SB101-4, or equivalent
Secondary Source Calibration Buffer, pH 7.00 ± 0.01	This standard solution is used to verify calibration before sample analysis. Manufactured by Oakton – p/n 00654-04.	Fisher p/n13300153, or equivalent

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Primary Calibration Buffer, pH 2.00 ± 0.02	Fisher p/n SB96-1, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 7.00 ± 0.01	Fisher p/n SB107-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 10.00 ± 0.02	Fisher p/n SB115-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 12.45 ± 0.01	MG Scientific p/n T-00654-12, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Secondary Source Calibration Buffer, pH 4.00 ± 0.01	Fisher p/n SB101-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Secondary Source Calibration Buffer, pH 7.00 ± 0.01	Fisher p/n13300153, or equivalent	Manufacturer's recommended expiration date	Store at room temperature

10. Calibration

10.1. Calibration - The measurement system must be calibrated daily with four calibration buffer solutions. These solutions must bracket the range of pH values measured. Additional calibration points are recommended provided the pH meter can accept additional calibration data points.

10.1.1. Press ***power*** to turn the meter on. Press **2nd/channel** until the correct input channel is selected.

10.1.2. Press the **mode** key until the pH mode indicator is displayed.

10.1.3. Press **calibrate.** CALIBRATE is displayed. The time and date of the last calibration is displayed.

10.1.4. After a few seconds ENTER NO. BUFFERS is displayed. Use the numeric keys to enter the number of buffers to be used, then press **yes**. Enter four for the number of buffers to be used.

10.1.5. Place a stir bar in the beaker, put the beaker on the stirrer, and adjust the stirrer to a moderate speed. When the BUFFER 1 prompt appears place electrode(s) in first buffer containing stir bar on.

10.1.6. When the electrode signal has stabilized, the prompt READY CAL AS will appear. Use the numeric keys to enter the value of the buffer. Press **yes** to enter the value.

10.1.7. Rinse the electrode with DI water and gently wipe with a tissue. Repeat steps 5 and 6 for each buffer.

10.1.8. The average electrode slope is displayed at the SLOPE prompt.

10.1.9. The meter automatically proceeds to MEASURE mode.

10.1.10. Record the readings for each buffer used during calibration along with the slope in the pH logbook. (See Attachment I.)

10.2. Initial Calibration Verification – The calibration must be verified with two secondary source calibration buffers prior to conducting measurements on samples. Perform initial calibration verification using the two secondary source calibrations buffers (4.00 and 7.00 pH Units). The control criteria for this verification is listed in Table 12.1.

10.3. Continuing Calibration Verification – The calibration must be verified with a single point verification buffer (pH 7.00) every 10 samples. The control criterion for this verification is listed in Table 12.1.

11. Procedure

11.1. Water Samples

11.1.1. Allow samples to warm to room temperature. Unless a temperature compensated probe is used, samples must be within ±2°C of the standard buffer solutions used for calibration.

11.1.2. Place the sample in a 50-mL disposable beaker, using sufficient volume to cover the sensing elements of the electrode and provide adequate clearance for the magnetic stirring bar. Place a magnetic stirring bar in the beaker and place the beaker on the stirrer. Turn on the stirrer to a moderate speed. The sample should be noticeably mixing but should not develop a vortex.

11.1.3. Rinse the electrode with DI water and gently wipe with a tissue. Place the pH electrode into the sample beaker, taking care to provide adequate clearance for the stirring bar.

11.1.4. Wait for the meter reading to stabilize and note the pH.

11.2. Soil and Solid Samples

11.2.1. Place 20g of sample into a 50-mL disposable beaker. Add 20mLs of DI water and a stirring bar to the beaker and place it on the stirrer. Turn the stirrer to a moderate speed and continuously stir the suspension for 5 minutes.

11.2.2. If the matrix is hydroscopic and has absorbed all the water leaving no remaining free liquid for analysis, repeat the preparation using 20g of sample and 40mLs of DI water.

11.2.3. Turn off the stirrer and allow the sample to sit, permitting the suspended particulates an opportunity to settle to the bottom of the container. Samples may be allowed to sit for up to one hour prior to analysis. Excessive solids in suspension may speed the clogging of the frit between the electrodes.

11.2.4. Place the pH electrode and ATC electrodes into the sample beaker, taking care to immerse the combination electrode just below the suspension. Wait for the meter reading to stabilize and record the pH result and time analyzed. Record the results to 0.1 pH units.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Secondary Source Calibration Buffers	4.00 and 7.00 pH Units Secondary Calibration Buffer Solutions	Daily after calibration	± 0.1 pH Units	Re-calibrate
CCV Buffer	7.00 pH Units Secondary Calibration Buffer Solution	One after every 10 samples and at the end of the batch of samples.	± 0.1 pH Units	Terminate analysis, correct the problem, re-calibrate the system, and re-analyze the check solutions and all samples that are not bracketed by acceptable QC.
Duplicate Sample	Sample	One per batch of 20 or fewer samples for soil matrices. None required for aqueous samples.	5.0% RPD	Terminate analysis, correct the problem, re-calibrate the system, and re-analyze the check solutions and all samples that are not bracketed by acceptable QC.
Calibration of pH Probe				Calibrate the temperature function of the pH probe on an annual basis as per the current revision of PASI SOP S-GB-Q-030, *Support Equipment.* Record the calibration in the thermometer calibration logbook and label the probe with the date of calibration.

13. Method Performance

13.1. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement). All results must be in control to qualify the analyst for reporting sample results (within 0.1 pH units of the true value). Results of DOC studies for each analyst shall be retained in the lab quality assurance office. DOCs must be repeated annually. For this test four analysis of the second source pH 7 buffer will serve as the DOCs.

13.2. Method Modifications: If the matrix is hydroscopic and has absorbed all the water leaving no remaining free liquid for analysis, repeat the preparation using 20g of sample and 40mLs of DI water. This is a method modification of 9045C. Method 9045C requires a 1:1 ratio, but since this would not be possible with a sample of this nature the noted procedure will be followed and the sample qualified as such.

14. Pollution Prevention and Waste Management

14.1. Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

14.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. References

15.1. Method 4500-H^+ B., Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

15.2. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 9040B "pH Electrometric Measurement".

15.3. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 9045C "Soil and Waste pH".

15.4. Pace Analytical Quality Manual; latest revision.

15.5. NELAC Standard; Quality Systems section; latest revision.

16. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

Attachment I - pH Logbook Template.

17. Revisions

Document Number	Reason for Change	Date
SOT-ALL-I-015-rev.00	Restructured document format to new corporate template. Included requirement for duplicate analysis for all aqueous matrices	12Dec2006
S-ALL-GB-I-015-REV.00	Corporate Template updated for use in the Green Bay Laboratory	27Aug2008
S-ALL-GB-I-015_REV.01	Update Signature Page Update Benchsheet	26May2009
S-GB-S-071-Rev.00	Updated SOP numbering following current revision of S-ALL-Q-003 Updated Waste Management and Handling SOP throughout document. Updated pH Logbook to new revision.	27Sep2011
S-GB-I-071-Rev.01	Section 13.2 – Added Method Modification information	26Sept2012

ATTACHMENT I
pH Logbook

Pace Analytical

pH (SW 846 9040B, SW 846 9045C, SM 4500-H+B)

Logbook #

Meter: Orion Model 720A (40WET8)	Meter Serial Number: 008577	
Meter Calibration Date:	Analyst:	
Initial Calibration pH Buffer Solutions		
Value	**pH Readings**	**Reagent Code**
pH 2.00		
pH 7.00		
pH 10.0		
pH 12.45		
Slope:		
Second Source pH Buffer Solutions		
pH 4.00		
pH 7.00		

Sample No.	Date	Time	Matrix	pH.	Adjusted pH	Chlorine	Worksheet #	Analyst

Calibrate pH meter daily with pH buffers 2.0, 7.0, 10.0 & 12.45. Verify the calibration with the second source pH buffers 4.00 and 7.00 (Control Limits +/-0.10 SU). Analyzed second source pH buffer 7.00 after every 10 readings (Control Limits +/- 0.10 SU). Analyze duplicate sample every 20 samples (Control Limits +/- 0.10 SU). For SW846 9040B analyze consecutive measurements of the sample to verify they read within +/- 0.10 SU)

F-GB-I-009-Rev.06 (28Sep2011)

Review/Date_____

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STANDARD OPERATING PROCEDURE

Measurement of Volatile Solids and Solids in Waters

Reference Methods: Standard Methods 2540-B, -C, -D, -E, -G, ASTM D2974 and EPA 160.4

SOP NUMBER:	S-GB-I-068-Rev.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S- GB-I-068-Rev.00

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/03/12

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PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose /Identification of Method

1.1 This Standard Operating Procedure (SOP) describes operations used to measure the parameters in the following table:

Table 1.

Test		Matrix	Method	Reports as
TS	Total Solids	Water	SM 2540B	mg/L
TS	Total Solids	Solid	SM 2540G	%
TSS	Total Suspended Solids	Water	SM 2540D	mg/L
TDS	Total Dissolved Solids	Water	SM 2540C	mg/L
TVS	Total Volatile Solids	Water	EPA 160.4	mg/L
TVS	Total Volatile Solids	Solid	EPA 160.4	%
TVSS	Total Volatile Suspended Solids	Water	EPA 160.4	mg/L
OM	Organic Matter	Solid	ASTM D2974-87(C)	%
FOC	Fractional Organic Carbon	Solid	ASTM D2974-87	%

2. Summary of Method

2.1 Total Solids (TS) – A well-mixed sample is evaporated to dryness and the residue solids are measured gravimetrically.

2.2 Total Suspended Solids (TSS) – A well-mixed sample is filtered. The residue collected by the filter is dried and measured gravimetrically.

2.3 Total Dissolved Solids (TDS) – A well-mixed sample is filtered. The filtrate passing through the filter is evaporated to dryness and the residue solids are measured gravimetrically.

2.4 Total Volatile Solids (TVS, TVSS) – Residue obtained from the determination of TS, or TSS is ignited at 550ºC in a muffle furnace. The loss of weight on ignition is reported as % Volatile Solids for solid samples and mg/L Volatile Solids for liquid samples.

2.5 Fractional Organic Carbon (FOC) - Residue obtained from the determination of TS is ignited at 440ºC in a muffle furnace. The weight loss after ignition is multiplied by a 0.58 factor and reported as % Fractional Organic Carbon.

2.6 Organic Matter (OM) - Residue obtained from the determination of TS is ignited at 440ºC in a muffle furnace. The weight loss after ignition is reported as % Organic Matter.

3. Scope and Application

3.1 This method is applicable to the determination of Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS), and Total Volatile Solids (TVS) in water samples.

3.2 This method is applicable to the determination of Total Solids (TS) and its Volatile (TVS) fractions in soil and semisolid samples. The terms total solids apply to matter left after drying in an oven at 103-105º C and igniting in a muffle furnace at a 550ºC temperature and a defined time interval.

3.3 This method is applicable to the determination of Fractional Organic Carbon (FOC) and Organic Matter (OM) in soil and semisolid samples. The term fractional organic carbon (FOC) applies to the weight loss after ignition, multiplied by a 0.58 factor and reported as % Fractional Organic Carbon.

3.4 This procedure is restricted to use by, or under the supervision of, analysts experienced with the use of laboratory information systems, balances, desiccators, and ovens. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1 This SOP applies to surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, soils, and sludges.

5. Limits of Detection and Quantitation

5.1 The reporting limit (LOQ) for all analytes is listed in Table 1 for the listed methods. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

Table 2.
REPORTING LIMITS

COMPOUND NAME	Aqueous	Solids
	Units mg/L	Units %w/w
	LOQ	LOQ
TS	20	0.1
TSS	10	
TDS	20	
TVS, TVSS	20	
FOC		0.058
OM		0.1

6. Interferences

6.1 Non-representative materials, e.g., leaves and sticks should be removed from the sample prior to measurement unless it is determined that their inclusion is desired.

6.2 Measurements are subject to negative bias for samples containing significant quantities of ammonium carbonate, volatile organics, or other volatile materials that could be lost during drying.

6.3 The residue of samples for TS and TDS that are highly mineralized, especially containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require longer drying, desiccation, and rapid weighing.

6.4 Samples for TS and TDS containing high concentrations of bicarbonate will require careful, and possibly prolonged, drying to ensure that all bicarbonate is converted to carbonate.

6.5 The volumes of aliquots for TS and TDS should be selected to limit the total residue to 200 mg to prevent the residue from crusting over and trapping water during drying.

6.6 Samples for TSS with high TDS, such as saline waters, brines, and some wastes, may be subject to positive bias. Care must be taken to properly rinse the filter to minimize the bias.

7. Sample Collection, Preservation Shipment and Storage

Table 3
SAMPLE COLLECTION, PRESERVATION, HOLD TIME SHIPMENT AND STORAGE

Matrix	Analytical Method	Container(s)	Preservation / Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	SM 2540B SM 2540C SM 2540D EPA 160.4	Clean Plastic or glass containers	None / 7 Days	On ice ≤6° Celsius	Walk-in cooler ≤6° C
Soils	SM 2540G ASTM D2974-87	Clean Plastic or glass containers	None / 7 Days	On ice ≤6° Celsius	Walk-in cooler ≤6° C

8. Definitions

8.1 Batch – A grouping of 20, or less, environmental samples of similar matrix, which are prepared and/or analyzed together with the same method.

8.2 Blank Sample – A sample analyzed to determine potential contamination attributed to sample handling and analysis.

8.3 Duplicate Sample – A second aliquot of the same environmental sample analyzed in the same manner as the original sample in order to evaluate precision.

8.4 Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required.

8.5 Laboratory Control Sample (LCS) – A blank matrix sample spiked with a known concentration of analytes of interest. Laboratory accuracy is evaluated using the LCS.

8.6 Laboratory Information Management System (LIMS) – This is a system for transferring, processing, storing, and reporting analytical results.

8.7 Replicate Samples – Samples collected at the same time, from the same place, for the same analysis, as the original sample in order to determine precision between samples.

8.8 Pace Reporting Limit (PRL) – The lowest level that will be reported by this method. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.

8.9 Solid – matrix such as soils, sediments, sludge, organic liquids, oils or aqueous products and by-products of industrial processes, and aqueous samples with more than 10% settleable solids.

8.10 Total Solids Content (TSC) – Total Solids Content is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature of 103-105ºC.

8.11 Volatile Solids – Residue obtained from the determination of TS is ignited at 550ºC in a muffle furnace. The loss of weight on ignition is reported as % Volatile Solids.

8.12 Refer to Section 10 of the Pace Quality Manual for additional terms used in this SOP.

9. Equipment and Supplies

9.1 Analytical balance – Electronic, capable of weighing to 0.1 mg.

9.2 Vacuum Filtration System – Includes filter holder, membrane filter funnel, vacuum flask and vacuum pump.

9.3 Glass Fiber Filters (for TDS) – GE Water & Process Technologies PreSep Prefiler, Glass, 1.0 Micron, 47mm, or equivalent.

9.4 Glass Fiber Filters (for TSS) – Pre-weighed from Environmental Express, ProWeigh 47mm Glass Fiber Filters, 1.5 µm, with matched aluminum evaporating dishes, or equivalent.

9.5 Graduated Cylinders – 25, 50, 100, 250, 500 and 1000mL.

9.6 Wide bore serological pipettes and pipette helper.

9.7 Drying Ovens – Capable of maintaining 103-105ºC and 178-182ºC.

9.8 Desiccator

9.9 Indicating Desiccant – Drierite Anhydrous Calcium Sulfate, Stock # 2305, or equivalent.

9.10 Laboratory Scoopulas

9.11 Muffle furnace (for volatile solids) – Capable of maintaining temperature of 440ºC and 550ºC.

9.12 Evaporating Beaker (TS/TDS/TVS/OM/FOC) – 50 mL

10. Reagents and Standards

10.1 ERA DataPacK Total Volatile Solids Standard, Cat # 913 for TVS, OM, and FOC analysis, or equivalent.

10.2 ERA QC Plus Solids, Cat #4033 for TS, TSS, and TDS analyses, or equivalent.

10.3 NSI Solutions, Cat # QCI-084 for TS, TSS, and TDS analysis, or equivalent.

10.4 Nanopure Reagent Water – ASTM Type II, or equivalent.

11. Calibration and Standardization

11.1 Balance Calibration – The only required calibration is the analytical balance.

11.2 The balance must be calibrated at least annually by an outside agency and checked **daily** across the range of use, using Class 1 or 2 weights as per the most current version of SOP S-GB-Q-030 *Support Equipment.*

11.3 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

11.3.1 1g for TSS and TVSS with a tolerance of +/-0.0100g

11.3.2 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

12. Procedure

12.1 Create the analytical batch in Horizon.

12.2 Open the Electronic Logbook and create a new worksheet for the batch to be analyzed.

12.3 Pre-Weighed Dried Beaker. Ignite the evaporating beaker at 550ºC for 1 hour in a muffle furnace. Cool in desiccator and store there until ready to use. Record their weight prior to use.

12.4 Check that the oven temperature is within required specifications before placing samples into oven as per the most current version of SOPS-GB-Q-030 *Support Equipment*. Document temperature in the Electronic Logbook.

12.5 **Weighing Dry/Muffle Weights**

12.5.1 Oven temperatures are the same as those described for the test being conducted.

12.5.2 Place the samples back into the oven for 15 minutes and repeat desiccating, cooling, and weighing steps until weight change is $\leq$0.0005g.

12.5.3 If the first and second weights agree within 0.0005 g, or if the weight loss between the first and second weighing is less than 4%, the analysis is complete. If not, place them back into the oven for and additional 15 minutes. Cool in the desiccator, and weigh a third time.

12.5.4 If the third weigh is within 0.0005 g of either the first or second weight or if the weight loss between the second and the third weighing is less than 4%, the analysis is complete.

12.5.5 If the weight loss between the second and third weighing is more than 0.0005g and 4%, heat, cool, and weigh the sample a fourth time and use the fourth weight to calculate the result.

12.6 **Total Solids Determination (TS)**

12.6.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be $\leq$0.0005g before use.

12.6.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in Electronic Logbook.

12.6.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of NSI Solutions (or equivalent) and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in Electronic Logbook.

12.6.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.6.5 Sample

12.5.4.1 For Solid Samples – Weight out 1 to 1.5g of sample in a tared and dried evaporating beaker. Record the weight in Electronic Logbook.

12.5.4.2 For Aqueous Samples - Shake the sample thoroughly and measure an aliquot (between 1mL and 50mL, typically 50mL) into a pre-dried evaporating beaker using a graduated cylinder. Record the volume in Electronic Logbook.

12.6.6 Place the samples to dry in the oven overnight at 103-105°C. (Overnight is a period of time $\geq$ 8 hours.)

12.6.7 After drying overnight, remove from oven and place into the desiccator to cool. After the samples have cooled, obtain the weight of the samples as per Section 13.5, and record in Electronic Logbook.

12.6.8 If Total Volatile Solids (TVS) are also to be performed proceed to Section 12.9. Note: TVS utilizes a different LCS than the TS.

12.7 **Total Suspended Solids Determination (TSS)**

12.7.1 Record Filter IDs and weights in Electronic Logbook for samples and batch QC.

12.7.2 Method blank (MB) – Using a graduated cylinder measure out 1000mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.7.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of Alpha-Trol (or equivalent); record the volume in Electronic Logbook.

12.7.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.7.5 For wastewater, the Pace Analytical Services, Inc. - Green Bay Laboratory uses 500mL in order to meet Wisconsin Reporting Limit requirement of 2.0 mg/L.

12.7.6 For South Carolina work a sample volume is chosen to yield between 2.5 and 200 mg dried residue. If volume filtered fails to meet minimum yield, increase sample volume up to 1 L.

12.7.7 Shake the sample thoroughly and measure the chosen aliquot using a graduated cylinder.

12.7.8 Prepare the filter system with a fresh pre-weighed filter. Handle the filter with forceps only. Wet the filter with a small volume of Nanopure water to seat it. Discard the rinse water.

12.7.9 Filter the sample and wash the filter 3 times with approximately 10mL of Nanopure water. Maintain the filter vacuum for about 1 minute after filtration is complete.

12.7.10 Remove the filter with forceps and place it back into the evaporating dish.

12.7.11 Place the samples (filters and residue) to dry in the oven a minimum of 3 hours at 103-105°C.

12.7.12 After drying, remove from oven and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.7.13 If Total Volatile Suspended Solids (TVSS) are also to be performed proceed to Section 11.10. TVSS utilizes a different LCS than TSS.

12.8 **Total Dissolved Solids (TDS)**

12.8.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.8.2 Method blank (MB) – Using a graduated cylinder measure out 50mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.8.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of APG Solids APG plus Standard (or equivalent), record the volume in Electronic Logbook.

12.8.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.8.5 Shake the sample thoroughly and measure a 50mL aliquot using a graduated cylinder.

12.8.6 Prepare the filter system with a fresh filter. Handle the filter with forceps only. Wet the filter with a small volume of Nanopure water to seat it. Discard the rinse water.

12.8.7 Filter the sample and transfer the filtrate to a pre-weighed dried evaporating beaker.

12.8.1 Place the samples to dry in an oven that has it temperature under a program. The oven starts out at <100 degrees C and increases in temperature according to the program until it reaches 178 - 182 degrees C. It is then held there for a minimum of 1 hour.

12.8.2 After drying overnight, remove from oven and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.9 **Total Volatile Solids (TVS)**

12.9.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.9.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.9.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.9.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.9.5 Sample

12.5.4.3 For Solids Samples – Weight out 1 to 1.5g of sample in a pre-dried, tared evaporating beaker. Record the weight in Electronic Logbook.

12.5.4.4 For Aqueous Samples - Shake the sample thoroughly and measure an aliquot (between 1mL and 50mL, typically 50mL) into a pre-dried evaporating beaker using a graduated cylinder. Record the volume in Electronic Logbook.

12.9.6 Follow TS Section to obtain a dried residue.

12.9.7 Place the evaporating beaker or filter and evaporating dish into the muffle furnace at **550°C** for 1 hour.

12.9.8 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.10 Total Volatile Suspended Solids (TVSS)

12.10.1 Method blank (MB) – Using a graduated cylinder, measure out 1000mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.10.2 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.10.3 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.10.4 Shake the sample thoroughly and measure the chosen aliquot (see sections 11.7.5 and 11.7.6) using a graduated cylinder. Record the volume in Electronic Logbook.

12.10.5 Follow TSS Section to obtain a dried residue.

12.10.6 Place the filter and evaporating dish into the muffle furnace at **550ºC** for 1 hour. Write the filter number on the evaporating dish – paper label will burn off.

12.10.7 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5 in Electronic Logbook.

12.11 Fractional Organic Carbon (FOC) and Organic Matter (OM)

12.11.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.11.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pour into a tared evaporating beaker, record the weight in Electronic Logbook.

12.11.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker, and record the weight in Electronic Logbook.

12.11.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.11.5 Samples – Into a tared evaporating beaker weigh out about 1-1.5g of sample. Record the weight in Electronic Logbook.

12.11.6 Follow TS Section to obtain a dried residue.

12.11.7 Place the evaporating beakers into the muffle furnace at **440ºC** for 1 hour.

12.11.8 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5 in Electronic Logbook.

12.12 **Entering Results into Horizon**

12.12.1 Post samples and batch QC using Electronic Logbook.

12.12.2 Evaluate batch QC in Horizon.

13. Quality Control

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 Method Blank (MB) – A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples.

13.2.1 TS, TDS, TVS, FOC, and OM all use 50mL of deionized water for the method blank.

13.2.2 TSS and TVSS both use 1000mL of deionized water for the method blank.

13.2.3 For tests that are reported in %, the MB is evaluated in mg/Kg. The mg/kg result must be less than 20 mg/kg. Data can be reported if the sample mg/Kg result is 10x greater than the method blank. The value reported in Epic Pro will be adjusted to 0%.

13.2.4 For tests that are reported in mg/L the absolute value must be < PRL.

13.2.5 When measurements are above the PRL, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples analyzed since the last compliant calibration blank

13.2.6 If the analyte is detected in the method blank between the MDL and the PRL, then samples need to be qualified with a "B" when MDL reporting is required and sample results are greater than the MDL and less than 10 times the absolute value detected in the blank. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample-by-sample basis. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

13.3 Laboratory Control Sample (LCS) – The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples.

13.3.1 ERA DataPacK Total Volatile Solids Standard, Cat. # 913 is used for TVS, FOC, and OM analysis. Equivalent can be used.

13.3.2 APG Solids APG Plus standard, Cat #4033 is used for TDS analysis. Equivalent can be used.

13.3.3 NSI Solutions, Cat # QCI-084 for TS, TSS, and TDS analysis, or equivalent. Equivalent can be used.

13.3.4 A Laboratory Control Spike Duplicate (LCSD) must be analyzed if the client requests one.

13.3.5 The LCS recovered concentration must be within calculated in-house limits or default limits of $\pm$ 20%.

13.3.6 For tests that are reported in %, the LCS is evaluated in mg/L.

13.3.7 When measurements are outside the control limits, check for errors in calculations, standards preparation, and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.3.8 If no errors are found, sufficient sample is available, it is within hold, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still outside the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.3.9 If sufficient sample volume is not available or the samples are now outside of hold, report the sample data with a qualifier flag (L0) on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.3.10 When an LCSD is performed, the precision between the LCS and LCSD must be ≤10% RPD.

13.3.11 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.3.12 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.4 Duplicate – Analyze one sample in duplicate per 10 samples or less. The sample used for the duplicate is selected at random, unless specified by the client. The relative percent different (RPD) between duplicates must be ≤10%.

13.4.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.4.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an "R1" data qualifier

13.5 Constant Weight – Place the samples back into the 103-105ºC oven, and/or 440/550ºC muffle furnace for 15 minutes and repeat cooling, weighing, and desiccating steps until weight change is ≤0.0005g or the weight loss between the two weights is less than 4%.

13.6 Balance – The balance must be verified daily across the range of use, before use, as per the most current version of SOP S-GB-Q-030 *Support Equipment*.

13.6.1 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

13.6.2 1g for TSS and TVSS with a tolerance of +/-0.0100g

13.6.3 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

13.7 Oven – The oven temperature must be within required specifications before placing samples into oven and the temp is recorded in the Electronic Logbook with the correction factor, as per the most current version of SOP S-GB-Q-030 *Support Equipment.*

13.8 Hold – When preparation of a sample exceeds 7 days past the time of collection, notify the project manager before proceeding. If a sample is run past 7 days after collection, flag the result with an "appropriate data qualifier and fill out a LabTrack report.

13.9 See attachments Table A and Table B for a summary of QC

14. Data Analysis and Calculations

14.1 Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS) in mg/L $= \frac{(A - B) \times 1000 \times 1000}{V}$

Where:	V = sample volume (mL)
Total Solids	A = final weight (dry weight of residue and dish, g) B = dish weight (g)
Total Suspended Solids	A = final weight (dry weight of residue and filter, g) B = filter weight (g)
Total Dissolved Solids	A = final weight (dry weight of residue and dish, g) B = dish weight (g)

14.2 Volatile Solids (TVS and TVSS mg/L) $= \frac{(A - D) \times 1000 \times 1000}{V}$

14.3 Volatile Solids (TVS %) $= \frac{(A - D) \times 100}{(A - B)}$

14.4 % FOC $= ((1-((D-B) / (A – B))) \times 100) \times (0.58)$

14.5 % OM = ((1-((D-B) / (A-B))) x 100)

Where:

A = dry weight (weight of dried residue and dish, g)
B = dish weight (g)
C = weight of wet sample and dish (g)
D = muffled weight (weight of residue and dish after ignition, g)
V = volume (mL)

14.6 Precision - The relative percent difference (RPD) is calculated as:

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

Where: **S** = sample value
D = duplicate value

14.7 Constant Weight is calculated as:

$$\textbf{Constant Weight (\%)} = \frac{|FW - CW| \ * 100}{(FW + CW)/2}$$

Where: FW = First Weighing, mg

CW = Confirmation Weighing, mg

If FW – CW is 0.0005 g or less, then there is not a need to calculate as a percentage

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See Section 13 and Table 3: Quality Control, and Table 4: Data Assessment for a listing of Quality Control Criteria.

Table 5.
QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SM 2540G – ASTM D2974-87** **Frequency**	**Acceptance Criteria**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	For solid samples the MB must be < 20 mg/Kg and is reported as 0%. For aqueous samples the MB absolute value must be <RL. Detections > MDL but < RL must be evaluated for data qualification.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. None performed on TVSS.	Recovery must be within +/- 20% of the true value. RPD must be < 10%
Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.	RPD must be < 10%

Table 6.
ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**SM2540G – ASTM 2974-87 If these conditions are not achieved ⇒**
Method Blank	• 1
Duplicate	• 2
Accuracy & Precision Laboratory Control Spikes	• 3

1. In the absence of project specific requirements, sample detects less than 20 times the method blank contamination level are reported with the appropriate data qualifier. Sample detects greater than 20 times the method blank contamination are reported without qualification.
2. In the absence of project specific or method requirements, in-house generated limits will be used. If the DUP fails precision control limits flag the parent with the appropriate data qualifier.
3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate qualifier qualifiers to reflect the deficiencies.

16. Corrective Actions for Out-of-Control Data

16.1 See Section 13 and Table 6.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 If not specifically listed in Section 13 or Table 6, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers.

18. Method Performance

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 Demonstration of Capability (DOC) – Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC). DOC's must be repeated annually. Analyze 4 replicate environmental sample aliquots. Calculate the mean and standard deviations of the results. The relative percent different (RPD) between replicates must be $\leq$ 5% or the study must be repeated.

19. Method Modifications

19.1 Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1 Maintain the analytical balance, ovens, and furnaces according to the most current revision of SOP S-GB-Q-030, *Support Equipment.*

21. Troubleshooting

21.1 Refer to the support equipment operating manual.

22. Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001 *Waste Handling and Management* most current revision or replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. References

25.1 Pace Analytical Quality Assurance Manual (most current revision or r*eplacement).*

25.2 *T*he NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (most current revision or replacement).

25.3 Method 2540-G, Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992.

25.4 ASTM D2974-87, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, May 29, 1987

25.5 VA30007.00.004, Recommendations Regarding the Sampling and Analysis of Fractional Organic Carbon (FOC) in Soils, Technical Decision Compendium, Rule OAC 3745-300-07, March 2000

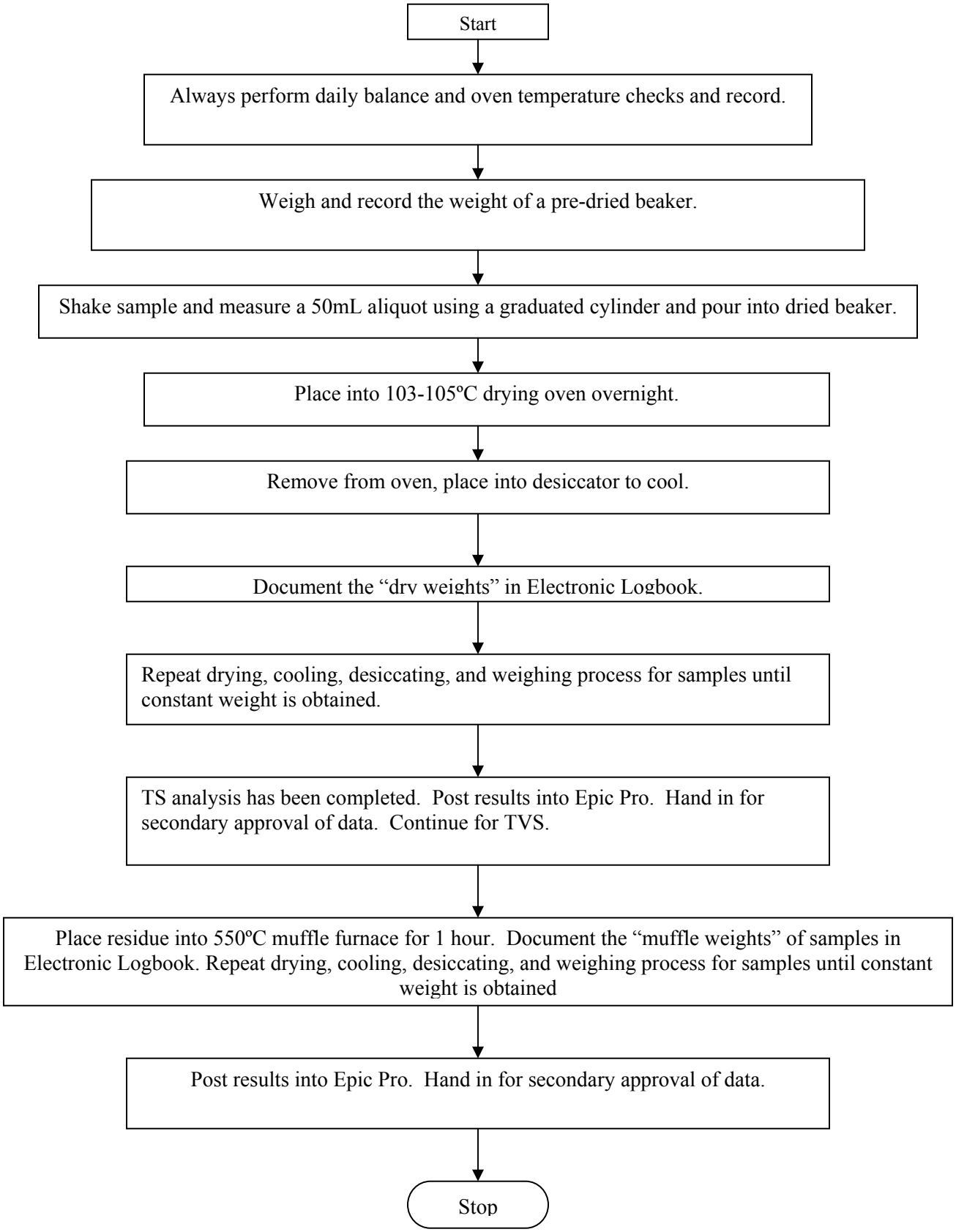
26. Tables, Diagrams, Flowcharts, Appendices, Addenda, etc.

26.1 Flow charts – Attachment I.

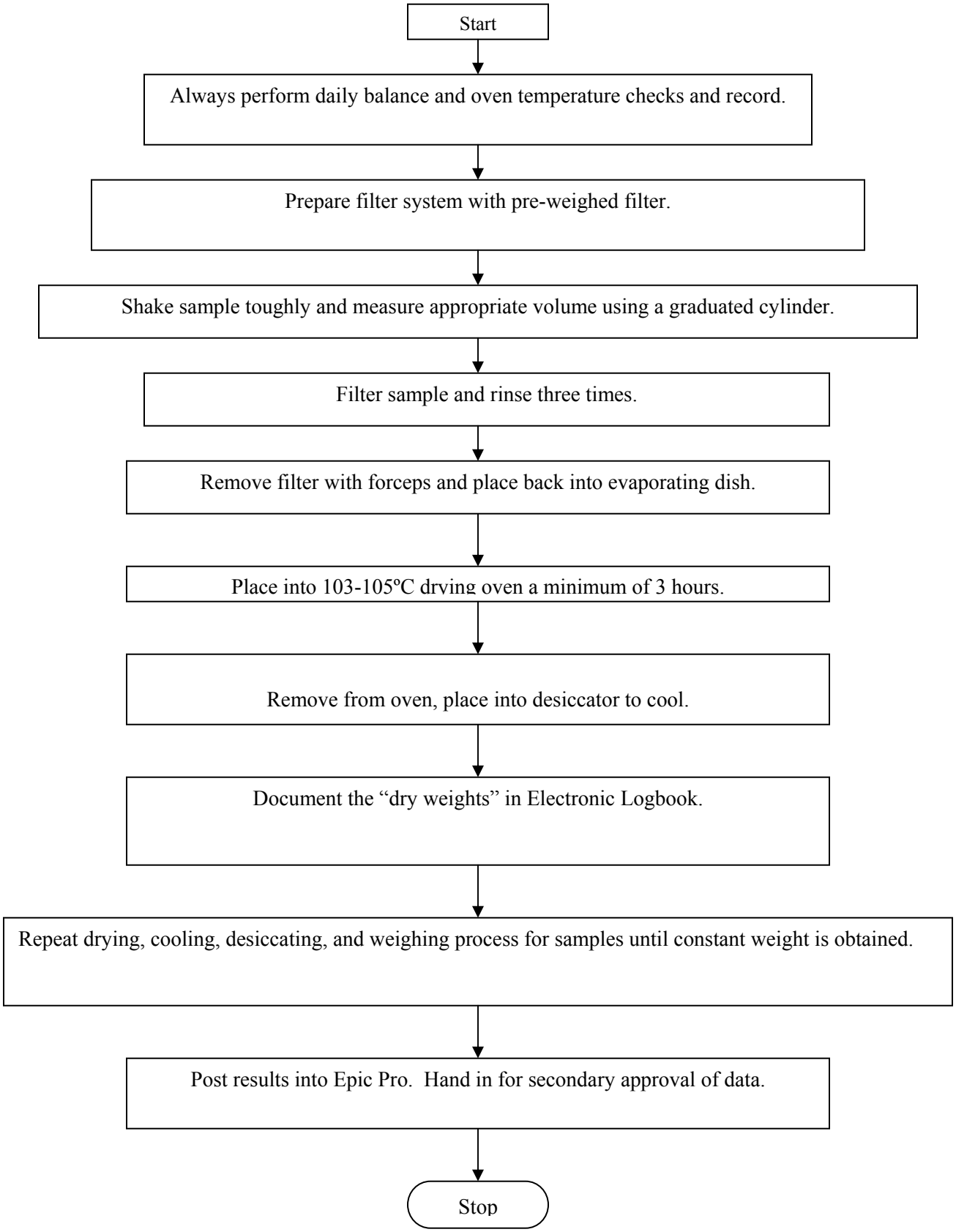
27. Revisions

Document Number	Reason for Change	Date
S-ALL-GB-I-014-Rev.1	Consolidated SOPs ALL-I-014-rev.1, ALL-GB-I-014-rev.1, and GB-I-050-Rev.0. Section 12.6.2 – added South Carolina minimum residue requirements.	24Aug2007
S-ALL-GB-I-014-Rev.2	Corrected FOC and OM calculations and definitions	05Oct2007
S-ALL-GB-I-014-Rev.3	Clarified analysis steps for each test. Changed qualifiers to match Epic Pro.	28Apr2008
S-ALL-GB-I-014-Rev.4	Included notations on how to evaluate MB and LCS for tests reporting in %. Updated Signature Page to Periodic Review.	29May2009
S-GB-I-068-Rev.00	Updated numbering format to match SOP: S-ALL-Q-003, Document Numbering Changed Qualifier from & to L0 and * to R1.	21Sept2010
S-GB-I-068-Rev.01	Updated SOP format to be consistent with SOP: S-GB-Q-017 *Preparation of SOPs* Updated SOP references throughout document. Throughout document: Changed LimsLink to Electronic Prep Log. Sections 2, 3.2, 8.1.2: Removed information relating to Fixed Solids (FS). Sections 2, 3.3, 12.11: Removed information relating to ASH. Section 12.8.1: Changed temperature program to ramp up to 100 deg C. Section 12.10.2: Added LCS requirement to TVSS methods. Tables A and B: Moved and renamed in appropriate sections of SOP.	29Nov2012

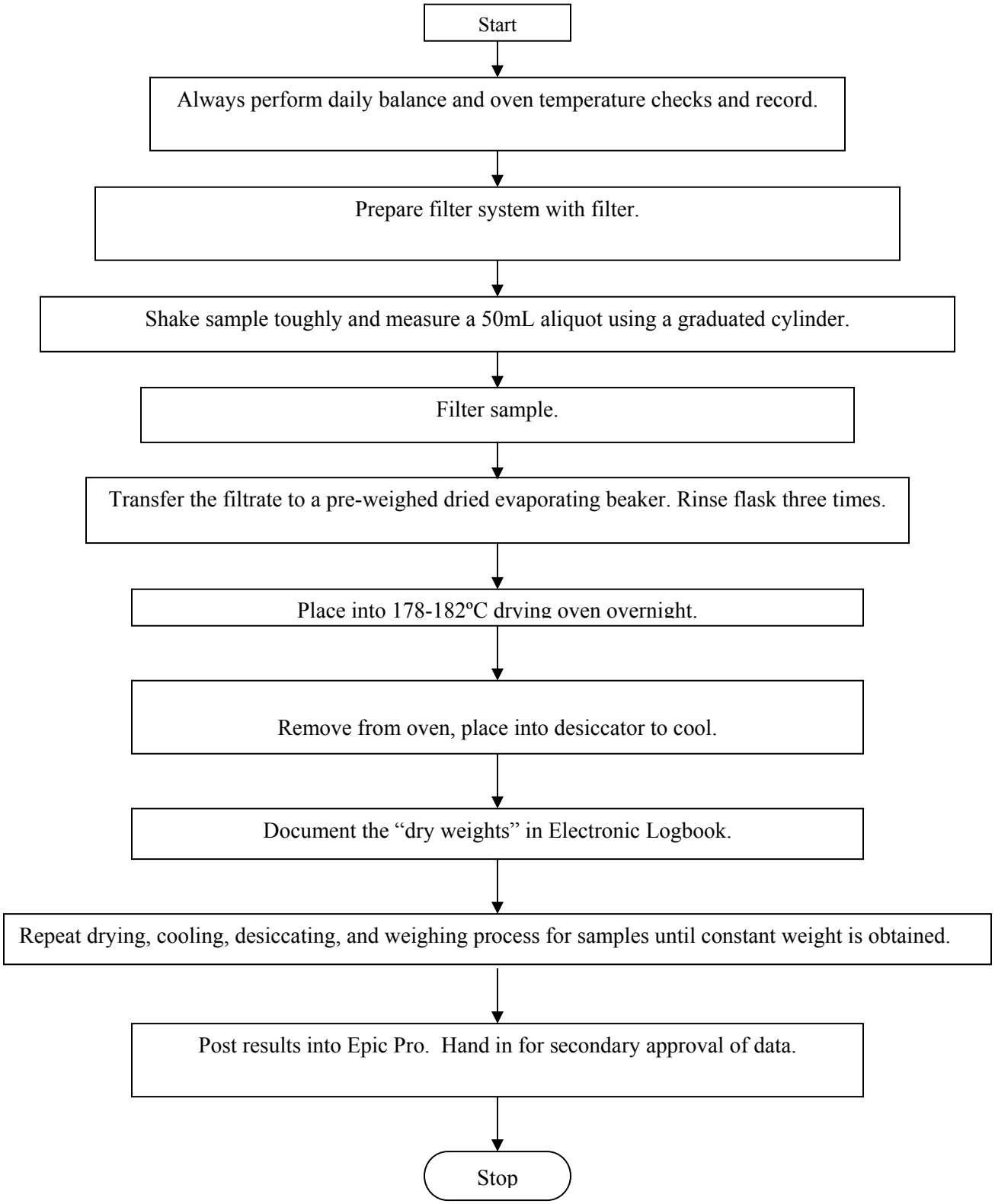
Attachment I – TS and TVS FLOWCHART



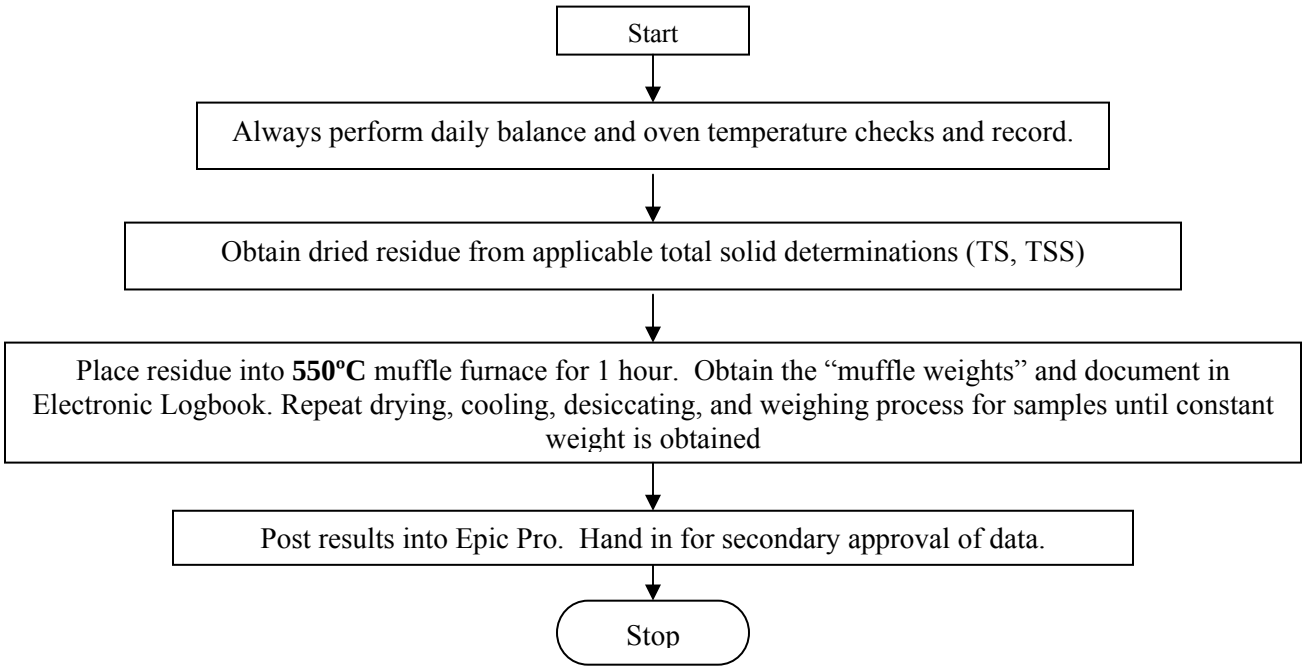
Attachment I – TSS FLOWCHART



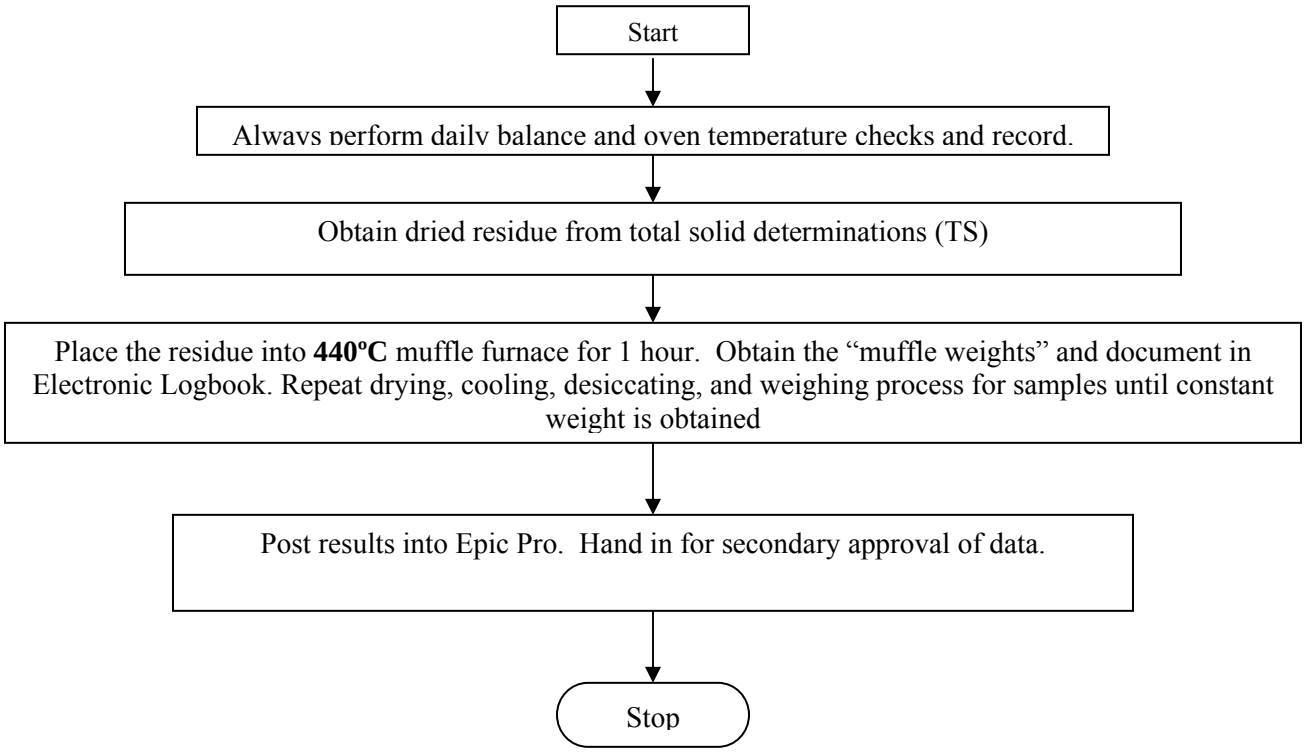
Attachment I – TDS FLOWCHART



Attachment I – TVS and TVSS FLOWCHART



Attachment I – OM and FOC FLOWCHART



APPENDIX D

Laboratory QA Manuals and SOPs

Quality Systems Manual

For

Environmental Chemistry Consulting Services, Inc. (ECCS)
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March 1, 2013

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ACRONYMS

A list of acronyms used in this document and their definitions are:

AgChem	–	Agricultural chemical
AIHA	–	American Industrial Hygiene Association
ANSI	–	American National Standards Institute
ASTM	–	American Society for Testing and Materials
Blk	–	Blank
BS	–	Blank spike, same as LCS
°C	–	Degrees Celsius
CAF	-	Corrective action form
CAL	–	Calibration
CAS	–	Chemical Abstract Service
CCB	–	Continuing calibration blank
CCV	–	Continuing calibration verification
COC	–	Chain of custody
DATCP	–	Department of Agriculture, Trade and Consumer Protection (Wisconsin)
DOC	–	Demonstration of capability
DoD	–	Department of Defense
Dup	–	Laboratory duplicate
ECCS	–	Environmental Chemistry Consulting Services, Inc.
EPA	–	Environmental Protection Agency
g/L	–	Grams per liter
GC/MS	–	Gas chromatography/mass spectrometry
HPLC	–	High performance liquid chromatography
IBL	–	Instrument blank
ICB	–	Initial calibration blank
ICP-MS	–	Inductively coupled plasma-mass spectrometry
ICal	–	Initial calibration
ICV	–	Initial calibration verification
ISE	–	Ion selective electrode
lb/in^2	–	Pound per square inch
LCS	–	Laboratory control sample
LCS-CL	–	LCS control limit
LELAP	–	Louisiana Environmental Laboratory Accreditation Program
LIMS	–	Laboratory information management system
LOD	–	Limit of detection
LOQ	–	Limit of quantitation
MDA	–	Minnesota Department of Agriculture
MDL	–	Method detection limit
mg/kg	–	Milligrams per kilogram
mg/L	–	Milligrams per liter
MS	–	Matrix spike or mass spectrometer

MSD	–	Matrix spike duplicate or mass selective detector
MSDS	-	Material safety data sheet
µg/L	–	Micrograms per liter
µg/kg	–	Micrograms per kilogram
NELAC	–	National Environmental Laboratory Accreditation Conference
NELAP	–	National Environmental Laboratory Accreditation Program
NIST	–	National Institute of Standards and Technology
OM	–	Operations manager
PCB	–	Polychlorinated biphenyl
PEM	–	Performance evaluation mixture
PT	–	Proficiency test(ing)
PQL	–	Practical quantitation limit
PS	–	Post-digestion spike
QA	–	Quality assurance
QAPP	–	Quality assurance project plan
QC	–	Quality control
QM	–	Quality manager
QS	–	Quality system
QSM	–	Quality system manual
R	–	Correlation coefficient
R^2	–	Coefficient of determination
RES	–	Chromatography resolution check
RL	–	Reporting limit
RPD	–	Relative percent difference
RSD	–	Relative standard deviation
SAP	–	Sampling and analysis plan
SDWA	–	Safe Drinking Water Act
SI units	–	SI base units, NIST Special Publication 330 (SP 330), The International System of Units (SI)
SOP	–	Standard operating procedure
SRD	–	Serial dilution
SVOC	–	Semi-volatile organic compound
PAH	–	Polynuclear aromatic hydrocarbons
TNI	–	The NELAC Institute
TUN	–	MS Tune
TPH	–	Total petroleum hydrocarbon
VOC	–	Volatile organic compound
XRF	–	X-ray fluorescence
WDNR	–	Wisconsin Department of Natural Resource

1.0 INTRODUCTION

The Environmental Chemistry Consulting Services, Inc. (ECCS) quality system manual (QSM) codifies our quality system (QS), with the goal of providing the highest quality laboratory services to its clients. This introduction includes the QSM applicability, a brief overview of the ECCS organization, and our mission statement.

1.1 Applicability

This QSM is applicable to the operation and management of the QS for ECCS. The QSM applies to ECCS testing services that are seamlessly performed in both our fixed-facility laboratory in Madison, Wisconsin and on site utilizing (a) one of a number of mobile laboratories, (b) locally-leased construction trailers, or (c) existing site structures. The QSM defines the policies, procedures, and documentation that assure analytical services continually meet a defined standard of quality that is designed to provide clients with data of known and documented quality and, where applicable, demonstrate regulatory compliance. This manual can and should be supplemented by project-specific requirements.

Policy

This QSM provides for the operation and management of the QS for ECCS' on-site and fixed facility laboratory services operations. It does not apply to SDWA compliance monitoring or chemistry consulting services.

1.2 Company

ECCS provides three partially integrated services to the environmental consulting and engineering community: (1) environmental laboratory services occurring in both a fixed facility and on site, (2) Safe Drinking Water Act (SDWA) compliance monitoring, and (3) chemistry consulting services. Since inception, ECCS has created a unique business model that effectively shares technical and support staff between its service areas. The on-site and fixed facility laboratory operations seamlessly share resources (staff, instrumentation, supplies, and management) and systems via a single profit center. Normal business support operations such as sales and marketing, project management, human resources, health and safety, quality assurance (QA), purchasing, reporting, filing, and archiving serve both on-site and fixed facility laboratory operations under a single management system. This sharing concept allows on-site and fixed facility laboratory services to be seamless from an operations and a QS standpoint.

The business model requires a special company culture for success. Employees (chemists) must be experienced, self starters, skilled multi-taskers, and on-site client services representatives. As such, the owners created a culture with core values that have empowered and trusted employees to treat the company as their own. The core values listed below have nourished the growth and success of the company to date and will continue to lead ECCS to future growth and success:

- Empower and trust employees to treat the company as their own

- Be there to provide 110% support to fellow team members
- Deliver quality analytical services that are unique and meet or exceed the needs of our clients and the regulatory community
- Embrace the concept of educating our clients and regulators to create marketplace
- Realize the client's specific needs are paramount and above ours
- Believe that our reputation is everything
- Maintain growth and profitability to allow employees to have opportunity and be rewarded for their efforts, performance, and individual or team results
- Assure competitive (or better) salaries and benefits
- Strive for excellence and continual improvement.

1.3 Mission

ECCS on-site and fixed facility laboratory services mission is to provide faster, better, and more cost-effective solutions to our clients' project-specific analytical needs. ECCS uses its experience and expertise to translate U.S. Environmental Protection Agency (EPA) test methods into project specific analytical solutions that can be effectively applied in an on-site laboratory environment, that are more appropriate to the project specific analytical needs, and that are cost-effective.

2.0 SCOPE OF DOCUMENT

This document provides for the policies and procedures required to successfully analyze for a wide range of environmental contaminants using standard, non-standard, and internally developed test methods in ECCS' on-site and fixed facility laboratory operations.

2.1 Scope of Analytical Testing

Appendix A provides a list of the current ECCS test methods. Newly developed test methods not listed in Appendix A must follow the requirements of this QSM.

2.2 Client Expectations

This document provides for the policies and procedures required to meet or exceed the expectations of our clients and the regulatory community.

2.3 Table of Contents and Appendices

The organization of the document generally follows the outline of "2003 NELAC Standard, Chapter 5.0, Quality Systems."

2.4 Acronyms

Acronyms are used throughout the document and the operation of the quality system. A listing of acronyms and their associated definition are provided after the table of contents.

2.5 Definitions

Many terms are used in the operation of the quality system. A listing of terms used and their definition is provided below.

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.

Accrediting Authority: The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.

Accreditation body: Authoritative body that performs accreditation.

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.

Analyst: The designated individual who performs the "hands-on" analytical test 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: 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.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

As-is Basis: Test results that are reported on an as-received basis, meaning they have not been corrected for moisture content.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria.

Audit: A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria.

Bench Sheet: A form used to track the preparation of samples through a preparation or extraction process. It is used to document environmental conditions, initial and final volumes, who prepared the samples when, and what spikes and spike amounts were added to the samples and quality control samples. A separate bench sheet is prepared for each analytical batch.

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blind Sample: A sub-sample for analysis with a composition known to the submitter. The analyst/ laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration: Set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

- 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).
- In calibration in accordance with test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by

the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Method: A defined technical procedure for performing a calibration.

Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.

Calibration Standard: A substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM): A reference material is one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

Chain of Custody Form: A 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.

Chain of Custody: An unbroken trail of accountability that verifies the physical security of samples, data, and records.

ChemStation: A chromatography data system from Agilent used for chromatography techniques including HPLC and GC that is capable of data acquisition, integration, quantitation, and reporting.

Client: Any individual or organization for which items or services are furnished or work performed in response to defined requirements and expectations.

Conflict of Interest: A personal interest or relationship, as defined by law or regulation, that conflicts with the faithful performance of your official duty.

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original test method. These may include, but are not limited to:

- Second column confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors; or
- Additional cleanup procedures.

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.

Consensus Standard: A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.

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.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Correlation Coefficient: The quantity r, called the linear correlation coefficient, measures the strength and the direction of a linear relationship between two variables.

Coefficient of Determination, r^2: Statistical method that explains how much of the variability of a factor can be caused or explained by its relationship to another factor. Used in trend analysis, it is computed as a value between 0 (0 percent) and 1 (100 percent) higher the value, better the fit. Symbolized by r^2 because it is square of the coefficient of correlation symbolized by r, it is an important tool in determining the degree of linear-correlation of variables (goodness of fit) in regression analysis.

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Quality Objectives: Qualitative and quantitative specifications used to design a study that will limit uncertainty to an acceptable level.

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.

DataTool: An application that interfaces data acquisition software with Promium Element for the purpose of automating raw data uploads.

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 and are suitable for final decision-making.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate acceptable accuracy.

Detection: Any concentration of an analyte which equals or exceeds the laboratory's detection limit.

Detection Limit (DL): 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.

Digestion: A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form.

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Dry Weight Basis: Test results that have been adjusted for moisture content.

Duplicate: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Element: A laboratory information management system developed by Promium, Inc., used to track samples from receipt, through the extraction and analysis process, to reporting, invoicing, and disposal.

Eluent: A solvent used to carry the components of a mixture though a stationary phase.

Elute: To extract; specifically, to remove (adsorbed material) from an adsorbent by means of a solvent.

Elution: A process in which solutes are washed though a stationary phase by the movement of a mobile phase.

Enforcement Standard (ES): A numerical value expressing the maximum concentration of a substance in groundwater which is adopted under s. 160.07, Stats., and s. NR 140.10 or s. 160.09, Stats., and s. NR 140.12. These standards are toxicologically derived to protect human health. Analytical values above the ES trigger the procedure prescribed in s. NR 140.26. (State of Wisconsin Term)

Environmental Data: Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.

Environmental Monitoring: The process of measuring or collecting environmental data.

Enviroquant: The environmental quantitation module associated with the Agilent MSD Productivity ChemStation used for mass spectral interpretation. It is part of the complete chromatography data system that controls instrument conditions for a GC/MS and enables data acquisition, integration, quantitation, and reporting.

Extraction: A preparation step for samples that removes the target analytes from the sample matrix and exchanges them into a solvent.

False Negative: An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.

False Positive: An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

Finding: 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.

Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

Homologue: One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, CH3OH (methanol), C2H5OH (ethanol), C3H7OH (propanol), C4H9OH (butanol), etc., form a homologous series.

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.

Interference, spectral: Occurs when particulate matter from the atomization scatters the incident radiation from the source or when the absorption or emission of an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.

Interference, chemical: Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.

Internal Standard: 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 test method.

International System of Units (SI): The coherent system of units adopted and recommended by the General Conference on Weights and Measures.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.

Instrument Detection Limit (IDL) is the concentration equivalent to a signal, due to the analyte of interest, which is the smallest signal that can be distinguished from background noise by a particular instrument. The IDL should always be below the **method detection limit**, and is not used for compliance data reporting, but may be used for statistical data analysis and comparing the attributes of different instruments. The IDL is similar to the "critical level" and "criterion of detection" as defined in the literature.

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: A body that calibrates and/or tests.

Laboratory Control 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.

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Legal Chain of Custody Protocols: Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. An LOD is analyte and matrix-specific and may be laboratory dependent.

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.

Linear Calibration Range (LCR): The region of a calibration curve within which a plot of the concentration of an analyte versus the response of that particular analyte remains linear and the correlation coefficient of the line is approximately 1 (0.995 for most analytes). The plot may be normal-normal, log-normal, or log-log where allowed by the analytical test method. At the upper and lower bounds of this region (upper and lower limits of quantitation), the response of the analyte's signal versus concentration deviates from the line.

Manager: The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.

Management: Those individuals directly responsible and accountable for planning, implementing, and assessing work.

Management System: System to establish policy and objectives and to achieve those objectives.

Matrix: The substrate of a test sample

Field of Accreditation Matrix: These matrix definitions shall be used when accrediting a laboratory.

- Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- Non-Potable Water: Any aqueous sample excluded from the definition of Drinking Water matrix. Includes surface water, groundwater, effluents, water treatment chemicals, and TCLP or other extracts.
- Solid and Chemical Materials: Includes soils, sediments, sludges, products and by-products of an industrial process that results in a matrix not previously defined.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped in accordance with the origin.

- 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. (NELAC)

NIST Library: A mass spectral database containing nearly 108,000 spectra (NIST 98 version) mostly from quadrupole mass spectrometers, used to match acquired spectra with known standards through a mass spectral search program.

Quality System Matrix: These matrix definitions are an expansion of the field of accreditation matrices and shall be used for purposes of batch and quality control requirements. These matrix distinctions shall be used:

- Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
- Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous Liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped in accordance with the origin.
- Solids: Includes soils, sediments, sludges and other matrices with > 15% settleable solids.
- Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
- 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.

Matrix Spike: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a test method's recovery efficiency.

Matrix Spike Duplicate: A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Maximum Contaminant Level (MCL): A numerical value expressing the maximum permissible level of a contaminant in water which is delivered to any user of a public water system.

Maximum Contaminant Level Goal (MCLG): The maximum level of a contaminant in drinking water at which no known or anticipated adverse effect on the health of persons would

occur, and which allows an adequate margin of safety. Maximum contaminant level goals are non-enforceable health goals.

May: Denotes permitted action, but not required action.

Measurement Quality Objectives (MQO): The desired sensitivity, range, precision, and bias of a measurement.

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 Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: One way to establish a detection limit, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Method of Standard Additions: A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.

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 Accreditation Database: The publicly accessible database listing the accreditation status of all laboratories participating in NELAP.

National Environmental Laboratory Accreditation Conference (NELAC): A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP.

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which NELAC is a part.

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).

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Nonconformance: An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

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: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

Preventive Action Limit (PAL): The Wisconsin Preventative Action Limit is a numerical value expressing the maximum concentration of a substance in groundwater which is adopted under s. 160.15, Stats., and s. NR 140.10, 140.12 or 140.20. Reported values above the PAL trigger the procedure prescribed in s. NR 140.22. The PAL is typically set at 1/10th of the enforcement standard if the substance is carcinogenic, mutagenic, and teratogenic or has a synergistic effect. The PAL is 20% of the enforcement standard for other substances of public health concern.

Procedure: A specified way to carry out an activity or process. Procedures can be documented or not.

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Testing Oversight Body/Proficiency Testing Provider Accreditor (PTOB/PTPA): An organization with technical expertise, administrative capacity and financial resources sufficient to implement and operate a national program of PT provider evaluation and oversight that meets the responsibilities and requirements established by NELAC standards.

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 Study Provider: Any person, private party, or government entity that meets stringent criteria to produce and distribute NELAC PT samples, evaluate study results against published performance criteria and report the results to the laboratories, primary accrediting authorities, PTOB/PTPA, and NELAP.

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

Quantitation Range: The range of values in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard. The quantitation range lies within the calibration range.

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

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.

Reference Material: A material or substance with 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 Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Replicate Analyses: The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.

Reporting Limit: A client-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix. The default reporting limit is equal to the lowest calibration standard in an analytical run.

Requirement: Denotes a mandatory specification; often designated by the term "shall".

Retention Time: The time between sample injection and the appearance of a solute peak at the detector.

Sample: Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis

Sampling and Analysis Plan (SAP): See Quality Assurance Project Plan

Second Source Calibration Verification (ICV): A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.

Selectivity: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.

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.

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.

Sequence: A list of samples in order of injection processed on an instrument. A sequence is also referred to as an analytical run.

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.

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.

Spike: A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: (Document) The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.

Standard: (Chemical) Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by the US National Institute of Standards and Technology (NIST) and characterized for absolute content, independent of analytical test method.

Standard Method: A test method issued by an organization generally recognized as competent to do so.

Standard Operating Procedure (SOP): 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.

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 test method.

Statistical Outlier: **A**n observation or data point that appears to deviate markedly from other members of the population in which it occurs. The presence of outliers must be verified using an approved statistical method, at the 1% significance level for Wisconsin compliance.

Supervisor: The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Target Analytes: Analytes specifically named by a client (also called project-specific analytes).

Technical Director: Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.

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 in accordance with a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.

Test Method: An adoption of a scientific technique for performing a specific measurement as documented in a laboratory SOP or as published by a recognized authority.

Testing Laboratory: Laboratory that performs tests.

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the test method.

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.

Work Order: A group of samples logged in together under one or more chain of custodies for a given client and project. A work order may be combined with other work orders to form a sample delivery group.

3.0 ORGANIZATION

ECCS is a legally identifiable organization. Through application of the policies and procedures outlined in this manual, ECCS assures that it is impartial and that personnel are free from undue commercial, financial, or other pressures that might influence their technical judgment. ECCS is responsible for carrying out on-site and fixed facility laboratory services that meet the needs of the client, the requirements of various regulatory agencies and codes, and other accrediting authorities.

3.1 Overview

ECCS is a small commercial laboratory business that operates on-site and fixed facility laboratory services operations from facilities located at 2525 Advance Road in Madison, Wisconsin. The fixed laboratory facility provides analytical services, primarily pesticide residue testing services, for the AgChem marketplace. ECCS on-site laboratory services are provided from its fleet of mobile laboratories, and from custom on-site facilities designed to meet our client-specific analytical testing needs.

3.2 ECCS Formal Management Structure

ECCS formal management structure includes the titles president, operations manager (OM), and quality manager (QM). A formal operations team also exists that includes the formal managers listed above plus other key staff including representatives from project management, discipline team leaders, and office/human resources (HR). Other key support roles include the LIMS manager and purchasing. This management structure operates seamlessly whether work is carried out in its fixed facility, or at sites away from its fixed facility including mobile or temporary on-site testing facilities. See Appendix F for job descriptions.

3.3 Technical Discipline Team Leaders

ECCS has established technical discipline team leaders for the categories GC/MS, GC/HPLC, and metals/inorganics. The team leaders have direct technical and training responsibility for staff that works on test methods within the technical discipline.

3.4 Lead Chemist

The Lead Chemist is central to a successful, high quality on-site laboratory project. Senior chemists are authorized by management to accept the role of Lead Chemist to be responsible for each on-site testing project that occurs. The diagram in Appendix B depicts the relationship of various operational sectors and the Lead Chemist during the execution of an on-site laboratory services project.

3.5 Organizational Chart

An organizational chart provided in Appendix C is current as of February 1, 2013. The assignment of responsibilities, authorities, and interrelationships of the personnel who manage, perform, or verify work affecting the quality of environmental tests is documented in Section 16, Personnel of this manual.

Policy

Management has overall responsibility for the technical operations and authority needed to generate the required level of quality for its laboratory operations.

Policy

Management's commitment to quality and to the QS is stated in the Quality Policy Statement (See Section 4.0), which is upheld through the application of related policies and procedures.

Policy

Management ensures technical competence of personnel operating equipment, performing tests, evaluating results, or signing reports, and limits authority to perform laboratory functions to those appropriately trained and/or supervised.

3.6 Maintenance of the Quality System

Management bears specific responsibility for the QS. This includes defining roles and responsibilities to personnel, approving documents, providing required training, providing a procedure for confidential reporting of data integrity issues, and periodic review of data, policies and procedures, and documentation practices.

3.7 Audit Findings and Corrective Actions

Management ensures that audit findings and corrective actions are completed within required time frames.

3.8 Designated Alternates

Designated alternates are appointed by management during the absence of key management staff if the absence is more than 15 days. If the key management staff absence exceeds 65 consecutive calendar days, the primary accrediting authority shall be notified in writing.

3.9 Position Descriptions

Management has defined the minimum level of education, qualifications, experience, and skills necessary for all positions in the laboratory with position descriptions (See Appendix F).

3.10 Training

Training is an integral component to ECCS' QS and records are maintained as described in Section 18.0, Training.

4.0 QUALITY SYSTEM

4.1 Quality Policy Statement

The objective of the QS and the commitment of management are to consistently provide our clients with data of known and documented quality that first and foremost meets the specific needs of their projects. ECCS uses good professional practices to continually improve quality, provide the highest quality of service to our clients, and comply with the various regulatory and accrediting authority standards as appropriate. This policy is implemented and enforced through the unequivocal commitment of management to the principles, practices, and ethics policies and procedures outlined in this QSM and associated SOPs.

4.2 Management Commitment Statement

Top management is committed to continual improvement in the company's service to its clients through the use of the quality policy statement and objectives, audit results, analysis of data, corrective and preventative actions, and management review. As a result, it has implemented and maintained management and quality systems that are appropriate to the scope of its business activities including:

- QSM and associated SOPs,
- Employee development plans,
- Training/retraining exercises,
- Project closeout meetings with subsequent staff discussion, and
- Continual management and quality review that occur as a result of weekly operations team meetings and weekly staff meetings.

4.3 Quality Systems

The company's QS is documented in this QSM and associated documents that collectively describe the policies, procedures, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of the organization for ensuring quality in its work processes, products, and services. The QSM is maintained current and up-to-date by the QM.

Policy

Management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work

4.4 Quality Manager

The QM serves as the focal point for implementation of the QS described in this QSM. The QM has the authority and/or responsibility to:

- be the focal point for the QS and have oversight of quality control data;
- use available tools, such as audit and surveillance results, control charts, PT results, data analysis, corrective and preventative measures, customer feedback, and management reviews to monitor laboratory practices to implement, maintain, and improve the QS;
- keep the QSM current;
- ensure that all staff understand their contribution to the QS;
- ensure communication takes place at all levels of the business regarding the quality system;
- evaluate data objectively and perform assessments without outside influence;
- conduct internal audits on all QSs at least annually;
- be knowledgeable about various analytical test methods and regulatory requirements including those of accrediting authorities;
- notify management and staff of audit findings and corrective actions; and
- evaluate the effectiveness of training.

Policy

Management ensures that the laboratory's policies and objectives for quality are documented by reference or by inclusion in the QSM, and that the QSM is communicated to, understood by, and implemented by all personnel concerned.

Policy

Where the QSM documents laboratory requirements, a separate SOP or policy is not required.

Procedure

All employees sign a form, kept with their training records, that states that they have read and understand the current version of the QSM and quality policy statement. FORM-GEN-009-4 of standard operating procedure (SOP) "GEN-009, Documentation of Training" is used to acknowledge that employees have read and understand quality related documents. Changes to the QS will sometimes be sent to employees via e-mail. This is especially true for employees that are assigned to on-site laboratory projects. In these instances, a return e-mail is used as documentation in lieu of FORM-GEN-009-4.

5.0 DOCUMENT MANAGEMENT

This section describes procedures for document management, which includes preparation, review, control, distribution, and revisions. The purpose of document management is to preclude the use of invalid or obsolete documents.

Policy

Documents are defined as SOPs, manuals, policy statements, regulations and standards, text books, software and other normative documents that form part of ECCS' management system.

Policy

Internal documents including this QSM are scheduled for review and updated if necessary in accordance with SOP "GEN-001, Document Preparation, Review, and Control."

5.1 Status of Documents

Documents are controlled, replaced, or archived.

- A controlled document is one that is in use, has been reviewed and approved, issued, tracked, and uniquely identified as part of the management system. Controlled documents may be internal documents or external documents.
- A replaced document is one that is out of date and has been superseded by more recent versions.
- An archived document is one that is currently not in use or has been replaced by a newer revision.

Procedure

Documents are prepared, controlled, revised, distributed, and archived in accordance with ECCS SOP "GEN-001, Document Preparation, Review, and Control." This SOP codifies the header requirements for all ECCS SOPs and the formatting of non-analytical test method SOPs.

Procedure

Analytical test method SOPs are prepared in accordance with ECCS SOP "GEN-002, ECCS Analytical Method Standard Operating Procedure (SOP) Creation and Revision Guidance."

Policy

Controlled copies of documents are available at all locations where operations are essential to the effective functions of the laboratory. A controlled copy of essential documents also exists on the server (ECCSDC) in the folder "Element Server Folders/Updates/User."

Policy

Handwritten changes to controlled documents are not allowed, except by the QM. Handwritten notes for suggested changes to the next version can be made to the original document in possession of the QM. Any change must be initialed and dated.

Procedure

A master list of controlled documents that includes distribution, location, and revision numbers is maintained by the QM in an Excel file titled "Document Tracking." The controlled document master list is updated each time a new or revised document is approved for distribution.

Procedure

Archival of documents is addressed in SOP "GEN-001, Document Preparation, Review and Control."

5.2 Electronic Document Changes

Procedure

All changes made in LIMS are tracked using the audit trail feature, including the essence of the change that was made, date, and author.

Procedure

Changes to documents are incorporated into a new revision and issued as soon as practical. Revision of documents is addressed in SOP "GEN-001, Document Preparation, Review and Control." The altered text in a new revision of a document is documented electronically in Microsoft Word and kept on file in archived drafts folders.

5.3 Standard Operating Procedures

SOPs are used to ensure consistency of application of common procedures and written to describe in detail how to accurately reproduce laboratory processes.

Policy

The laboratory has SOPs for all analytical test methods within its scope, and for procedures that are part of the QS that accurately reflect how the analytical process is performed. All master copies of SOPs are maintained in the QMs office. Where equipment manuals or published methods accurately reflect laboratory procedures in detail, a separate SOP is not required.

Procedure

SOP "GEN-002, Guidance for Preparation of Analytical Method Standard Operating Procedures (SOP)" provides the procedure for creation and content of analytical test method SOPs.

Procedure
SOP "GEN-001, Document Preparation, Revision, and Control" provides the procedure for creation and content of non-analytical method SOPs and documents.

Policy

Copies of all SOPs and documents are accessible to all personnel.

Policy

ECCS' core business often requires modifications to SOPs to meet project and client specific objectives. Any deviation from a test method is documented, including both a description of the change made and a technical justification.

Policy

Deviations from SOPs require documentation on a corrective action form, on the chain of custody forms, in LIMS, or in field logbooks, as applicable.

6.0 REVIEW OF REQUESTS, TENDERS, AND CONTRACTS

Policy

The review of all new work assures that oversight is provided so that requirements are clearly defined, and the organization has adequate resources, capability, and test methods applicable to the customer's needs. This process assures that all work will be given adequate attention to avoid pitfalls that may compromise data quality.

6.1 Procedure for the Review of Work Requests – Fixed Facility

The OM determines if the laboratory has the necessary accreditations, resources, including schedule, equipment, deliverables, and personnel to meet the work request.

The project assistant (PA) or the project manager (PM) informs the client if the review of the work request indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily.

The client is informed of any deviation from the work request including the test method or sample handling processes. All differences between the request and the final contract are resolved and recorded before any work begins. It is necessary that the work request be acceptable to both the laboratory and the client.

The review process is repeated when there are amendments to the original contract by the client. Project personnel are given copies of the amendments if the amendment is applicable to their specific task. Copies of all amendments are maintained in the project file.

6.2 Procedure for the Review of Work Requests – On-Site Laboratories

Each client request for on-site laboratory work follows the procedure listed above and in greater detail provided in SOP "GEN-013, On-Site Testing."

7.0 SUBCONTRACT LABORATORY

A subcontract laboratory is defined as a laboratory external to this organization that performs testing services for the organization.

Policy

When subcontracting analytical services, the laboratory assures work requiring accreditation is placed with an appropriately accredited laboratory or one that meets applicable statutory and regulatory requirements for performing the tests.

Procedure

A list of subcontractors with a copy of the accreditation certificate is maintained as evidence of compliance.

7.1 Client Notifications

The PM notifies the client of the intent to subcontract either verbally, in an e-mail, or in writing. This communication is documented.

Policy

The laboratory performing the subcontracted work is identified in the final report. ECCS assumes responsibility to the client for the subcontractor's work, except in the case where a client or a regulating authority specifies which subcontractor is to be used.

8.0 PURCHASING SERVICES AND SUPPLIES

ECCS ensures the services and supplies required to operate its business are effective.

8.1 Services

Services refer to balance calibration, Class 1 weight set calibration, service contracts for instrumentation, supply vendors, etc.

Policy

The laboratory ensures that purchased supplies and services that affect the quality of environmental tests are of the required or specified quality by using approved suppliers and products.

Policy

The laboratory has procedures for purchasing, receiving, and storage of supplies that affect the quality of environmental tests.

Procedure

The OM or designee reviews and approves requests for services and supplies and signs the authorization prior to ordering. Project managers and/or lead chemists also have that authority for on-site laboratory projects.

8.2 Suppliers

Evaluation of suppliers is accomplished by ensuring the vendor ships the product or material ordered against the purchase requisition form.

Procedure

A signature line is included on the purchase requisition to indicate that the items requested have been reviewed and meet quality specifications (like specific grades mentioned in SOPs).

8.3 Routine Supplies

Routine supplies such as solvents and bottles do not require a purchase requisition because the vendor quality has been deemed acceptable by ECCS.

Procedure

Supplies received are reconciled against the packing list and inspected for damage. Supplies, reagents, and chemical standards are checked-in and distributed to the appropriate individuals, departments or storage areas. Reagents and standards are logged into LIMS and then labeled appropriately for documentation purposes. Documentation requirements for reagents and standards include:

- date of receipt;
- expiration date, if applicable;
- source;
- lot or serial number;
- calibration and verification records; and
- certifications.

Procedure

Supplies received are stored in accordance with manufacturer's instructions, laboratory SOP, or test method specifications.

9.0 SERVICE TO THE CLIENT

Clients seek the on-site and fixed facility laboratory services that ECCS offers.

9.1 Client Communication

ECCS maintains and documents communication with clients and/or their representatives for the purposes of seeking feedback, both positive and negative, and clarifying requests. Each request is reviewed to determine the nature of the request and the laboratory's ability to comply with the request within the confines of prevailing statutes and/or regulations without risk to the confidentiality of other clients.

On-site laboratory projects require heightened upfront communication with the client and documentation to ensure that project-specific objectives are known and met. Refer to SOP "GEN-013, On-site Testing" for more information about upfront communication.

Policy

Client feedback is analyzed to improve the QS, testing activities, and service to the client.

9.2 Client Confidentiality

Policy

Information cannot be divulged or released to a third party without proper authorization from the client.

Policy

All electronic data (storage or transmissions) are kept confidential, based on current electronic technology, as required by client, ECCS policy, or regulation.

Procedure

The following notice shall accompany all e-mail and facsimile correspondence with clients:

NOTICE-- This email may contain confidential and privileged information for the sole use of the intended recipient. Any review or distribution by others is strictly prohibited. If you are not the intended recipient, please contact the sender immediately and delete all copies.

10.0 COMPLAINTS

The purpose of this section is to assure that customer complaints are addressed and corrected, if required.

Policy

All customer complaints are documented by the person receiving the complaint and addressed by appropriate personnel. If it is determined that a complaint is without merit, it is documented, and the client is contacted. If the complaint has merit, the issue is resolved.

Policy

The Project Manager or Project Assistant reviews all complaints and determines appropriate action after discussing with the QM or OM. Corrective action is initiated in cases where the complaint is the result of a laboratory error. See Section 12 for corrective action procedures.

11.0 NON-CONFORMING WORK

Non-conforming work is work that does not meet quality system acceptance criteria or the agreed to requirements of the client. Examples of non-conformance include but are not limited to quality control failures, PT failures, findings as a result of internal/external audits or management reviews, customer complaints or negative feedback, etc.

Policy

All employees should be able to identify potential non-conformance situations, determine if it will be permitted, and take appropriate action. All employees have the authority to stop work on samples when any aspect of the process does not conform to SOP or client requirements.

- The responsibilities and authorities for the management of non-conforming work are detailed in Section 12 – Corrective Action. The procedures for investigating and taking associated corrective actions for non-conformance are also described.
- The OM, QM, and/or PM evaluate the significance of identified non-conforming work, and take immediate corrective action, where warranted. If client data has been impacted, the client is notified within five working days.
- When an investigation of non-conformance indicates that the root cause of the non-conformance requires that a test method be restricted or suspended until modifications are implemented, the QM will immediately notify all personnel of the restriction/suspension.
- The OM, QM, or PM will notify staff when resumption of work is authorized.

12.0 CORRECTIVE ACTION

This section describes the policies and procedures and designated authorities for implementing corrective action when non-conforming work has been identified.

Policy

When a non-conformance is identified, action is taken to identify the potential root cause(s) and the likely corrective actions. The most likely corrective action(s) is chosen and implemented that will eliminate or prevent recurrence of the non-conformance. The implementation of corrective actions is monitored for effectiveness.

12.1 Implementation of Corrective Action

Policy

Non-conformances are documented, and if the evaluation requires cause analysis, the cause and corrective action are also documented, and the corrective action is implemented.

Procedure

Corrective action(s) for simple, non-recurring nonconformance can be recorded in raw data packages, logbooks, email, or other appropriate documents.

Procedure

A Corrective Action Form (CAF) is implemented for non-conformance that requires formal corrective action. See Appendix D to view the ECCS Corrective Action Form. Proper implementation and documentation of events leading to problem resolution allows information to be recalled to provide insight into the same or similar non-conformance at a later date.

- Analysts are responsible for initiating or recommending corrective actions and ensuring that an exceedance of quality control acceptance criteria is documented. When a quality control limit is exceeded, a systematic approach to problem resolution is taken. A CAF is initiated that documents the corrective action process to its completion. The root cause is identified and corrective action is evaluated by the analyst for the need for samples to be reanalyzed or appropriately qualified in accordance with ECCS SOP "GEN-015, Qualification of Data." The completed CAF is maintained with the data until approval by the secondary data reviewer. Once approved, the carbon copy of the CAF is routed to the QM to monitor trends and on-going analyst performance. The original is kept with the analytical run raw data file. Also, a CAF documents the disposition of any data that doesn't meet the requirements of the quality control program. Analysts implement CAFs when quality control results fail.
- The QM implements a CAF for each PT failure or finding from internal/external audits or management reviews. The root cause is evaluated by the discipline team leader and

corrective actions are implemented. Typical corrective actions include analyst training, improvements to SOPs, or changes to the Quality Manual.

- When customer complaints or negative feedback are identified, if warranted, a CAF is implemented by the PM. The root cause is evaluated by the PM, QM or designee and corrective actions are implemented. Typical corrective actions may include correcting data and re-issuing a report, or discovery of a lesson learned that is used for staff training for future project work.
- Where uncertainty arises regarding the analysis of the root cause or appropriate corrective action for a test method nonconformance, the technical discipline team leader is consulted to determine the appropriate corrective actions to be initiated.

Procedure

Analysts routinely implement corrective actions for data with unacceptable quality control measures. First level correction may include re-analysis without further assessment. If the test method SOP addresses the specific actions to take, they are followed. Otherwise, A CAF must be initiated.

Procedure

Data reviewers and the QM review CAFs and suggest improvements, alternative approaches, and procedures where needed. The review of CAFs is performed prior to the data being reported.

Procedure

If the data to be reported are affected adversely by the non-conformance, the data are qualified and a case narrative is included with the final report.

Procedure

The discovery of a non-conformance for results that have already been reported to the client must be immediately evaluated for the significance of the non-conformance, its acceptability to the client, and determination of the appropriate corrective action. If the data reported to the client are affected adversely by the non-conformance, the client is notified within 5 working days.

12.2 Training Associated With Corrective Actions

Policy

Changes to SOPs, the QM, or other policies or procedure that occur as a result of a corrective action require staff training.

Procedure

Training following changes to SOPs, the QM, or other policies or procedures as a result of corrective action is required and generally occurs at regularly scheduled Wednesday staff

meetings and operations team meetings.

12.3 Monitoring of Corrective Action

Policy

The QM or designee will monitor implementation and documentation of the corrective action to assure that the corrective actions were effective.

Procedure

The QM or designee is responsible to monitor corrective actions.

- Routine CAFs are routed to the QM for further review. Findings are summarized and when necessary, the QM will provide necessary training or reminders at regularly scheduled Wednesday staff meetings and operations manager meetings.
- Corrective actions that were implemented as a result of internal/external audits and management reviews are monitored and findings documented within the associated findings report.
- PMs are required to follow up as necessary with clients who have voiced complaints.

12.4 Exceptionally Permitting Departures from Documented Policies and Procedures

An example of a planned departure is: The initial sample volume or final extract volume is adjusted to meet project reporting limit requirements. This change to the SOP requirements would be documented in the project file and in the case narrative as an exception to our SOP.

Policy

The laboratory allows the release of non-conforming data that are considered exceptional departures from documented policies or procedures only with approval of the QM or designee on a case-by-case basis. Project specific planned departures from procedures or policies are documented in the project file and do not require further approval.

13.0 PREVENTIVE ACTION

Preventive action includes, but is not limited to, review of quality control data to identify quality trends, discussion of quality related issues at the regularly scheduled staff meetings, annual budget reviews, annual managerial reviews, scheduled maintenance, routine preventive maintenance, validating LIMS and spreadsheet calculations, and other actions taken to prevent problems. Preventive action, rather than corrective action, aims at minimizing or eliminating inferior data quality or other non-conformance through scheduled maintenance and review, before the non-conformance occurs.

Policy

When improvement opportunities are identified or if preventative action is suggested, action plans are developed, implemented, and monitored to reduce the likelihood of or recurrence of previous nonconformances.

Procedure

The QM initiates and monitors preventative actions when improvement opportunities arise.

Policy

All employees should be actively involved in performing preventive maintenance on their instruments. Preventive maintenance is addressed in the laboratory test method SOPs and associated procedures. Employees also have the authority and responsibility to recommend new or additional preventive action procedures. Management is charged with implementing preventive actions to improve laboratory operations, data quality, and maintain laboratory equipment.

Policy

Preventive maintenance to all instruments is documented in the instrument maintenance logs.

Policy

The QM or his designee periodically reviews logbooks and documents the review by initialing and dating the logs.

14.0 CONTROL OF RECORDS

Records are a subset of documents, usually data recordings that include annotations, such as daily refrigerator temperatures posted to a laboratory form, lists, spreadsheets, or analyst notes on a chromatogram. Records may be on any form of media, including electronic and hard copy. Records allow for the historical reconstruction of laboratory activities related to sample-handling and analysis.

Policy

The laboratory maintains a record system appropriate to its needs, documents all laboratory activities, and complies with applicable standards or regulations as required.

Procedure

The laboratory retains all original observations, calculations and derived data, calibration records, and reports for a minimum of five years. For the state of Louisiana these records will be retained for 10 years. Observations, data, and calculations are recorded at the time they are made.

Policy

When mistakes are made in technical records, each mistake is crossed out with a single line (not erased, made illegible, or deleted) and the correct value entered alongside. Corrections are signed or initialed and dated by the person making the correction. For electronic systems, all changes are tracked by the audit trail or by added notes. When changes are made to technical records for reasons other than for correction of transcription errors, the reason for the change is recorded on the document.

Policy

Records of all procedures to which a sample is subjected while in the possession of the laboratory are kept.

14.1 Raw Instrument Data

Policy

Raw instrument data are stored electronically, when possible, so that a complete trail of data reports before any manual manipulation or integration can be reproduced. The electronic data in original or reprocessed format are considered the official record. Instrument sequence data packages contain the final processed data used to calculate the results presented in the final report. Where available in the instrument software, all electronic tracking and audit functions are enabled. Where raw data cannot be stored electronically, hard copy is maintained.

14.2 Records Management and Storage

Policy

Records, including electronic records, are retrievable, legible, protected from deterioration or damage, and held secure and in confidence.

Policy

The QM shall maintain a log of names, initials, and signatures for all individuals responsible for signing or initialing any hard copy laboratory record. LIMS also maintains a complete audit trail of changes made by any staff member.

Policy

The laboratory maintains a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting.

Policy

Archived information and access logs are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

Policy

In the event that the laboratory transfers ownership or goes out of business, records will be maintained or transferred in accordance with client instructions.

Procedure

ECCS network drives are backed-up on a daily basis. Access to protected records is limited to ECCS staff to prevent unauthorized access or amendment.

14.3 Chain of Custody

Chain of Custody (COC) is the documentation trail of sample possession that accompanies samples submitted to the laboratory. Documentation is critical to providing legal evidence to support the maintenance of sample integrity. Authoritative sources suggest the following guidelines. A sample is under custody if:

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

See Appendix E for the ECCS COC.

Procedure

SOP "GEN-003 Chain-of-Custody, Log-in, Tracking Procedures and Sample Containers" provides ECCS COC procedures.

14.4 Evidentiary Chain of Custody Records

Evidentiary COC records including complete custody tracking of a sample throughout the laboratory testing process can be used as legal evidence. At the time of this revision, the laboratory has not handled evidentiary samples. Therefore, this subject is currently not applicable.

15.0 AUDITS AND MANAGEMENT REVIEW

The QS requires continuous monitoring by the QM and periodic review by executive management.

15.1 Types of Audits

Audits measure laboratory performance and verify compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the QS. They are also instrumental in identifying areas where improvement in the QS will increase the reliability of data. Audits are of four main types: internal, external, performance, and management reviews.

15.2 Internal Audits

Policy

The laboratory conducts internal audits encompassing its total QS at least annually. Audits are carried out by trained and qualified personnel independent of the area to be audited, except when it can be demonstrated that an effective audit will be carried out.

Procedure

Annually, the QM prepares a schedule of internal audits to be performed during the year. These audits are conducted to verify compliance with all requirements of the QS and accrediting authority Standards. Internal audits are conducted in accordance with SOP "QAU-001, Internal Audits." Four types of internal audits are conducted including:

- internal audits of the QS
- data audits,
- on-site testing audits, and
- on-site testing project closeout audits.

15.3 Internal Audits of the QS

Procedure

The QM conducts internal audits that encompass the total requirements of the QS on at least a yearly basis in accordance with SOP "QAU-001, Internal Audits."

15.4 Data Audits

Policy

Data audits are conducted on approximately 10% of raw data packages in accordance with SOPs "QAU-001, Internal Audits" and "GEN-016, Data Review Procedures." The data audits are scheduled to include data from both the fixed-based lab and on-site testing projects and each test method. Data audits are an essential part of the laboratory's ethics and data integrity review processes.

15.5 On-Site Testing Audits

Policy

On-site testing audits are conducted on approximately 10% of the on-site testing projects in accordance with SOP "QAU-001, Internal Audits."

15.6 On-site Testing Project Closeout Audits

Policy

On-site testing project closeout audits are conducted on approximately 10% of the on-site testing projects in accordance with SOP "QAU-001, Internal Audits."

15.7 External Audits

Policy

It is the laboratory's policy to cooperate and assist with all external audits, whether performed by clients or an accrediting authority.

Policy

All external audits are fully documented and tracked to closure.

Procedure

Management ensures that all areas of the laboratory are accessible to auditors as applicable and that appropriate personnel are available to assist in conducting the audit. Any findings related to an external audit follow corrective action procedures. Management ensures that corrective actions are carried out within the timeframe specified by the auditor(s).

15.8 Performance Audits

Performance audits may be proficiency test samples, internal single-blind samples, double-blind samples through a provider and/or client, or anything that tests the performance of the analyst and test method.

The policy and procedures for proficiency test samples are discussed in Section 25.4.

15.9 Management Reviews

Policy

The President and the management team of ECCS reviews the QS on at least a yearly basis. The QM maintains records of the management review findings and any resulting actions.

Procedure

All components of the QS are reviewed over the course of each year, and findings are recorded. Management review of QS items will take place at one or more of the regularly scheduled operations team meeting. The reviews will be planned so that each item listed below will be addressed at least once per year. The management team assures that actions are performed within agreed time frames. The following table provides a list of review items and associated responsibilities.

Review Item	When Reviewed	How Reviewed	How Documented
Suitability of policies and procedures	Annually	Special meeting of Ops team	Memo to file and presented to entire team
Reports from managerial personnel	Annually	Annual reports from fleet manager, EH&S manager, QA manager, and team leader addressed to Ops Manager and President	Annual report from each functional manager discussing (1) key lessons learned from previous year, (2) following year priorities, (3) following year budget.
Outcome of recent internal audits	As conducted	Findings (written report) are presented by the QA manager to the Ops Team, and action items and corrective actions are distributed. Findings are also presented to the entire team in a subsequent Wednesday meeting.	Written report to President
Corrective and preventive actions	Real-time	QA manager reviews corrective actions in real time and discusses with Ops Manager.	Spreadsheet tracking corrective actions: "Audits-corrective actions"

Review Item	When Reviewed	How Reviewed	How Documented
Assessments by external bodies	As conducted	Findings (written report) are presented by the QA manager to the Ops Team, and action items and corrective actions are distributed. Findings are also presented to the entire team in a subsequent Wednesday meeting.	External entities document assessment. ECCS formally responds in writing.
Results of inter-laboratory comparisons or Proficiency Tests	As conducted	QA manager reviews PT results and discusses findings with Ops team. "Failures" are discussed and CAFs are prepared as needed.	CAF
Changes in the volume and type of the work	Weekly	Monday Ops Meeting and Wednesday meeting.	Wednesday meeting minutes
Client feedback Complaints	As available	Project manager seeks client feedback whenever possible. For on-site testing projects, ECCS has a formal client interview process in person whenever possible initiated by sales and marketing or client services representative.	To extent possible and available, feedback is included in Project Closeout Form (GEN-013-5). Alternatively, client e-mails are saved by President and shared with team as appropriate. Any corrective actions are tracked in spreadsheet: "Audits-corrective actions." On-site testing project feedback interviews are copied to the project folder and results shared with entire team.
Recommendations for improvement	As conducted	Team leaders offer recommendations for improvement during Monday Operations Team meetings	Meeting notes

16.0 PERSONNEL

ECCS has a unique personnel culture because of its seamless on-site and fixed facility laboratory operations. Some personnel have day to day position descriptions that remain the same. Others have several position descriptions or roles based on their current work assignment.

16.1 Position Descriptions

Full position descriptions are found in Appendix F.

President

The President provides leadership to position the company at the forefront of the industry; develops a strategic plan to advance the company's mission and objectives and to promote revenue, profitability and growth as an organization; leads the marketing/sales efforts; and oversees company operations to ensure production efficiency, quality, service, and cost-effective management of resources.

Operations Manager

The operations manager manages operational issues associated with all on-site and fixed facility laboratory activities.

Quality Manager

The quality manager has the authority and responsibility for ensuring that the QS is implemented and followed. The QM has direct access to the OM but is independent of operations.

Discipline Team Leader

The discipline team leader role description at ECCS is synonymous with the NELAC term "Technical Director." Team leaders are responsible for the technical oversight and training of their respective teams including development and implementation of relevant SOPs. The three team leader roles at ECCS are:

- GC/MS
- GC and HPLC
- Inorganics and wet chemistry

Lead Chemist

The lead chemist provides for the day-to-day operations management of ECCS' on-site laboratory projects. The lead chemist determines project technical requirements; prepares necessary equipment, supplies and vehicles (if required); conducts/supervises (when required) on-site testing operations; decommissions the project's on-site laboratory unit; prepares the final report; and provides feed back to the laboratory management.

Chemists

Chemists provide solution-oriented and quality objective-oriented service to our clients; have the skills to be the entire laboratory staff (for example, in an on-site laboratory environment); provide for technical training of staff; and are the primary day-to-day analysts in the on-site and fixed facility laboratories.

Client Services Personnel

The project manager provides support to sales/marketing staff in design and procurement of new projects; program management of all projects assigned.

The project assistant role provides for the day-to-day cradle to grave peripheral support of on-site and fixed facility laboratory projects by working with the project manager, the OM, and/or lead chemists to coordinate typically non-technical logistical issues during project planning and initiation; raw data downloads to LIMS; final report preparation; file review and closure; and client feedback episodes.

17.0 DATA INTEGRITY AND ETHICS

Data integrity is the result of the processes that together assure valid data of known and documented quality. Data integrity and ethics procedures in the laboratory include training, signed and dated data integrity documentation for all laboratory employees, periodic monitoring of data integrity, and documented data integrity procedures.

Policy

ECCS management adheres to a zero-tolerance policy for improper, unethical, or illegal actions.

Policy

Data integrity procedures are reviewed annually and are updated as needed. Data integrity procedures are periodically monitored through regularly scheduled internal audits, in-depth data review, records review, or other thorough check processes.

Procedure

SOP "GEN-008, Laboratory Ethics and Data Integrity," provides for laboratory ethics and data integrity.

17.1 Data Integrity and Ethics Training

Policy

Data integrity training is provided for all (including temporary) employees initially upon hire and annually thereafter.

Procedure

Attendance at an initial data integrity training (part of new employee orientation) and the annual refresher training is recorded with a signature attendance sheet or other form of documentation that demonstrates all staff members have participated and understand their obligations related to data integrity.

17.2 Detecting and Deterring Improper, Unethical or Illegal Actions

Procedure

SOP "GEN-008, Laboratory Ethics and Data Integrity," provides for procedures to detect and deter improper, unethical or illegal actions.

18.0 GENERAL TRAINING

Policy

All personnel are appropriately trained and competent in their assigned tasks before they contribute to functions that can affect data quality. It is management's responsibility to assure personnel are trained.

Policy

Only trained personnel are authorized to perform specific tasks.

Policy

SOP "GEN-009, Documentation of Training," addresses documentation of training.

Procedure

New staff members are given introductory training and orientation upon arrival. This training is documented.

18.1 Attendance

Attendance at training sessions is documented on signature sheets that are maintained on file by the QM. Training also occurs through email correspondence from the QM. Documentation occurs with a reply from the trainee that they have completed the training assignment.

18.2 Initial Training

The initial training for a new task consists of:

- All documentation involved with a new and unfamiliar task is read and understood by the trainee.
- Training is performed under the direct supervision of a qualified analyst or technician. During the time the analyst is training, the trainee may sign laboratory notebooks or logbooks, but laboratory notebooks must be cosigned by the senior analyst, who is responsible for the data generated.
- The trainee demonstrates competency in the new task before they can operate independently. The competency for a test method is accomplished by a demonstration of capability as indicated in Section 20.1. Approval of competency is noted by the initials or signature of the qualified senior analyst on the training form.
- Each step of the training process is documented.

18.3 Ongoing Training

Ongoing training consists of the following:

- The employee attests, through signature, email correspondence, or equivalent that they have read, understood, and agreed to follow the latest version of the QSM and SOPs.
- Annually, the analyst/technician shows ongoing proficiency in each test method they perform. Proof of acceptable on-going training is documented by the annual demonstration of capability (DOC) for each analyst and each test method. See SOP "GEN-021, Demonstration of Capability (DOC) Determinations," for the initial and ongoing DOC procedures**.**
- Other training as determined by management.

19.0 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

Policy

ECCS laboratory facilities are designed and organized to facilitate testing of environmental samples. Environmental conditions are monitored to ensure that conditions do not invalidate results or adversely affect the required quality of any measurement.

Policy

In the event of a power failure or other interruption of normal laboratory operations, all criteria of the analytical test method SOP (instrument tuning, calibration, etc.) must be demonstrated prior to resuming sample analysis. All test method requirements shall apply.

Policy

Environmental tests are stopped when the environmental conditions jeopardize the results.

Policy

Access to, and use of areas affecting the quality of the environmental tests is controlled by restricting areas to authorized personnel only.

Policy

The laboratory work spaces are adequate for their use, and appropriately clean to support environmental testing and ensure an unencumbered work area.

Procedure

Laboratory space is arranged to minimize cross-contamination between incompatible areas of the laboratory. In the fixed facility, volatile organics analysis is performed and samples and sample extracts are stored in a separate building, away from the main lab where solvent extractions occur to prevent contamination.

Policy

When a sample(s) is suspected to contain high concentrations of target analytes, great care is taken to ensure cross contamination does not occur.

Procedure

When a sample(s) is suspected to contain high concentrations of target analytes, a note is added to the comment section in LIMS. In the both the VOC and SVOC area, known highly contaminated samples are segregated from other samples in a separate area of cold storage.

Procedure

If the laboratory conditions are required to be controlled by test method or regulation, the adherence is recorded.

19.1 Availability of Equipment and Reference Materials

All equipment and reference materials required for the accredited tests are available in the laboratory. Test method SOPs list all equipment and supplies required for completion of accredited tests. Records are maintained for all equipment, reference measurement materials, and services used by the laboratory.

Policy

Reference materials traceable to national standards of measurement or to national standard reference materials are stored away from heavy use areas. Certificates of Traceability are available for thermometers, balances, and Class 1 weights. Each certificate obtained must be endorsed by the recognized Accreditation Body's symbol. The reference materials are used only for calibration to maintain the validity of performance.

Policy

The laboratory uses NIST traceable digital thermometers for all temperature measurements.

Procedure

The NIST traceable digital thermometers are either re-certified or purchased new on a yearly basis. The re-certified thermometers are checked using a certified NIST traceable thermometer in accordance with "GEN-010, Thermometer Policy and Calibration Checks." The data for re-certified thermometers are recorded in a logbook.

20.0 TEST METHODS AND METHOD VALIDATION

Policy

A new test method is validated before it is put into use. All new test method's validations are documented.

20.1 Demonstration of Capability

A DOC is a procedure to establish the ability of the analyst to generate data of acceptable accuracy and precision. See SOP "GEN-021, Demonstration of Capability (DOC) Determinations," for initial and ongoing DOC procedures.

Policy

DOCs that are prepared by a technician and then analyzed by an analyst are applied to the training files of each individual. The sample preparation applies to the technician and the analysis applies to the analyst.

Policy

New or inexperienced technicians/analysts work under the direct supervision of an experienced analyst who has completed acceptable DOCs.

Policy

The laboratory confirms that it is capable of generating data of acceptable accuracy and precision for all analytes with each test method before employing them.

Procedure

Initial DOCs are documented and approved by the OM or the QM. Completed forms are kept in the training files for each analyst. DOCs are performed for each analyte whenever the method, personnel, analytes, or instrument type is changed. DOCs are performed in accordance with SOP "GEN-021, Demonstration of Capability (DOC) Determinations."

On-Going DOC

On-going DOCs are demonstrated at least annually in accordance with "GEN-021, Demonstration of Capability (DOC) Determinations."

20.2 On-site Laboratory Project Requirements

On-site laboratory projects require an initial DOC before sample results can be reported. When the members of the on-site laboratory change, the new members must demonstrate proficiency as described above in Section 20.1.

20.3 Initial Test Method Evaluation

Initial test method evaluation involves the determination and documentation of the calibration range, interferences, LOD, confirmation of the LOQ, an evaluation of precision and bias, and an evaluation of the selectivity of the test method.

Procedure

LODs are determined in accordance with SOP "GEN-019, Limit of Detection (LOD) Determinations."

Policy

The lowest calibration standard is typically defined as the LOQ unless otherwise specified by a regulatory agency or client. LOQ precision and accuracy data are collected during LOD studies in accordance with SOP "GEN-019, Limit of Detection (LOD) Determinations."

Policy

The LOQ for water samples is adjusted for each sample based on volume of water used. The LOQ for soil samples is adjusted for the weight of soil used and percent solids.

Policy

If results are not reported below the calibration range (lowest calibration standard), the LOD determination is not required. If an LOD study is not performed, reporting unqualified concentrations less than the lowest calibration standard is not allowed.

Precision and Bias

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, and RSD based on the mean value of the replicates.

Bias is the systematic error that contributes to the difference between the mean of a statistically significant number of test results and the theoretical value.

Policy

Precision and bias are evaluated for standard and non-standard test methods.

Procedure

Precision and bias are evaluated for standard test methods through the performance of DOCs, LODs, and test method specific quality control samples.

When using non-standard, modified standard or laboratory-developed test methods, precision is compared to the criteria established by the client (when requested), the test method, or the laboratory. When established criteria are not available, the assessment of precision and bias should be based on the science of the test method and practical experience.

Selectivity

Selectivity is the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.

Policy

The laboratory evaluates selectivity through procedures defined in the test method SOPs which may include choice of detectors, mass spectral tuning, second column confirmation, retention time windows, and sample blanks.

20.4 Measurement Uncertainty

The total measurement uncertainty for a particular sample is the sum (combining the components) of the uncertainties of all of the steps of the sample collection and analytical processes, including, but not limited to, sample plan variability, spatial and temporal sample variation, sample heterogeneity, calibration/calibration check variability, extraction variability, weighing variability, and analytical system variability. Examples of measurement uncertainty currently calculated by the laboratory include:

- LCS control limits calculated and expressed at the 99% confidence level by multiplying the standard deviation obtained from 20 or more data points times 3.
- Calculation of LODs in accordance with EPA LOD guidelines.

Policy

The laboratory calculates and provides measurement uncertainty upon client request.

Procedure

The laboratory uses A2LA policy "P103b – Annex, Policy on Estimating Measurement Uncertainty for Life Sciences Testing Labs" as guidance when measurement uncertainty is requested by a client.

NOTE: Example calculation for measurement uncertainty at the concentration of the LCS: The standard deviation (SD_p) calculated during the LCS limit determination is used as an estimate of combined standard uncertainty. To use the LCS SD_p, the LCS must go through all of the steps of the analytical method. Measurement uncertainty for a defined LCS matrix = k * SD_p, where k is the coverage factor. For 95% confidence using 20 LCS results, k = 2.086. A minimum of 20 LCS results should be used. The coverage factor changes slightly based on the number of LCS data points (degrees of freedom) used. 10=2.228, 20=2.086, 40=2.021, 60=2.000, 120=1.980

20.5 Laboratory-Developed or Non-Standard Method Validation

Policy

Laboratory developed, modified standard test methods, and non-standard test methods require testing of reference standards, comparison of results to those achieved using standard test methods, and interlaboratory comparisons in addition to test method validation procedures described above.

Procedure

Method validation for EPA reference test methods consists of performing DOCs and LODs. DOCs are evaluated in accordance with SOP "GEN-021, Demonstration of Capability (DOC) Determinations." LODs are evaluated in accordance with SOP "GEN-019, Limit of Detection (LOD) Determinations."

Policy

Where applicable, the laboratory validates non-standard test methods, laboratory designed/developed test methods, standard test methods used outside their published scope, and amplifications and modifications of standard test methods to confirm that the test methods are fit for the intended use.

Policy

The range and accuracy of the values obtainable from validated test methods (*e.g.*, the uncertainty of the results, detection limit, selectivity of the test method, linearity, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object) is assessed for the intended use and whether it is relevant to the client's needs.

20.6 Control of Data

Policy

All calculations and all relevant data are subject to appropriate checks in a systematic manner.

Policy

Commercial off-the-shelf software (*e.g.*, word processing, database, data acquisition and statistical programs) used within the designed application range is considered sufficiently validated when in-house programming is not used. However, at a minimum a sample data set is used to verify automated data reduction processes.

Procedure

The laboratory assures that computers and software are protected, maintained, and secure through measures such as documentation, locked access, and control of the laboratory environment.

Procedure

The laboratory procedure to insure that reported data are free from transcription and calculation errors and that all quality control measures are reviewed and evaluated before data are reported is found in SOP "GEN-016, Data Review Procedures."

The laboratory assures that computers, user-developed computer software, automated equipment, or microprocessors used for the acquisition, processing, recording, reporting, storage, or retrieval of environmental test data are:

- documented in sufficient detail and validated as being adequate for use;
- protected for integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;
- maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data; and
- held secure including the prevention of unauthorized access to, and the unauthorized amendment of computer records.

21.0 EQUIPMENT

The laboratory provides all the necessary equipment required for the correct performance of the scope of environmental testing presented in this QSM.

21.1 General Equipment Requirements

Policy

All equipment and software used for testing and sampling is capable of achieving the accuracy required and complies with the specifications of the environmental test method as specified in the laboratory test method SOP.

Policy

Equipment is operated by authorized personnel only.

Policy

Manufacturers' manuals and maintenance/calibration logs are readily available for use by laboratory personnel.

Policy

All equipment is calibrated or checked before being placed into use to ensure that it meets laboratory specifications and the relevant standard specifications.

Policy

Test equipment, including hardware and software, are safeguarded from adjustments which would invalidate the test results by limiting access to the equipment and using password protection where possible.

Policy

Equipment that has been subject to overloading, mishandling, produced suspect results, or been shown to be defective or outside specifications is taken out of service, isolated to prevent its use, or clearly labeled as being out of service until it has been shown to function properly. If it is shown that previous tests are affected, then procedures for non-conforming work are followed.

Policy

When equipment is needed for a test that is outside of permanent control of the laboratory, the lab ensures the equipment meets the requirements of this manual prior to its use by inspecting or otherwise testing it.

Policy

Each item of equipment and the software used for testing and significant to the results is uniquely identified, and records of equipment and software are maintained in the maintenance log and/or computer system for each instrument. This information includes the following:

- identity of the equipment and its software;
- manufacturer's name, type identification, serial number or other unique identifier;
- checks that equipment complies with specifications of applicable tests;
- dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration, where applicable;
- maintenance plan where appropriate, and maintenance carried out to date; documentation on all routine and non-routine maintenance activities and reference material verifications;
- any damage, malfunction, modification or repair to the equipment;
- date received and date placed into service (if available); and
- condition when received, if available (new, used, reconditioned).

21.2 Support Equipment

Support equipment includes, balances, ovens, refrigerators, freezers, temperature measuring devices, and volumetric dispensing devices.

Policy

All support equipment is maintained in proper working order and records are kept of all repair and maintenance activities, including service calls.

Procedure

All raw data records are retained to document equipment performance. These records include logbooks, data sheets, or equipment computer files.

Procedure

All support equipment is calibrated or verified annually over the entire range of use using NIST traceable references where available. The results of the calibration of support equipment are within specifications or the equipment is removed from service until repaired.

Procedure

Support equipment such as balances, ovens, refrigerators, freezers, and water baths are checked in accordance with SOP "GEN-005, Daily Equipment Checks," to ensure they are operating within the expected range for the application for which the equipment is to be used.

Policy

Class B volumetric glassware is check for accuracy by lot before use. Class A and B glassware is checked for accuracy upon evidence of deterioration.

Policy

Mechanical volumetric dispensing equipment is checked for accuracy quarterly in accordance with SOP "GEN-014, Quarterly Calibration of Air Dispensing Pipette Devices." Bottle-top Optifix volumetric dispensers are checked prior to use for each batch and documented on the LIMS bench sheet.

Support Equipment Maintenance

Policy

Regular maintenance of support equipment, such as balances and fume hoods is conducted at least annually. Maintenance on other support equipment, such as ovens, refrigerators, and electronic thermometers is conducted on an as-needed basis.

Procedure

Records of maintenance to support equipment are documented in instrument maintenance logs. Each piece of support equipment does not necessarily have its own logbook. Maintenance logbooks may be shared with equipment that is housed in the same laboratory area. Each entry made in a maintenance logbook should be initialed and dated.

Support Equipment Calibration and Performance Checks

Calibration and performance check requirements for analytical support equipment are found in the Table 21.2 below. For analytical instrumentation, the calibration requirements are found in the SOP for each analytical test method.

Calibration date and expiration date (when recalibration is due) is recorded for equipment requiring calibration, where applicable.

21.3 Analytical Equipment

Policy

All equipment is properly maintained, inspected, and cleaned.

Procedure

Maintenance of analytical instruments and other equipment may include regularly scheduled preventive maintenance or maintenance on an as-needed basis due to instrument malfunction and is documented in instrument maintenance logs, which become part of the laboratory's permanent records. Each entry made in a maintenance logbook should be initialed and dated. Maintenance for analytical equipment is included in the SOP for each analytical test method.

Table 21.2 Calibration and Maintenance of Support Equipment

Instrument	Activity	Frequency	Documentation
Balance	1. Calibration check 2. Clean 3. Check alignment 4. Weighing	1. Annually 2. Before use 3. Before use 4. Daily before use	1. Date on balance post annual service in logbook 4. Logbook
ASTM Class 1 Weights	1. Only use for the intended purpose 2. Use plastic forceps to handle 3. Keep in case 4. Calibration check	4. Once every 5 years	4. Keep certificate in analytical balance logbook
Working Class Weights	1. Use plastic forceps to handle 2. Keep in case 3. Calibration check	3. Every 2 years	3. Logbook
Electronic Thermometers	Check calibration or replace annually	Annual check vs. current traceable or purchase new	Calibration information and certificates in the logbook or on file
Automatic or digital pipettes	Calibrate for accuracy and precision using reagent water and analytical balance	Quarterly	Bench sheet or logbook
Refrigerators and Freezers	Thermometers are immersed in liquid to the appropriate immersion line	Temperatures are recorded each day in use	1. Logbook
Bottle-top Volumetric Dispensers	1. Clean 2. Verify accuracy	1. When switching solvents. 2. Prior to each batch.	1. not required 2. LIMS bench sheet or logbook
Glass Microliter Syringes	1. Clean 2. Verify accuracy	1. Before each use 2. Upon Receipt	1. not required 2. logbook

22.0 CALIBRATION

Initial calibration (ICAL) and continuing calibration verification (CCV) are an important part of ensuring data of known and documented quality. If more stringent calibration requirements are included in a mandated test method or by regulation, those calibration requirements override any requirements outlined here or in test method SOPs. Specifics for conducting instrument calibrations are provided in test method SOPs.

Policy

ICALs are verified with a standard obtained from a second source traceable to a national standard when commercially available. If a second source is not available, a standard prepared from a separate lot may be used as long as the manufacturer can demonstrate the lot was prepared independently from other lots purchased.

Procedure

ICAL standards prepared from new stock standards are verified and documented with a second source standard. The second source is also most often used as the LCS spiking solution. LCS recovery trends are monitored to assure ongoing standards integrity. Initial and second source standards must agree within test method specified control limits.

Policy

Any samples that are analyzed after an unacceptable ICAL are re-analyzed or the data are reported with qualifiers, appropriate to the scope of the unacceptable condition.

Policy

Quantitation is always determined from the ICAL unless the test method or applicable regulations require quantitation from the continuing calibration.

Policy

The lowest calibration standard is the lowest concentration for which quantitative results can be reported without qualification except where defined by a regulatory agency or client.

Policy

The highest calibration standard is the highest concentration for which quantitative results can be reported without being qualified.

Procedure

The ICAL includes calculations, integrations, acceptance criteria, and associated statistics referenced in the test method SOP. Acceptance criteria for initial calibration are listed in the test method SOPs.

Sufficient raw data records are collected to allow reconstruction of the ICAL. These include, at a minimum, calibration date, test method, instrument, analysis date, analyte names, analyst's signature or initials, concentration and response, calibration curve or response factor, or unique equation or coefficient used to reduce instrument responses to concentration.

Under Method 8000B, corrective actions must be performed when the ICAL results are outside the test method acceptance criteria. For any ICAL that does not meet the test method criteria, the cause of the failure and any corrective action must be documented on a CAF. The analyst should review the calibration results to ensure that the problem is not associated with just one of the ICAL standards. If the problem is associated with just one of the standards, that standard may be reanalyzed and the RSD recalculated (Note: this reinjection is only allowed once). If the cause cannot be determined, and the condition resolved, the calibration curve must be re-prepared and any samples analyzed after the ICAL must be reanalyzed.

Normally standards should not be dropped from an initial calibration. If a standard to be dropped is the upper point of the curve, then the concentration of the next lower standard becomes the high calibration point. Any samples with analyte concentrations above that point must be diluted, or the analytes that are greater than the curve must be qualified as estimates.

Dropping a mid-point standard is not allowed unless there is justification (e.g., a bad purge for VOCs, internal standards were not added, an obvious syringe error, etc.) The dropping of a mid-point standard without a valid reason (i.e., simply to meet calibration curve acceptance criteria) is prohibited. If the lowest standard is dropped, the analyst has the following options: 1) raise the reporting limit up to the level of the next lowest acceptable standard, or 2) as discussed above, reinject the lowest standard later in the run and include the reinjection in the standard curve (Note: this reinjection is only allowed once). As stated above, any corrective action must be documented on a CAF that is included with the raw data.

- Results that are less than the lowest calibration standard are considered to have increased uncertainty, and are reported with qualifier codes and explained in the case narrative. See SOP "GEN-015, Qualification of Data."
- Results that are greater than the highest calibration standard are either diluted to within the calibration range or considered to be an estimate, and reported with a qualifier code and explained in the case narrative. See SOP "GEN-015, Qualification of Data."

22.1 Continuing Instrument Calibration

Policy

The validity of the initial calibration is verified prior to sample analysis by use of an initial calibration verification (ICV) standard.

Policy

The validity of continued calibration is verified during sample analysis by use of CCV standards as described in each test method SOP. Calibration shall be verified for each analyte listed in a test method, except for multi-component analytes such as Aroclors, toxaphene, chlordane, or total petroleum hydrocarbons, where a representative mixture can be used.

Policy

Corrective action is initiated for CCV results that are outside of acceptance criteria. Samples are re-analyzed and/or results are qualified in accordance with SOP "GEN-015, Qualification of Data."

Procedure

CCV requirements are specified in the test method SOPs.

- The calculations and associated statistics for continuing calibration are included or referenced in the test method SOP.
- Sufficient raw data records are retained to allow reconstruction of the CCV and the associated ICAL.

22.2 Unacceptable Continuing Calibration Verification

Policy

Corrective action is required following an unacceptable CCV. If following the corrective action, the instrument cannot produce a CCV within the acceptance criteria, a new initial calibration is performed and/or the associated data is qualified in accordance with SOP "GEN-015, Qualification of Data."

23.0 MEASUREMENT TRACEABILITY

Measurement traceability is another factor that determines the correctness and reliability of the environmental test measurements performed.

23.1 Measurement Quality Assurance

Policy

All equipment used that affects the quality of test results are calibrated prior to being used to obtain analytical results and on a continuing basis. These calibrations are traceable to national standards of measurement where available.

Policy

If traceability of measurements to The International System of Units (SI) is not possible or not relevant, evidence for correlation of results through inter-laboratory comparisons, proficiency testing, or independent analysis is provided.

Procedure

All equipment that affects the quality of test results is calibrated in accordance with the minimum frequency suggested by the manufacturer, by regulation, by test method, or as needed.

Reference Standards

Reference standards are standards of the highest quality available at a given location, from which measurements are derived.

Policy

Reference Standards, such as ASTM Class 1 weights, are used for calibration only and for no other purpose unless it is shown that their performance as reference standards will not be compromised.

Procedure

Reference standards, such as ASTM Class 1 weights, are calibrated by an entity that can provide traceability to national or international standards. Class 1 weights are sent out every five years for calibration to a national standard.

Procedure

Working class weight sets are sent out biennially for a calibration check in accordance with "GEN-024, Working Class Weight Set Calibration".

23.2 Reference Materials

Reference materials are substances that have concentrations that are sufficiently well established to use for calibration or as a frame of reference.

Policy

Reference materials, where commercially available, are traceable to national standards of measurement, or to Certified Reference Materials, usually by a Certificate of Analysis.

Policy

Internal reference materials, such as working standards or intermediate stock solutions, are checked as far as technically and economically possible.

Procedure

Purchased reference materials require a Certificate of Analysis where available. Otherwise, purchased reference materials are verified by application to a certified reference material, inter-laboratory comparison, and/or demonstration of capability.

Procedure

Internal reference materials, such as working standards and intermediate stock solutions, are checked by comparison with second source standards or materials that have been compared to second source standards.

Procedure

Thermometers are checked at two temperatures that bracket the target use temperature against a NIST certified thermometer or are replaced annually.

23.3 Transport and Storage of Reference Standards and Materials

Policy

The laboratory handles and transports reference standards and materials in a way that protects their integrity.

Procedure

Reference standards and materials are protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials.

Policy

Reference standards and materials are stored in accordance with manufacturer's recommendations and separately from working standards or samples.

Policy

On-site laboratory standards are transported under cool/refrigerated conditions. In an on-site laboratory it may be necessary to store working standards in the same refrigerator/freezer as unknown samples. However, the unknown samples are typically prepared and/or analyzed upon receipt and prior to storage in the refrigerator. Where possible the standards are stored in a separate area.

23.4 Labeling of Reference Standards, Reagents, and Materials

Policy

Reference standards and materials are tracked from purchase/receipt, and storage through disposal with the LIMS and with associated labeling.

Policy

Reagent quality is verified during routine blank analysis and is tracked by manufacturer's lot number. Reagents are tracked from purchase/receipt by the LIMS and with associated labeling.

Procedure

The procedures for receipt, documentation, handling, and disposal of reference standards are addressed in SOP "GEN-004, Analytical Standards and Reagents."

24.0 SAMPLE MANAGEMENT

While ECCS does not participate in the collection of samples and has little control over its client's sampling activities, documented sample management techniques are important in maintaining the validity of the sample while in the confines of its laboratories.

24.1 Sample Receipt

Procedure

When samples are received at the laboratory, their condition is documented. They are given unique identifiers. And they are logged into the ECCS LIMS system. See SOP "GEN-003, Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers."

24.2 Sample Acceptance

Policy

For the minimum conditions a sample must meet upon receipt, see SOP "GEN-003, Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers." If these conditions are not met, the client is contacted prior to any further processing.

The sample acceptance policy, including specific instructions, is provided to sample collection personnel with the bottle order. These instructions emphasize the need for use of water resistant ink, use of appropriate containers, and adherence to holding times, sample volume requirements, sample preservation, and what to do with compromised samples.

Procedure

SOP "GEN-003, Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers**"** contains the various checks that are performed on samples at the time of receipt and the required actions and documentation in the event that the criteria of the SOP are not met.

Procedure

All information provided with samples is maintained by administrative staff in the original project folders. A copy of the information is attached to the LIMS work order provided to the analyst.

24.3 Sample Identification

Policy

Samples including subsamples, extracts, and digestates, are uniquely identified in a permanent chronological record (ECCS LIMS) to prevent mix-up and to document receipt of all sample containers.

Procedure

SOP "GEN-003, Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers" addresses the sample receipt and log-in procedure.

24.4 Sample Storage

Procedure

Storage conditions (i.e., temperature) are monitored as required by SOP "GEN-005, Daily Equipment Checks."

- Samples are held secure and stored apart from contaminating sources such that cross-contamination between samples is minimized. All portions of samples, including extracts, digestates, leachates, etc. are maintained in accordance with test method required conditions.
- For on-site laboratory projects, where only one refrigerator may be available, standards should be isolated from samples through the use of a secondary container.

24.5 Sample Management

Policy

Samples are managed for disposal in accordance with federal, state and local regulations. Procedures are available for the management of samples, digestates, leachates, and extracts.

Procedure

SOP "GEN-003, Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers" provides detailed information regarding sample management.

24.6 Sample Transport

Policy

Samples that are transported under the responsibility of the laboratory, where necessary, are done so safely and in accordance with storage conditions. This includes moving bottles within the laboratory. Specific safety requirements are addressed in the ECCS Chemical Hygiene Plan and in test method SOPs.

24.7 Sampling Records

Policy

ECCS does not collect samples except for our SDWA compliance monitoring business. These samples are collected in accordance with SDWA guidelines. The samples are then subcontracted to SDWA certified labs for analysis.

24.8 Subsampling

Policy

Sub-sampling is performed in such a manner to obtain a representative sample. See SOP "GEN-023, Sub-sampling Soil Samples" for sub-sampling procedures.

Policy

When an unusual sample matrix is noted or the presence of an inordinate amount of extraneous material exists in a sample, the condition is noted in LIMS.

Procedure

SOP "GEN-022, Compositing Soil Samples" provides the procedure for compositing soil samples, when required by our clients.

25.0 QUALITY OF TEST RESULTS

This section provides for the policies and procedures needed to assure applicable quality control principals are implemented. Method-specific quality control procedures are found in the individual test method SOPs.

25.1 Essential Quality Control Procedures

Policy

All essential quality control elements are collected and assessed on a continuing basis.

Policy

Quality control test results are collected in LIMS such that trends are detectable, and where practicable, are statistically evaluated.

Policy

For test methods that do not provide acceptance criteria for an essential quality control element or where no regulatory criteria exist, acceptance criteria are developed based on practical experience.

Policy

The quality control procedures specified in test methods are followed by laboratory personnel. The most stringent of control procedures is used in cases where multiple controls are offered. If it is not clear which is the most stringent, that mandated by test method or regulation is followed.

Procedure

To monitor the validity of environmental tests performed, review includes any one or combination of the techniques below:

- use of certified reference materials and/or internal quality control using secondary reference materials;
- participation in proficiency testing programs;
- replicate testing using the same or different test methods;
- retesting of retained samples.

Written procedures to monitor quality controls including acceptance criteria are located in the test method SOPs, except where noted, and include:

- use of laboratory control samples (LCS) and blanks to serve as positive and negative controls;
- use of LCSs to monitor test acceptability of laboratory results;

- use of matrix spike samples and/or matrix spike duplicate samples to monitor precision and accuracy associated with a specific sample matrix;
- use of duplicate samples to monitor precision associated with a specific sample matrix;
- use of ICALs, CCVs, certified reference materials, and PT samples to monitor accuracy of the test method;
- measures to monitor test method capability, such as limit of detection, limit of quantitation, and/or range of test applicability, such as linearity;
- use of regression analysis, internal/external standards, or statistical analysis to reduce raw data to final results;
- use of reagents and standards of appropriate quality per test method SOP; and,
- procedures to ensure the selectivity of the test method.

25.2 Internal Quality Control Practices

Analytical data generated with quality control samples that fall within prescribed acceptance limits indicate the test method is "in control." Quality control samples that fall outside acceptance limits indicate the test method is "out of control" (non-conforming) and that corrective action is required or that the data are qualified.

Policy

Detailed quality control procedures are included in test method SOPs or as general guidelines set forth in this section.

Policy

All quality control measures are assessed and evaluated on an on-going basis so that trends are detected.

Acceptance or rejection criteria are created in accordance with laboratory policy where no test method or regulatory criteria exist. Acceptance criteria define the boundary for the appropriate response from laboratory personnel, such as corrective action, reporting with qualifiers, reanalysis, review, and others.

Procedure

The following general quality control measures are used:

Positive and negative controls such as:

- Blanks (negative)
- LCS (positive)

Selectivity is assured through:

- choice of detectors;
- absolute and relative retention times in chromatographic analyses;
- second-column confirmation when using non-selective detectors; and
- use of acceptance criteria for mass-spectral tuning (found in test method SOPs);
- use of the correct test method in accordance with its scope assessed during test method validation.

Consistency, variability, repeatability, and accuracy are assured through:

- proper installation and operation of instruments in accordance with manufacturer's recommendations or in accordance with the processes used during test method validation;
- monitoring and controlling environmental conditions (temperature, access, proximity to potential contaminants);
- selection and use of reagents and standards of appropriate quality;
- cleaning glassware appropriate to the level required by the analysis. Cleaning procedures are specified in SOP "GEN-006, Glassware Cleaning."
- following SOPs and documenting any deviation, assessing for impact, and treating data appropriately;
- testing to define the variability and/or repeatability of the laboratory results, such as replicates or MS/MSD samples; and
- use of measures to assure the accuracy of the test method, including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

Test method capability is assured through:

- establishment of the limit of detection;
- establishment of the limit of quantitation;
- establishment of the range of applicability such as linearity or acceptable regression analysis;
- analysis of a set of acceptable LODs; and
- analysis of a set of DOC samples.

Data Reduction is assured to be accurate by:

- selection of appropriate formulae to reduce raw data to final results, such as regression;
- periodic review of data reduction processes to assure applicability; and
- data reduction, and statistical interpretations specified by each test method.

25.3 Quality Control Samples

Table 25.3 summarizes the key elements of the ECCS quality control system. Corrective actions are specified in the test method SOP and/or in accordance with SOP "GEN-015, Data Qualification."

Table 25.3 Key Elements of Quality Control Program

Item	Frequency	Acceptance Criteria
Method Blank (Negative Control)	1/ batch of 20 or less or 1/day, whichever is greater.	Applicable regulatory requirement
Laboratory Control Sample (Positive Control)	1/ batch of 20 or less or 1/day, whichever is greater.	Laboratory control limits
Matrix Spike and/or Matrix Spike Duplicate Samples	1/ batch of 20 or less or 1/day, whichever is greater.	Laboratory control limits
Duplicate Samples	1/ 10 or less or 1/day, whichever is greater.	Laboratory control limits
Surrogates	Per test method requirement	Laboratory control limits
Continuing Calibration Verification	Per test method requirement	Test method specific
Initial Calibration Verification	Following each initial calibration.	Test method specific

Procedure

All QC samples are processed in the same manner as field samples. QC samples are analyzed and reported with the associated field samples. Method blanks are used to assess the preparation batch for possible contamination during the preparation and processing steps.

25.4 Method Blanks

A method blank is a sample of a matrix similar to the batch of associated samples 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 procedure, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Policy

Method blanks are prepared from analyte free water or sand in accordance with test method SOPs. Method blank test results are reviewed in accordance with applicable regulatory requirements.

Policy

Samples associated with a method blank that exceeds acceptance limits are evaluated as to the appropriate corrective action for the samples (e.g. reprocessing or data qualification).

Procedure

The procedures for review of method blanks are included in SOPs "GEN-016, Data Review Procedures" and "GEN-015, Qualification of Data."

Policy

When a blank is determined to exceed acceptance limits, the cause must be investigated and measures taken to minimize or eliminate the problem.

Policy

Data that are unaffected by the blank test results (non-detects or other analytes) are reported unqualified.

Policy

When sample data are suspect due to the presence of a blank exceedance, samples are reanalyzed, and/or qualified in accordance with SOP "GEN-015, Qualification of Data."

25.5 Laboratory Control Samples

Laboratory control samples are used to evaluate performance of the total analytical system, including all preparation and analysis steps. The ECCS LIMS terminology for laboratory control samples is blank spikes.

Policy

Laboratory control samples are prepared from analyte free water or sand, and spiked with verified and known amounts of analytes for the purpose of establishing precision or bias measurements. LCSs are analyzed at a frequency specified in test method SOPs.

Procedure

Matrix specific LCS control limits for each test method are calculated based on the mean and standard deviation of recoveries from a significant pool (minimum of 20 points) of data collected over time. Acceptance limits are based on the 99% confidence limit (± 3 standard deviations) generated from the historical data. Acceptance limits for marginal exceedances are ± 4 standard deviations. Where there are not established test method criteria, or there are not 20 points available to calculate limits, interim limits are used.

Procedure

Corrective actions for out of control recoveries are given in the test method SOP and SOP "GEN-015, Data Qualification."

25.6 Matrix Spike/Matrix Spike Duplicate Samples

Matrix spikes and/or matrix spike duplicates are samples fortified with a known amount of analyte to help assess the effect of the matrix on test method performance.

Policy

Matrix spike results are used to help assess the effect of the sample matrix on test method performance.

Procedure

Test method SOPs provide the procedure for matrix spikes includes spiking appropriate analytes at appropriate concentrations, calculating percent recoveries and relative percent difference (RPD) for MS/MSDs, and evaluating and reporting the results. An RPD is calculated using the following formula:

$$\text{RPD} = \frac{|\text{R1} - \text{R2}|}{\left(\frac{\text{R1} + \text{R2}}{2}\right)} \times 100$$

Where: R1 = Result from the initial analysis
R2 = Result from the reanalysis

Procedure

Matrix specific matrix spike control limits for each test method are calculated based on the mean and standard deviation of recoveries from a significant pool (minimum of 20 points) of data collected over time. Acceptance limits are based on the 99% confidence limit (± 3 standard deviations) generated from the historical data. Where there are not established test method criteria, or there are not 20 points available to calculate limits, interim limits are used.

Procedure

Corrective actions for out of control matrix spike recoveries are found in test method SOPs and "GEN-015, Qualification of Data."

25.7 Duplicate Samples

Duplicate samples are replicate aliquots of the same sample that provide a measure of precision when the analyte is present.

Procedure

Precision for duplicate samples is expressed as relative percent difference (RPD). RPD is calculated using the following formula:

$$\text{RPD} = \frac{|\text{R1} - \text{R2}|}{\left(\frac{\text{R1} + \text{R2}}{2}\right)} \times 100$$

Where: R1 = Result from the initial analysis
R2 = Result from the reanalysis

25.8 Surrogates

Surrogates are substances with chemical properties and behaviors similar to the analytes of interest used to assess test method performance in individual samples.

Policy

Surrogates are added to all samples (in test methods where surrogate use is appropriate) prior to sample extraction.

Procedure

Surrogate recovery results are compared to the acceptance criteria calculated by LIMS on at least a yearly basis.

Procedure

Matrix specific surrogate control limits for each test method are calculated based on the mean and standard deviation of recoveries from a significant pool (minimum of 20 points) of data collected over time. Acceptance limits are based on the 99% confidence limit (±3 standard deviations) generated from the historical data. Where test method criteria are not established, or there are not 20 points available to calculate limits, interim limits listed in test method SOPs are used.

Procedure

Corrective actions for out of control surrogate recoveries are found in test method SOPs and "GEN-015, Qualification of Data."

25.9 Proficiency Test Samples or Inter-Laboratory Comparisons

Policy

The laboratory participates in PT studies a minimum of twice per year per matrix. The results of these PT studies are used for maintenance of certifications/accreditations held by ECCS. For accrediting bodies requiring two PTs, the analysis dates shall be at least five months apart and no longer than seven months apart unless the PT sample is being used for corrective action for which the analysis dates shall be at least fifteen days apart. PT samples analyzed for the purpose of initially accrediting additional analytes are analyzed at least fifteen days apart.

Policy

The laboratory may utilize internal PT samples at any time for quality monitoring and quality improvement as determined by the QM.

Policy

PT results are submitted directly from the PT provider to certification/accreditation bodies. In this manner, certification/accreditation bodies are notified of all PT results including any failure(s).

Procedure

Proficiency test samples may be analyzed in either the fixed-based laboratory or in an on-site laboratory. The laboratory performing the analysis is determined by the OM.

Policy

The laboratory implements corrective action procedures for failed PT samples.

Procedure

The QM implements a CAF for each PT failure or finding from internal/external audits or management reviews. The root cause is evaluated by the discipline team leader and corrective actions are implemented. If a problem is identified, the root cause of the problem and any corrective action taken is documented. Typical corrective actions include analyst training, improvements to SOPs, or changes to the Quality Manual. The documentation is reviewed by the OM and submitted to the QM for review and approval. This documentation is maintained on file with the PT sample results. This documentation must also be submitted to A2LA for failure of any analyte associated with DoD accreditation.

Policy

The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

Procedure

PT samples are treated as typical samples in the normal laboratory process where possible, including the same preparation, calibration, quality control and acceptance criteria, sequence of analytical steps, number of replicates, and sample Log-In. In the event that the laboratory is certified/accredited for the same analyte(s) by more than one test method, the PT sample may be split and analyzed by each test method, (*i.e.*, 8270 Pesticides by GC/MS and 8141 Pesticides by GC/NPD).

25.10 Data Review

Policy

All data generated by the laboratory is reviewed for compliance with the test method, laboratory and, where appropriate, client requirements.

Procedure

Data review procedures and documentation of the review are specified in SOP "GEN-016, Data Review Procedures."

26.0 REPORTING OF RESULTS

Policy

The result of each test carried out is reported accurately, clearly, unambiguously, and objectively, and complies with all specific instructions contained in the test method.

Policy

Procedures for the required steps and responsibilities for report generation, report review, and final authorization of reports are specified in SOP "GEN-017, Report Preparation and Authorization."

Policy

For projects requiring NELAP accreditation, ECCS will report the data from each NELAP accredited test in accordance with the requirements of the NELAP standard (See Table 26-1, Detailed Report Format). In addition, the test reports will comply with any specific reporting requirements of a state or regulatory jurisdiction, *i.e.*, Louisiana requires a Level III Expanded Report (See Table 26-1).

Policy

Reports may be customized based on a specific request made by the client or based on regulatory requirements.

Procedure

Non-accredited testing either performed in-house or by a subcontractor shall be clearly identified in the email that delivers the final report.

Policy

Data are reported without qualification if they are greater than the lowest calibration standard, lower than the highest calibration standard, and without compromised sample or test method integrity.

Policy

All results associated with the use of the NELAP accreditation must meet both the QC criteria of the test method/SOP and the QA criteria of Section 25 of this QSM prior to release as a "Final Report." As required by SOP "GEN-017 Report Preparation and Authorization," data released without the second party review must be annotated as a "Draft Report." With this proper documentation on the report, the data can be released as "NELAP accredited" prior to the second party review.

Policy

On rare occasions on-site testing data may be released as a "final report" before secondary review based on a client specific request and approval. The primary example of this situation is when a client has hired a single chemist laboratory for a remediation project that will require "final reports" to be issued "real-time" to be used for disposal of contaminated soil at a landfill. In these cases, the data must still undergo a secondary review when the mobile laboratory returns to home base.

Policy

Technical justification is required any time that a test result is not used, and the justification must be documented. If a sample is reanalyzed, *i.e.*, to confirm prior results, and the results of both the initial and reanalysis are considered to be valid, the actual final value reported shall be determined by a documented procedure.

Procedure

If the RPD between the results of the initial analysis and the reanalysis is ≤ 20, report the result from the original analysis with no qualification. If the RPD between the results of the initial analysis and the reanalysis is > 20, report the original result qualified as estimated. The origin of the reported value must be clearly documented in the raw data and in the case narrative. RPD shall be calculated as follows:

$$\text{RPD} = \frac{|\text{R1} - \text{R2}|}{\left(\frac{\text{R1} + \text{R2}}{2}\right)} \times 100$$

Where: R1 = Result from the initial analysis
R2 = Result from the reanalysis

26.1 Test Reports

Policy

The report format has been designed to accommodate each type of test performed and to minimize the potential for misunderstanding or misuse of the report.

Procedure

- Each report shall have a title
- The laboratory's name, address, phone number, and name of a contact person for questions
- The LIMS work order number or the project number if LIMS was not employed, a pagination system that ensures that each page is recognized as part of the test report, and a clear identification of the end of the report, such as "Page 10 of 10"
- The name and address of the client and project name, where applicable

- Identification of the test method used
- Chain-of-custody indicating the client's sample identification, the date of receipt, and information about the condition of the samples upon receipt
- Reference to project specific plans such as a known QAPP, where applicable
- Clear identification of the reporting limits and their basis after adjustment for sample volume, dilution, percent solids, etc.
- The reporting basis, "wet weight" or "dry weight" shall be clearly identified on each report
- Date and time of analysis for each test method
- The name, function, and signature or equivalent electronic identification of the person authorizing the report
- A statement indicating the test results relate only to the samples
- A certification statement indicating that the test results meet the requirements of NELAP and/or other listed accreditation programs
- If applicable, a case narrative, which is specific to the work order that discusses analytical anomalies, QC sample results that fall outside of the control limits in the ECCS test method, and deviations from either the test method or procedures in this QSM.

Report Styles

ECCS offers four levels of reports to meet the specific reporting needs of most projects. Table 26-1 contains the specific contents of each report format.

Test Results Obtained from Subcontractors

Test results obtained from subcontractors shall be clearly identified with the subcontractor name and applicable accreditation number.

Policy

When necessary, test results shall be qualified in accordance with the SOP "GEN-015 Data Qualification," unless other instructions are received via a site-specific quality assurance project plan (QAPP), the prevailing regulatory jurisdiction, or the client.

Policy

The final report contents shall be reviewed by the project manager or his designee prior to release.

Policy

For NELAP accredited work, each case narrative will contain a statement to the effect that the results relate only to the samples and a certification that the results are in compliance with the NELAP Standards or, provide reasons and/or justification if they do not comply.

Table 26.1 ECCS Report Deliverable Formats

Summary Report
Analytical sample results (Summary LIMS Report Format) Chain of custody documents Surrogate recoveries- where applicable
Detailed Report (Standard Format; Used for NELAP Accredited Test Results[1])
Analytical results (Detailed LIMS Report Format) Case narrative Chain of custody documents Surrogate recoveries- where applicable Method blank results[2] Blank spike results[2] Blank spike duplicate results- when applicable[2] Matrix spike/matrix spike duplicate summary[2] Sample duplicate results, where appropriate[2]
Expanded Detailed Report
Detailed report plus: GC/MS tuning- where applicable Internal standard area summary- where applicable Initial calibration summary Continuing calibration summary Sequence summary Other test method related QC results, e.g. serial dilution results for metals.
Full Reportables
Expanded detailed report plus: All raw data including chromatograms, quantitation reports and spectra (where applicable) for each sample, QC sample, and standard. Miscellaneous data including instrument sequences, extraction/digestion logs, etc.

Notes:

1. Specific state or federal regulatory jurisdictions may require additional information, i.e., Louisiana requires the Expanded Report.

2. Quality control samples are reported on a per batch basis.

When necessary for interpretation of the results or when requested by the client, test reports may include the following additional information:

- deviations from, additions to, or exclusions from the test method, information on specific test conditions, such as environmental conditions, and any non-standard conditions that may have affected the quality of the results, and any information on the use and definitions of data qualifiers
- a statement of compliance/non-compliance when requirements of the QS are not met, including identification of test results that did not meet regulatory sample acceptance requirements, such as holding time, preservation, etc.
- when opinions and interpretations are included, the basis for the opinions and interpretations are documented. Opinions and interpretations, if applicable, are included in the case narrative and are clearly identified as such. Opinions and interpretations may include: (1) an opinion on the statement of compliance/ noncompliance of the results with requirements; (2) fulfillment of contractual requirements; (3) recommendations on how to use the results; and (4) guidance to be used for improvements.
- identification of non-target analytes present in the sample, if any is noted as present by the analyst
- additional information which may be required by specific test methods or client

Test reports obtained from subcontractors are provided as hardcopy or electronically as a PDF file. A copy of the subcontractor's report is delivered to the client.

26.2 Electronic Documents, Electronic Signatures, and Electronic Transmission of Test Results

Policy

ECCS's policy on use of electronic documents, electronic signatures, and electronic transmission of test results includes the following:

- Where laws, regulations, or rules require a signature, that requirement is met if the document contains an *electronic signature*.
- Where laws, regulations, or rules require a written document, that requirement is met if the document is an *electronic record.*
- Each party to a transaction must agree to conduct the transaction electronically in order for *electronic transmission* to be valid. Consent may be implied from the circumstances, except with respect to the *electronic records* used to deliver information for which consumers are otherwise entitled by law to receive in writing.
- If a law, regulation, or rule prohibits a transaction from occurring electronically, the transaction must occur in the manner specified by law.

- If a law, regulation, or rule requires an *electronic signature* to contain specific elements, the *electronic signature* must contain the elements specified by law.
- If a law, regulation, or rule requires that a record be retained or retained in its original form, that requirement is satisfied by retaining an electronic record of the information in the record that accurately reflects the information set forth in the original record and remains accessible for later reference.
- All test results transmitted by telephone, fax, e-mail, or other electronic means must comply with the requirements of this QSM and associated procedures to protect the confidentiality and proprietary rights of the client. Fax, email or other electronic forms of transmittals must include the following notice:

  NOTICE-- This email may contain confidential and privileged information for the sole use of the intended recipient. Any review or distribution by others is strictly prohibited. If you are not the intended recipient, please contact the sender immediately and delete all copies.

26.3 Amendments to Test Reports

Policy

Amendments or changes to a test report after it has been issued are made only in the form of a "Revised Report." Revised reports must meet all the requirements for the initial report and the requirements of this QSM.

Procedure

Test reports can be revised at the client's request, such as to add a previously unrequested analyte, or because of an error on the part of the laboratory. In both cases the original report is referenced.

Procedure

Revised reports require the approval of the OM, the QM, or their designee. The QM maintains a list of revised reports in the "Primary QA Tracking" Microsoft Excel file, including the reason for the reissue and any corrective measures taken to avoid similar occurrences in the future, if applicable.

Appendix A

Summary of ECCS

Validated Test Methods

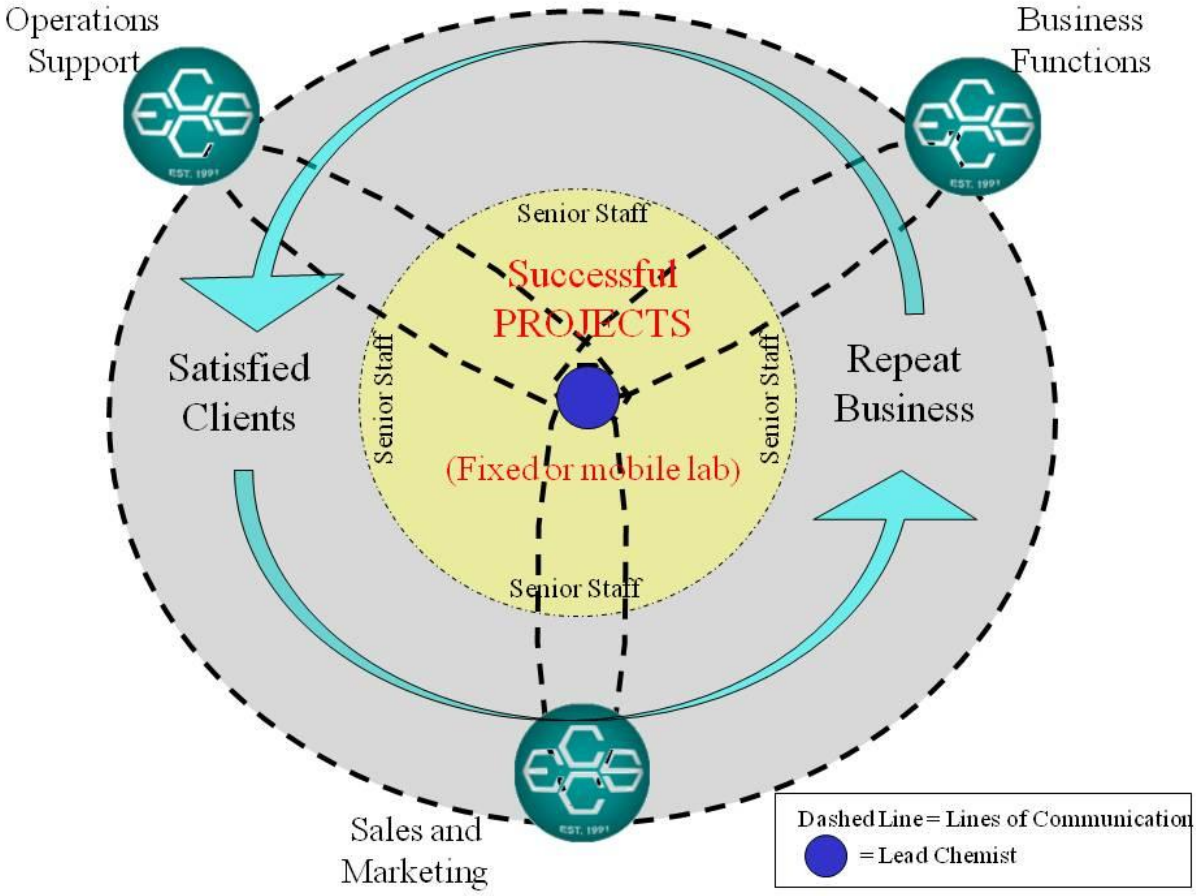
Summary of ECCS Validated Test Methods

Description	EPA Method Reference	ECCS Reference	Certification/Accreditation							
			WI	LA	IL	KS	NJ	TX	NC	DoD
VOCs										
VOCs [GC/MS, Purge & Trap]	EPA 8260B	LAM-004	X	X	X	X	X	X	X	X
VOCs [GC/MS, Dir. Inject]	EPA 8260B	LAM-010	X	X	X	X	X	X	X	X
VOCs [GC/MS, Air]	EPA 8260B	LAM-017								
Gasoline Range Organics [GC/MS]	8260B	LAM-004 Addendum 1								X
Diesel Range Organics [FID]	8015B	LAM-023		X	X	X	X	X	X	X
1,4-Dioxane [GC/MS SIM]	8260B - ECCS SOP	LAM-021	X	X	X		X	X	X	X
SVOCs										
Alkylated PAHs [GC/MS, SIM]	EPA 8270D	LAM-008	X	X	X	X	X	X	X	X
PAHs [GC/MS, Full Scan]	EPA 8270D	LAM-013	X	X	X	X	X	X	X	X
PAHs [GC/MS, SIM]	EPA 8270D	LAM-008	X	X	X	X	X	X	X	X
Organochlorine Pesticides [GC/ECD]	EPA 8081A	LAM-003	X	X	X	X	X	X	X	X
Neutral Extractable Pesticides [GC/MS]	EPA 8270D	LAM-006	X	X	X	X	X	X	X	X
Organophosphate Pesticides [GC/NPD]	EPA 8141A	LAM-022	X	X	X	X	X	X	X	
Acid Extractable Herbicides [HPLC]	EPA 8321	LAM-001	X	X	X		X	X		X
Acid Extractable Herbicides [GCMS]	EPA 8270D	LAM-014	X	X						X
Acid Extractable Herbicides [GCMS]	EPA 8151	LAM-014		X	X	X	X	X	X	
Pyrethroids [GC/MS]	EPA 8270D	LAM-024		X						
N-methylcarbamates [HPLC]	EPA 8318	LAM-020		X	X		X	X	X	
Urea Carbamates [HPLC]	EPA 8321	LAM-019	X	X	X			X		X
PCBs [GC/ECD]	EPA 8082	LAM-005	X	X	X	X	X	X	X	X
Explosives [GC/NPD, GC/ECD]	EPA 8095	LAM-018		X	X		P			X
Explosives [GC/MS]	EPA 8270D	LAM-032	X	X						
Explosives [HPLC/DAD]	EPA 8330A	LAM-031	X	X						
Inorganics										
Metals [XRF]	EPA 6200	LAM-011		X			X			X
Metals [ICP/MS]	EPA 6020B,	LAM-007		X		X	X	X		
Arsenic [GFAA]	EPA 7061A	LAM-033	X	X						
Nitrate, Colorimetric [FIA]	EPA 353.2	LAM-002	X	X	X	X		X	X	
Nitrate+Nitrite, [ISE]	SM4500-NO3-G (17th Ed.)	LAM-015		X						X
Nitrate+Nitrite, Colorimetric [FIA]	EPA 353.2	LAM-002	X	X	X	X		X	X	
Nitrite, Colorimetric [FIA]	EPA 353.2	LAM-002	X	X	X	X		X	X	
Ammonia, Colorimetric [FIA]	EPA 350.1	LAM-002	X	X	X	X		X	X	
Ammonia [ISE]	SM4500-NH3-D (20th Ed.)	LAM-015	X	X				X		X
Misc.										
Solids, Dry Weight	Solids, Dry Weight	GEN-007	X	X						
Total Suspended Solids	EPA 160.1	LAM-012	X	X	X	X		X		X
TCLP Extraction Procedure	EPA 1311	PRE-008		X				X		X
SPLP Extraction Procedure	EPA 1312	PRE-009		X				X		X
pH Measurements	EPA 9040C/9045D	LAM-035	X	X						
Paint Filter Test	9095D	LAM-034	X	X						

Appendix B

Lead Chemist Relationships to Operations

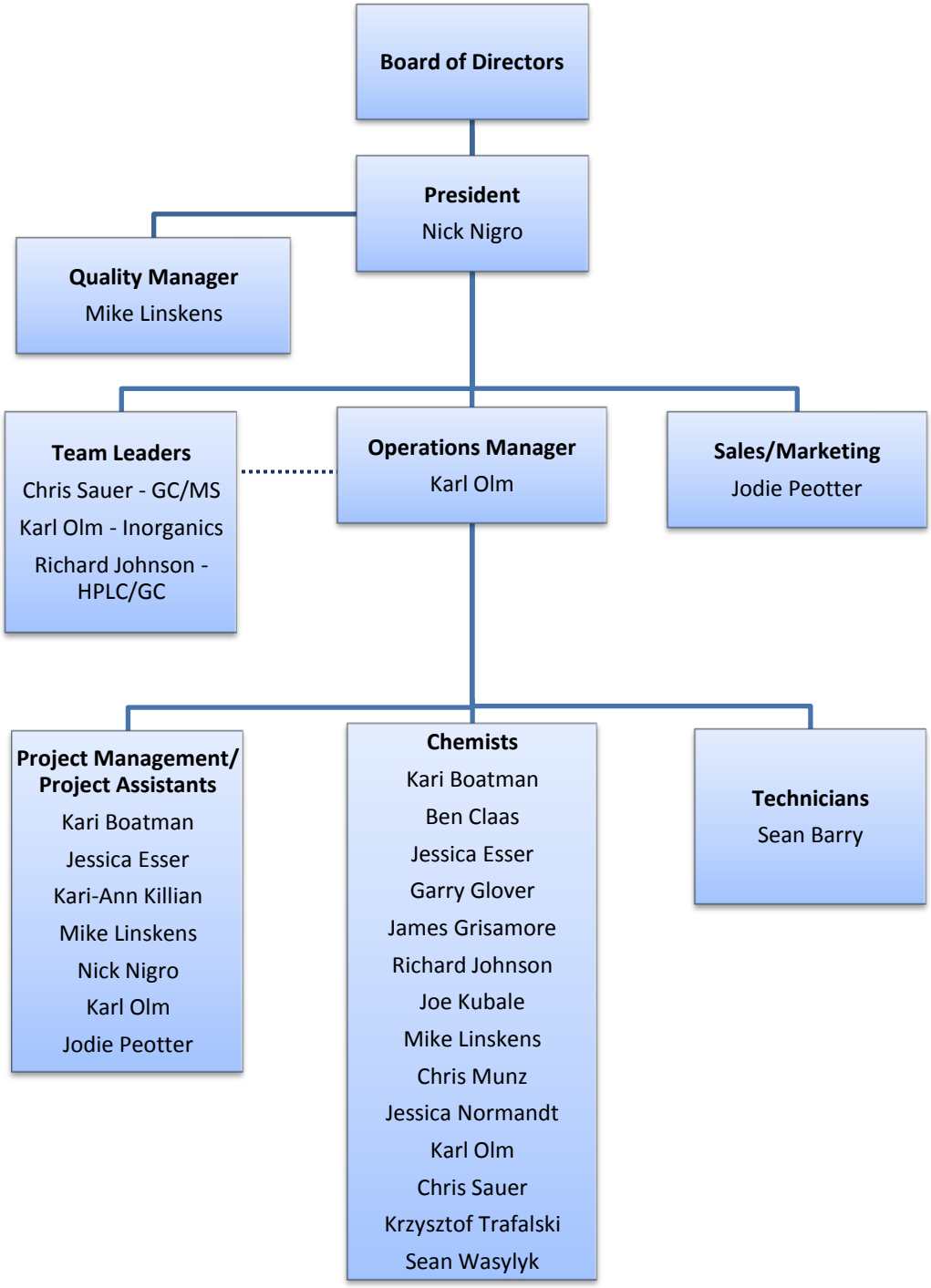
Lead Chemist Relationships to Operations



Appendix C

Organizational Chart

Organizational Chart



Appendix D

Corrective Action Form

CORRECTIVE ACTION FORM (CAF)

INITIALS:_____ DATE: _____

PROBLEM/DEFICIENCY: Test Method: _____

LIMS Work Order/ Sequence/Batch #	CCV(s)	Blank(s)	LCS(s)	MS/MSD	Surrogate(s)	Other
_____	____	____	____	____	____	____
_____	____	____	____	____	____	____
_____	____	____	____	____	____	____

Define Exceedance:_____

Root Cause (Why or how did this happen?):_____

Corrective Action:_____

Reviewer Approval:_____ Date_____

PLEASE KEEP BOTH COPIES ATTACHED. They will be separated during the review.
White copy of form is to be filed with Batch raw data. Yellow copy of form goes to QA Officer.

Appendix E

Chain-of-Custody Form

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

CHAIN OF CUSTODY

Page ____ of ____

Lab Work Order #:

Mail Report To:
Company:
Address:
E-mail Address:
Invoice To:
Company:
Address:

Project Number:
Project Name:
Project Location:
Turn Around (circle one): Normal Rush
If Rush, Report Due Date:
Sampled By (Print):

Analyses Requested
Preservation Codes

Sample Description	Collection Date	Collection Time	Matrix	Total # of Containers							Comments	Lab ID	Lab Receipt Time

Preservation Codes	Relinquished By:	Date:	Time:	Received By:	Date:	Time:
A=None B=HCL C=H_2SO_4 D=HNO_3 E=EnCore F=Methanol G=NaOH O=Other (Indicate)	Relinquished By:	Date:	Time:	Received By:	Date:	Time:

Matrix Codes	Custody Seal: Present/Absent Intact/Not Intact Seal #'s	Receipt Temp:
A=Air S=Soil W=Water O=Other	Shipped Via:	Temp Blank Y N

Download this form at www.eccsmobilelab.com.

WHITE - REPORT COPY YELLOW - LABORATORY COPY PINK - SAMPLER/SUBMITTER

Rev. 11/08

Appendix F

Job and Role Descriptions

Position Description

Title: President

Job Description Summary:
Provide leadership to position the company at the forefront of the industry. Develop a strategic plan to advance the company's mission and objectives and to promote revenue, profitability and growth as an organization. Oversee company operations to insure production efficiency, quality, service, and cost-effective management of resources.

Principal Accountabilities: (essential job functions and responsibilities)
1. Develop a strategic plan to advance the company's mission and objectives and to promote revenue, profitability, and growth as an organization.
2. Oversee company operations to insure production efficiency, quality, service, and cost-effective management of resources.
3. Plan, develop, and implement strategies for generating resources and/or revenues for the company.
4. Lead and/or oversee all company sales and marketing efforts.
5. Identify acquisition and merger opportunities and direct implementation activities.
6. Approve company operational procedures, policies, and standards.
7. Review activity reports and financial statements to determine progress and status in attaining objectives and revise objectives and plans in accordance with current conditions.
8. Evaluate performance of operations team and senior staff for compliance with established policies and objectives of the company and contributions in attaining objectives.
9. Promote the company through written articles and personal appearances at conferences and on radio and TV.
10. Represent the company at legislative sessions, committee meetings, and at formal functions.
11. Promote the company to local, regional, national, and international constituencies.
12. Other duties as assigned.

Required Experience and Skills:
1. Experience in strategic planning and execution. Knowledge of contracting, negotiating, and change management. Skill in examining and re-engineering operations and procedures. Experience in formulating policy, and developing and implementing new strategies and procedures. Ability to develop financial plans and manage resources. Ability to analyze and interpret financial data. Knowledge of public relations principles and practices. Knowledge of communication and public relation techniques. Ability to develop and deliver presentations. Ability to identify and secure funding/revenue sources.
2. Work requires professional written and verbal communication and interpersonal skills. Ability to communicate and interact with officials at all levels of government and to work effectively with a wide range of constituencies in a diverse community. Ability to motivate teams and simultaneously manage several projects.
3. This is normally acquired through a combination of the completion of a Masters Degree in Business Administration, Finance or Accounting and ten years of experience in a leadership role for a large division or company.

Position Description

Title: Operations Manager

Reports To:
President

Job Description Summary:
Provide for the day-to-day operations management of our Madison based laboratory; oversight/direction of Madison based rotating field staff; and maintenance and preparation of equipment and fleet (vehicles/trailers) in support of both Madison and field operations.

Supervisory Responsibilities:
Direct reports include the full-time laboratory staff. Log-In and reporting technicians and rotating field chemists are indirect reports.

Principal Accountabilities: (essential job functions and responsibilities)
1. Supervises laboratory operations to maintain quality and efficiency.
   a. Provides day to day direction to full time laboratory staff and rotating field staff to assure client and regulatory standards are being met.
   b. Strives to meet or exceed client turnaround time and quality expectations.
   c. In concert with QA officer, maintains QS and appropriate laboratory certifications and approvals.
   d. Actively seeks improvement to standard operating procedures and laboratory protocols.
   e. Reviews work, documents and provides both positive and developmental feedback, performance appraisals, on-going advice, council and recommendations for staff development and training of direct reports.
2. Provides day-to-day direction of rotating field staff.
   a. Maintains successful laboratory operations by scheduling available rotating field staff for laboratory work.
   b. Maintains a list of tasks to be accomplished in Madison and schedules available rotating field staff to complete the tasks.
   c. Provides on-going advice, council and recommendations for staff development and schedules cross-training of rotating field staff when applicable.
3. Maintains and prepares equipment/vehicles/trailers in support of both Madison and field operations.
   a. Maintains an adequate supply of equipment and supplies to support Madison and field operations including:
      (1) computer equipment and supplies
      (2) laboratory equipment and supplies
      (3) field operations infrastructure equipment and supplies
   b. Maintains and prepares vehicle and trailer fleet
   c. Implements fleet maintenance schedules including remote field vehicles
   d. Prepares vehicles/trailers for field operations as needed

Required Experience and Skills:
1. Bachelor of Science Degree
2. 10+ years of laboratory experience with an emphasis in pesticide residue analyses.
3. 5+ years of analytical project management experience.
4. 5+ years of ECCS on-site laboratory experience.

5. Excels in client management and service.
6. Strong interpersonal and oral and written communication skills.
7. Effective business management knowledge.

Position Description

Title: Quality Manager

Reports To:
President

Job Description Summary:
This position is responsible for developing, directing and coordinating a comprehensive QS program for use in ECCS' on-site and fixed-base laboratory operations; maintaining appropriate agency certifications and approvals; and daily review of fixed-base laboratory final reports.

Supervisory Responsibilities:
No direct supervisory responsibilities. Indirect responsibility to work closely with staff in a mentoring role.

Principal Accountabilities: (essential job functions and responsibilities)

1. Develops, directs and coordinates the on-site and fixed-base laboratories' quality assurance programs.
   a. Uses available tools, such as audit and surveillance results, control charts, PT results, data analysis, corrective and preventative measures, customer feedback, and management reviews to monitor laboratory practices to promote a continuous quality improvement process.
   b. Ensures all personnel understand their contribution to the quality system.
   c. Ensures communication takes place at all levels of the business regarding the quality system.
   d. Coordinates review and approval of, or develops standard operating procedures and QA Manuals.
   e. Coordinates quarterly and yearly PE sample requirements.
   f. Performs quality audit of approximately 10 % of on-site and fixed-base laboratory data.
   g. Performs internal system audits for the fixed-base laboratory on a yearly basis and a minimum of four on-site testing audits per year.
   h. Maintains organized quality assurance records by test method and by analyst.
2. Establishes and maintains appropriate agency certifications and approvals.
   a. Maintains current knowledge of and is the authoritative source for various agency certification programs and rules.
   b. Maintains certifications and approvals with timely submittal of each program's yearly requirements such as: program fees; yearly PE results; changes to test methods or updates to SOPs, etc.
   c. Coordinates any agency audit or visit.
3. Monitors fixed-base data quality by reviewing and signing each individual client report. Monitoring process should include at a minimum: review of associated raw data quality control results; check of sample reports for client provided field quality control samples (blanks, field duplicates); comparison to previous data, where possible; check appropriateness of test results in conjunction with likely DQO (potential false positives in a water sample).
4. Assists sales/marketing staff and project assistant with client development and proposal preparation.
5. Contributes to and monitors staff development and training protocols, competency standards and documentation requirements.
6. Provides semi-annual reports to management summarizing audit findings, agency issues, etc.

Required Experience and Skills:

1. Bachelor of Science Degree
2. 10+ years of laboratory experience with an emphasis in organics analyses.
3. 3+ years of ECCS on-site laboratory experience.
4. Excels in client interactions and service.
5. Strong interpersonal and oral and written communication skills.

Position Description

Title:
Team Leader

Reports To:
President

Job Description Summary:
This role description serves a critical function at ECCS that can be best summarized in two statements: consistency of ECCS work product and technical/personal growth of team. The team will be comprised of all ECCS staff that routinely performs test methods within the team's technical area of responsibility. The role of the Team Leader will be to coordinate the efforts of the team in an encouraging, collaborative manner.

Supervisory Responsibilities:
The Team Leader will have a direct technical and training oversight role of the entire team; however, more effort will be placed with less experienced staff. The Team Leader will participate in all project closeout meetings involving test methods within the team's technical area of responsibility and will be expected to provide primarily technical and potentially financial feedback. Feedback provided to individuals will be documented in their personnel file.

Principal Accountabilities: (essential job functions and responsibilities)

1. SOPs
   a. Oversees the revision or development of all SOPs assigned to the Team. Requires Team Leader to take first-hand responsibility (and signature of said SOPs), while at the same time seeking input, collaboration, and empowerment of all individuals within the team to support these efforts.
   b. At least annually reviews SOPs for conformance with best management practices, audit findings, and lessons learned. Ensures that entire team is versed, and trained, in team SOPs.
2. Technical
   a. Continually seeks opportunities, in conjunction with team, opportunities to evaluate new (or simply different) technologies for faster, better, cheaper test method implementation. Evaluations performed must be documented in a concise fashion and presented to the Ops Team.
   b. Provides annual summation to Ops Team on suitability of SOPs for continued promulgation, or recommends changes/ enhancements that need to be made.
3. Equipment
   a. Responsible for communicating equipment and technical support needs to the Operations Manager.
   b. Remains current on equipment and test methods used in the analysis of samples within Team. Is capable of providing insight into equipment purchases and analytical test methods.
   c. Assures that routine preventative maintenance is performed on equipment associated with Team.
4. Staff
   a. Conducts frequent formal and informal training sessions with team staff. Training can be on the job or in-house. All training is to be documented and placed in the employee file.
   b. Assures that the required initial Demonstration of Capability (DOC) protocols are completed and documented for all analysts/technicians assigned to the Team.
   c. Assures LODs are completed for associated test methods
5. ECCS Work Product

a. "Responsible" for the final work product for every project that falls within this area of expertise, whether the Team Leader is part of the project team or not. This will require multi-tasking in instances where the Team Leader is Lead Chemist for another project.
b. Team Leader is not responsible for reviewing every ECCS work product associated with team. Rather, they are to ensure that (1) the analysts are trained and capable of performing the test method, (2) the peer reviewer is someone from the team, or designee, that is equally as trained/competent, and (3) Team Leader routinely audits team work product, especially as needed when issues or non-conformances are identified and must be explained in the case narrative.

6. General
   a. Conducts routine team meetings to discuss pending and on-going project work, analytical issues, training needs, test method development, and technical advancements that might be evaluated to continue to push the concept of faster, better, and cheaper.
   b. Works collaboratively with the Operations Manager on all issues associated with successful implementation of this role description.
   c. Participates in weekly Ops Team meeting. Comes to said meeting prepared to talk about key priorities or issues associated with team.

Position Description

Title: Senior Chemist

Reports To:
Operations Manager

Job Description Summary:
This position requires the ability to function in several roles; as an analytical chemist; as a project manager; as a lead chemist; as a technical expert; as a mentor; while providing solution-oriented and quality objective-oriented analytical services to our clients in our fixed and on-site laboratories. This position requires approximately 50% travel to on-site laboratory operations.

Supervisory Responsibilities:
No direct supervisory responsibilities. Indirect responsibility to work closely with junior staff in a mentoring role.

Principal Accountabilities: (essential job functions and responsibilities)
1. Performs routine and non-routine laboratory testing of samples in accordance with the ECCS QS.
2. Maintains current knowledge and principles of EPA test methods and associated federal, state, and local regulations.
3. Maintains/repairs laboratory instrumentation and associated equipment .
4. Develops and validates new fixed or on-site laboratory test methods and prepares SOPs.
5. Keeps equipment operating by following operating instructions; troubleshooting breakdowns; maintaining supplies; performing preventive maintenance.
6. Maintain daily logs, equipment record books, and computer database of sample results.
7. Complete projects by assisting project team; attending and participating in group and project meetings.
8. Interacts with clients in a positive manner.
9. Serves as a technical expert and mentor to junior staff.
10. Actively promotes quality improvement.
11. Maintains awareness of occupational safety hazards and is skilled in implementing appropriate safety practices and recognizing and communicating hazards.
12. Functions in the role of project manager (see Project Manager role description).
13. Functions in the role of lead chemist (see Lead Chemist role description).

Required Experience and Skills:
1. Bachelor of Science Degree
2. 10+ years of laboratory experience with an emphasis in instrumental analyses.
3. Excels in client interactions and service.
4. Strong interpersonal and oral and written communication skills.
5. Skilled in various instrumental computer applications

Position Description

Title: Chemist

Reports To:
Operations Manager

Job Description Summary:
This position requires the ability to function as an analytical chemist in our fixed and on-site laboratory operations while providing solution-oriented and quality objective-oriented analytical services to our clients. This position requires approximately 50% travel to on-site laboratory operations.

Supervisory Responsibilities:
No direct supervisory responsibilities.

Principal Accountabilities: (essential job functions and responsibilities)
1. Performs routine testing of samples in accordance with the ECCS QS.
2. Maintains current knowledge and principles of EPA test methods and associated federal, state, and local regulations.
3. Keeps equipment operating by following operating instructions; troubleshooting breakdowns; maintaining supplies; performing preventive maintenance.
4. Maintain daily logs, equipment record books, and computer database of sample results.
5. Complete projects by assisting project team; attending and participating in group and project meetings.
6. Interacts with clients in a positive manner.
7. Actively promotes quality improvement.
8. Maintains awareness of occupational safety hazards and is skilled in implementing appropriate safety practices and recognizing and communicating hazards.
9. Functions in the role of Lead Chemist, where appropriate (see Lead Chemist role description).

Required Experience and Skills:
1. Bachelor of Science Degree
2. 2+ years of laboratory experience with an emphasis in instrumental analyses.
3. Excels in client interactions and service.
4. Strong interpersonal and oral and written communication skills.
5. Ability to work with various instrumental computer applications

Position Description

Title: Technician

Reports To:
Operations Manager

Job Description Summary:
This position primarily provides support to the fixed-based laboratory in Madison, WI. Occasional support at an on-site laboratory operation may be required. This position requires approximately 10% travel to on-site laboratory operations.

Supervisory Responsibilities:
No direct supervisory responsibilities.

Principal Accountabilities: (essential job functions and responsibilities)
1. Maintain sample Log-In and tracking records.
2. Provide sample preparation support for pesticide analyses in accordance with standard operating procedures.
3. Washing laboratory glassware
4. Prepares and analyzes soil and water samples for VOCs and SVOCs in accordance with the ECCS QS.
5. Keeps equipment operating by following operating instructions; troubleshooting breakdowns; maintaining supplies; performing preventive maintenance.
6. Maintain daily logs, equipment record books, and computer database of sample results.
7. Complete projects by assisting project team; attending and participating in group and project meetings.

Required Experience and Skills:
1. Bachelor of Science Degree preferred
2. Detail oriented
3. Solid written and verbal communication capabilities
4. Interested in a career in environmental chemistry
5. Ability to work with a variety of computer applications.

Position Description

Title: Project Manager (PM)

Reports To:
President

Job Description Summary:
This position is responsible for support of sales/marketing staff in design and procurement of new projects, program management of all projects assigned. The PM will also often serve in the role of Lead Chemist and/or Project Assistant.

Supervisory Responsibilities:
The PM often has project-related team leader/supervisory responsibility for one or more lab staff and the Project Assistant.

Principal Accountabilities: (essential job functions and responsibilities)
1. Provides support to sales/marketing staff in design and procurement of new projects.
   a. Provides technical support in development of project scope of work.
   b. Offers proposal design insight by relating similar project and/or client experiences.
2. Accepts responsibility of program management of new projects.
   a. Responsible for primary project management level functions and communication with the client.
      (1) Develops working relationship with client, ensuring project success and maintaining opportunities for repeat business.
      (2) Documents all pertinent information in project file.
      (3) Tracks budget, prepares invoices and communicates with client in this regard.
   b. Understands technical and logistical needs of the project and works with the Operations Manager to select/schedule Lead Chemist(s) and project team.
   c. Understands the ECCS business model and strives to help contribute to company business success and profitability.
      (1) Schedules staff effectively to reduce airfare costs.
      (2) Makes sure project details necessary for invoicing are submitted on a timely basis.
   d. Interacts with client, Lead Chemist and other project personnel to monitor project issues and progress.
   e. Prepares and/or reviews final report to the client.
   f. Upon project completion, reviews project file, completes project assessment and QA closeout forms, and seeks feedback from client, positive or negative.
3. Provides training/mentorship of staff regarding all aspects of project development, execution and closure.

Required Experience and Skills:
1. Bachelor of Science Degree or equivalent experience.
2. 3+ years of instrumental laboratory experience.
3. Proven understanding of ECCS business model.
4. Superior client service skills.
5. Strong interpersonal and oral and written communication skills.

Position Description

Title: Lead Chemist

Reports To:
Operations Manager

Job Description Summary:
This position provides for the day-to-day operations management of one of ECCS' on-site laboratory projects. The Lead Field Chemist accepts project assignments from the operations manager; determines project requirements and client interaction role; prepares necessary equipment, supplies and vehicles (if required); conducts/supervises (when required) on-site testing operations; decommissioning the project's on-site laboratory unit; prepares final report; and provides feed back to the project manager.

Supervisory Responsibilities:
The lead chemist may have project team leader/supervisory responsibility for one or more field staff.

Principal Accountabilities: (essential job functions and responsibilities)
1. Determines project requirements and client interaction role in concert with project manager and operations manager.
2. Prepares necessary equipment, supplies and vehicles (if required).
   a. Schedule on-site laboratory and truck, as needed.
   b. Gather equipment and supplies required to meet the needs of the project. Checklists exist for some of the more common ECCS project types.
   c. Checks equipment to assure proper operation.
3. Conducts and supervises (when required) on-site testing operations.
   a. For single person short term projects, the lead chemist is required to be responsible for all aspects of the project: truck driver, analyst, sample Log-In and reporting technician, sample preparation technician, QA manager, metrology guru, maintenance department, and most importantly client services representative.
   b. Actively pursues relationship with client's field team and project manager.
   c. For multiple person short term projects, the lead chemist is also responsible for supervision of support staff.
   d. For multiple person longer term projects, the lead chemist role will fall on several individuals. In this case, the current on-site lead chemist has primary responsibilities for: on-site client interactions, coordination of on-site operational activities, being an analyst, training/mentorship of junior staff, maintaining the QS, and meeting the client's day-to-day needs.
4. Decommissioning the project's on-site laboratory unit.
   a. Properly dispose of all waste generated by the operation.
   b. Restock trailer based supplies.
   c. Clean the trailer countertops and floors.
   d. Report any equipment, trailer or truck issues to the field operations manager.
5. Prepares final report.
   a. For small projects, the lead chemist is responsible for preparing the final techmemo style report.
   b. For large projects, the lead chemist provides input to the project assistant for the final report.
6. Provides feedback to the project assistant.
   a. For any size project, the lead chemist is responsible for immediately contacting the project manager to provide feedback about any client concern.

b. For small projects, the lead chemist is responsible for scheduling a project exit interview with the project manager. The primary responsibility of the lead chemist during this interview is to pass on pertinent information about the execution of the project and the interactions with the client.
c. For large projects the lead chemist needs to communicate with the project manager on a regular basis to provide feedback about day-to-day operations, upcoming schedules, pertinent client interactions and any issue which is or could be a client concern.

Required Experience and Skills:

1. Bachelor of Science Degree or equivalent laboratory experience
2. 3+ years of instrumental laboratory experience.
3. Previous ECCS on-site lab experience.
4. Experience with or training for the need for superior client service.
5. Strong interpersonal and oral and written communication skills.

Position Description

Title: Information System Administrator

Reports To: President

Job Description Summary:
The Information System Administrator provides day-to-day support of ECCS' laboratory information management system (LIMS) and associated software and hardware used in both our fixed and mobile laboratory environments. This position also encompasses support of all aspects of our IT infrastructure, including the ECCS client/server network, peripherals, and e-mail system. It is understood that ECCS will maintain third-party systems administration support.

Principal Accountabilities: (essential job functions and responsibilities)

1. LIMS Administration
   a. Support IT/LIMS Manager in the training of ECCS staff on routine and customized functions of our LIMS.
   b. Manage primary database (and field module databases) including routine O&M (e.g., updating program files, modifying user permissions, etc.) and non-routine activities (troubleshoot issues).
   c. Manage the report functionality of the LIMS, including supporting the generation of "standard" reports as well as modifying report templates (using Crystal Reports) to allow for customized report generation per client requests.
   d. Develop and validate new analyses to be added to the LIMS functionality as new ECCS methods are brought on-line.
   e. Administer the import/export database module for all mobile laboratory projects
2. Field Project Startups
   a. Establish a functional LIMS database for all mobile laboratory projects.
      i. Develop and export the project-specific database to be used on the specific field project
      ii. Configure the mobile laboratory with adequate hardware/software
   b. Implement a fully functional database, which at times will require travel to the site during the initial project startup to configure the database and ensure that all instruments and network are communicating.
      i. Ensure that said database is configured to meet all project-specific requirements including: method requirements, reporting limits, and reporting formats (inter alia).
      ii. Act as data technician for shorter projects and/or train local data technicians for longer projects.
3. Project Reporting (Mobile Laboratory Projects)
   a. Support the final report generation of all mobile laboratory projects. Although this task will be shared with others in the Data Management group, this position will be the primary individual for most projects as time and schedule allows. NOTE: "Final report" encompasses tabulated results, routine and customized EDD's, and Level IV hard-copy data packages.

   b. Establish systems and protocols to ensure the safe and efficient management of all ECCS raw data and reports (electronic and hard-copy)
4. Backup Project Assistant
   a. Support day-to-day activities including standards and sample log-in when all other tasks have been completed and/or when needed based on schedule and availability of others within the "project assistant" group.
   b. Support completion of project close-out documentation for mobile laboratory projects.
5. Network/ IT Support
   a. It is generally understood that at least initially this position will primarily be focused on LIMS administration. However, as all ECCS staff become experienced and confident with our LIMS system, we believe this role will involve increased time with the ECCS network and general IT environment. As stated previously, we will maintain third-party systems administration support to perform or support many of these key items.
      i. Assist in the operation and maintenance of PC equipment, printers and related equipment and respond to user complaints and inquiries.
      ii. Troubleshoot, diagnose, and resolve PCs, servers and other computer-related equipment problems to ensure functional operation.
      iii. Perform back-up procedures for assigned PC files; coordinate restoration and retrieval of files as needed; maintain all files in an accurate and efficient manner.
      iv. Coordinate and install cards, drives, memory, peripheral equipment and software into stand-alone or network connected PCs at client work locations.
      v. Verify hardware and software purchase requests.
      vi. Perform preventive maintenance and system administration duties including setting up and monitoring server back-up processes, adding new users, modifying user/group profiles, and monitoring and tuning server and system resources.
      vii. Coordinate and conduct user training, education and problem solving sessions; counsel users regarding system functionality.
      viii. Maintain network components, including routers, hubs, smart switches and cables.

Required Experience and Skills:

1. Strong interpersonal and oral and written communication skills.
2. Familiarity with ECCS LIMS and network.
3. Patience and willingness to work with ECCS staff.
4. Experience using Microsoft XP and Windows 2000 Operating Systems, Microsoft Office 2007, Adobe Acrobat, and Element LIMS.
5. Desire to learn new applications as needed (e.g. Crystal Reports).
6. Demonstrate pride of workmanship and interest in taking on additional responsibilities to contribute to team success.
7. Attention to detail
8. Availability to travel as needed for field projects

Position Description

Title: Project Assistant

Reports To:
Project Manager

Job Description Summary:
This position provides for continuous support of the Project Manager for fixed and on-site laboratory projects.

Supervisory Responsibilities:
None

Principal Accountabilities: (typical job functions and responsibilities)
1. Determines project peripheral needs with the Project Manager.
2. Provides for non-technical logistical issues during project planning, initiation and execution.
    a. Assures initial file information is complete including project set-up, analytical documentation, and client feedback forms.
    b. Acquires site location maps with driving directions.
    c. Prepares Health & Safety Plan information with directions to the nearest emergency health care facility.
    d. Makes hotel and transportation reservations.
    e. Coordinates generator rentals.
    f. Completes other tasks as assigned by the Project Manager.
3. Coordinates report generation.
4. Works with Operations Manager and Project Manager to assure project file and invoice are complete.
5. Upon direction from client services, contacts the client to obtain feedback.

Desired Experience and Skills:
1. Associates Degree or equivalent laboratory experience.
2. 2+ years of multi-dimensional project coordination experience.
3. Strong interpersonal, oral and written communication skills.
4. Proficiency in Microsoft Office suite and willingness to learn LIMS system.

Position Description

Title: Purchasing Agent

Reports To:
President

Job Description Summary:
This position is responsible for ordering supplies from vendors. This requires obtaining quotations from suppliers for new items and ensuring that we have current quotations for routine supplies.

Supervisory Responsibilities:
The Purchasing Agent has no supervisory responsibilities.

Principal Accountabilities: (essential job functions and responsibilities)
1. Order standard supplies as needed to maintain proper inventory.
   a. A technician will often conduct an inventory of standard supplies on a bi-monthly basis using an Excel spreadsheet. The purchasing agent then reviews the inventory and orders supplies as needed.
2. Order supplies from purchase requisition forms.
   g. When chemists need standards or other non-routine supplies they submit their requests on a purchase requisition form. The Purchasing Agent then orders the supplies as needed. This could involve consulting new vendors and establishing an account.

3. Obtain yearly quotes from vendors.
   a. Many of our vendors provide pricing on a yearly basis. The Purchasing Agent is responsible for ensuring that these quotes are completed and checking with other suppliers to ensure that ECCS is receiving the lowest price.

Required Experience and Skills:
1. Proven understanding of ECCS business model.
2. Superior client service skills.
3. Strong interpersonal and oral and written communication skills.

V0LATILE ORGANIC COMPOUNDS BY PURGE AND TRAP/GAS CHROMATOGRAPHY/MASS SPECTROMETRY

BASED ON EPA METHOD 8260B

1 SCOPE AND APPLICATION

1.1 This method is a capillary column gas chromatography/mass spectrometry (GC/MS) method used to determine part per billion concentrations of volatile organic compounds (VOC). The volatiles are introduced into the GC/MS system by the purge and trap technique. This method is based on Method 5030B and 8260B, and is used to determine volatile organic compounds in a variety of matrices. This method is applicable to soil, water, and waste samples.

Compound	CAS No.	Compound	CAS No.
1,1,1,2-Tetrachloroethane	630-20-6	Carbon Tetrachloride	56-23-5
1,1,1-Trichloroethane (1,1,1-TCA)	71-55-6	Chlorobenzene	08-90-7
1,1,2,2-Tetrachloroethane	79-34-5	Chlorodibromomethane	124-48-1
1,1,2-Trichloroethane (1,1,2-TCA)	79-00-5	Chloroethane	75-00-3
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Chloroform	67-66-3
1,1-Dichloroethane (1,1-DCA)	75-34-3	Chloromethane	74-87-3
1,1-Dichloroethene (1,1-DCE)	75-35-4	cis-1,2-Dichloroethene (cis-1,2-DCE)	156-59-2
1,1-Dichloropropene	563-58-6	cis-1,3-Dichloropropene	10061-01-5
1,2,3-Trichlorobenzene	87-61-6	Dibromomethane	74-95-3
1,2,3-Trichloropropane	96-18-4	Dichlorodifluoromethane	75-71-8
1,2,4-Trichlorobenzene	120-82-1	Di-isopropylether	108-20-3
1,2,4-Trimethylbenzene	95-63-6	Ethylbenzene	100-41-4
1,2-Dibromo-3-chloro-propane	96-12-8	Hexachlorobutadiene	87-68-3
1,2-Dibromoethane	106-93-4	Isopropylbenzene	98-82-8
1,2-Dichlorobenzene	95-50-1	Methylene chloride	75-09-2
1,2-Dichloroethane (1,2-DCA)	107-06-2	Methyl-tert-butylether (MTBE)	1634-04-4
1,2-Dichloropropane	78-87-5	m-Xylene	108-38-3
1,3,5-Trimethylbenzene	108-67-8	Naphthalene	91-20-3
1,3-Dichlorobenzene	541-73-1	n-Butylbenzene	104-51-8
1,3-Dichloropropane	142-28-9	n-Hexane	110-54-3
1,4-Dichlorobenzene	106-46-7	n-Propylbenzene	103-65-1
2,2-Dichloropropane	594-20-7	o-Xylene	95-47-6
2-Butanone (MEK)	78-93-3	p-Isopropyltoluene	99-87-6
2-Chlorotoluene	95-49-8	p-Xylene	106-42-3
2-Hexanone	591-78-6	sec-Butylbenzene	135-98-8
2-Pentanone,4-methyl (MIBK)	108-10-1	Styrene	100-42-5
4-Chlorotoluene	106-43-4	tert-Butylbenzene	98-06-6
Acetone	67-64-1	Tetrachloroethene (PCE)	127-18-4

Benzene	71-43-2	Tetrahydrofuran	109-99-9
Bromobenzene	108-86-1	Toluene	08-88-3
Bromochloromethane	74-97-5	trans-1,2-Dichloroethene	156-60-5
Bromodichloromethane	75-27-4	trans-1,3-Dichloropropene	10061-02-6
Bromoform	75-25-2	Trichloroethene (TCE)	79-01-6
Bromomethane	74-83-9	Trichlorofluoromethane	75-69-4
Carbon Disulfide	75-15-0	Vinyl chloride	75-01-4

1.1 This method is used to identify and quantitate volatile organic compounds that have boiling points below 200 °C.

1.2 The GC/MSD provides for selective detection of the VOCs by extracting specific ions for quantitation and qualification from the total ion chromatogram. Compound identification is performed by comparing the unknown compounds retention time and spectra against reference mass spectrum and retention time acquired from authentic standards.

1.3 The Chemist should be a chromatography expert capable of operating and maintaining the purge and trap GC/MS system and experienced in the analysis of VOCs. Training includes generating a valid initial calibration and an acceptable initial demonstration of capability study in addition to understanding the use of mass spectra for identification and quantitation.

2 SUMMARY OF METHOD

2.1 Water samples are analyzed directly without any sample preparation by GC/MS purge and trap. Soil samples require extraction with methanol. An aliquot is then added to reagent water and analyzed by purge and trap GC/MS.

2.2 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. This method purges the VOCs from the water matrix into a stream of inert helium gas. The VOCs are trapped from the gas stream onto an organic polymer at room temperature. When the polymer is rapidly heated, the VOCs desorb back into the gas stream and enter the GC. The analytes are introduced directly to a narrow-bore capillary column from the purge and trap using a direct split-injector interface at the gas chromatograph (GC). The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) directly interfaced to the GC. A total ion chromatogram of a mid-range calibration standard is provided in Figure 2.

2.3 Identification of target analytes is accomplished by comparing their mass spectra with the mass spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using an average response factor regression type analysis.

3 DEFINITIONS AND ACRONYMS

3.1 There are many terms and acronyms used throughout this document. Check the definitions and acronyms sections of the Quality Manual for complete explanations.

4 INTERFERENCES

4.1 Major contaminant sources are volatile materials and solvents that enter the atmosphere or diffuse into the de-ionized water which will be concentrated on the trap during the purge and trap operation. Special care must be used with solvents to avoid methylene chloride and acetone contamination. The VOC laboratory is isolated from the remainder of the laboratory across the parking lot in the annex. No solvents other than methanol must enter this laboratory. Methanol is stored apart from other solvents in the VOA annex.

4.2 Chloromethane can form as a by-product with hydrochloric acid preservation of water samples. Chloromethane can also be generated on a used trap contaminated with salts in the purge and trap system. Corrective action should be taken whenever the chloromethane level exceeds 2.0 µg/L. Corrective action could include, but not be limited to, replacing the trap or using an alternative chemical preservative for waters.

4.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free water or methanol and carried through the sampling, handling, and storage protocols serves as a check on such contamination. Trip blanks are issued to clients with each bottle order.

4.4 Contamination of the purge and trap system will occur when a sample is analyzed containing VOCs that exceed 100 µg/L. Erroneous positive results will occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing very high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations of volatile organic compounds, a calibration blank should be analyzed to check for cross contamination. This carry-over is minimized by application of single use glassware but not eliminated.

4.4.1 The purge and trap should be programmed to run through a “clean” cycle prior to the analysis of actual samples. This clean cycle is a two step process. In the first step, no purge tube is attached and for 1.5 minutes excess water is blown out of the purge lines. In the second step, a purge tube is attached and for 5 minutes clean helium gas is purged through the system. Finally, the sample is loaded into the purge tube through the 3-way valve without breaking the tube’s inert seal (See 9.6.1, Table 8).

4.4.2 Screening of unfamiliar samples prior to purge-and-trap GC/MS analysis is recommended to prevent contamination of the system. Screening of soil samples is accomplished by analyzing an aliquot of the soil methanol extract first by

Direct Injection GC/MS (see LAM-010). Alternative methods of screening soil extracts include GC with either electron capture detection (ECD) or flame ionization detection (FID). Screening of water samples is best accomplished with the purge and trap GC/MS system by performing a minimum 100 fold dilution on the sample. Alternatively, water samples may be screened by GC/ECD for common chlorinated VOCs – 1,1-DCE, 1,1-DCA, cis-1,2-DCE, PCE, TCE, and 1,1,1-TCA.

4.5 High levels of petroleum hydrocarbons, such as those found in diesel fuel, will generate interferences for 1,1,1,2-tetrachloroethane, 2-chlorotoluene, 4-chlorotoluene and 1,2,3-trichloropropane. Noisy quantitation ion profiles and poor qualifier ion matches associated with these compounds due to the petroleum interference will be evident during data analysis and will interfere with the accurate analysis of these compounds.

4.6 The internal standard, pentafluorobenzene, co-elutes and interferes with the qualifier ion for 1,1,1-TCA at m/z 61. Poor "Q Value" matches will result for 1,1,1-TCA from this interference at concentrations below 5 µg/L. Pentafluorobenzene elutes within the extraction windows for carbon tetrachloride at m/z 117 and 1,1-dichloropropene at m/z 75. These compounds may be detected by the contribution from pentafluorobenzene; however the retention time will not be accurate. Similarly, the surrogate, bromofluorobenzene, elutes within the extraction window of 1,2,3-trichloropropane at m/z 75.

4.7 Methanol from soil extracts and added to water samples interferes with bromomethane and chloroethane. The methanol has a quenching effect upon the operation of the MSD for the period that it co-elutes from the column with these compounds. The response of bromomethane and chloroethane is erratic and suppressed due to the methanol.

4.8 The response of the ketones (acetone, MEK, MIBK, 2-hexanone) and tetrahydrofuran is controlled by their purging efficiency. Their purging efficiency is affected by the ambient temperature of the sample in the purge tube. Every attempt should be made to maintain a stable temperature in the laboratory.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the ECCS Chemical Hygiene Plan (CHP), and this document. Refer to the CHP for more detailed safety information or for information not listed in this document.

5.2 Eye protection that protects against splash and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled during this procedure. Lab coats are recommended.

5.3 Employees must handle glassware and equipment carefully in order to prevent injury and accidents. Any damaged or broken glassware is to be discarded or moved to the glass repair box.

5.4 ECCS maintains a Material Safety Data Sheet (MSDS) for every chemical used in the laboratory. The MSDS file is kept in the main laboratory.

6 APPARATUS AND MATERIALS

6.1 Gas Chromatograph / Mass Spectrometer System

Gas Chromatograph:	HP 5890 with Split/Splitless Injector
Detector:	HP 5971 Mass Selective Detector
Purge and Trap:	2 Tekmar LSC 2000s and ALS 2016s with Duet interface
Trap:	Supelco Trap K
Column:	RTX-624, 30 m x 0.32 mm I.D., 1.8 µ film or equivalent
Data System:	Agilent MSD Productivity Chemstation with Enviroquant

6.1.1 GC/MS supplies list

Seals:	Dual Vespel Ring Inlet Seal, Restek Cat.#21239
Septa:	LB-2 Septa, 11mm, Supelco #164742
Ferrules:	1/16” x 0.5 mm Vespel/Graphite, Restek Cat# 20231/20249
Liners:	Gooseneck Splitless Liner 4 mm x 6.5 mm x 78.5 mm, Restek Cat.#20799

6.2 Computer Hardware: HP Compaq DC7600

6.3 Computer Software

6.3.1 Microsoft Windows 2000 operating system or equivalent current version

6.3.2 Agilent MS Productivity G1701BA with Enviroquant Data Analysis

6.3.3 Microsoft Office 2007 or equivalent current version

6.3.4 Promium Element Laboratory Information Management System (LIMS)

6.4 Shaker table

6.5 Ultrasonic bath

6.6 Centrifuge capable of holding 40 mL VOA vials

6.7 Balances

6.7.1 Top loader capable of weighting to 0.01 gram

6.7.2 Analytical capable of weighting to 0.0001 gram

6.8 Sample syringe: 10 mL Gastight with a Teflon® Luer-lock tip

6.9 Micro-syringes: Gastight, 10, 25, 100, 250, 500, and 1,000 µL

6.10 Vials:

6.10.1 40 mL VOA vials

6.10.2 40 mL VOA vials preserved with 0.5 mL of 1:1 HCL

6.10.3 40 mL VOA vials containing 10 mL of purge and trap grade methanol, labeled and pre-weighed. Used for sampling of soils using Lock and Load system. See GEN-003, Chain-of-Custody, Log In, Tracking Procedures, and Sample Tracking for more details.

6.10.4 GC vials: 2 mL amber autosampler vials Catalog. #40811A-120 and PTFE/silicon aluminum seals, Part #5150-11, LSDC

6.10.5 "Lock and Load" sampling supplies

6.10.6 Plastic syringes for collection of a nominal 10 gram soil sample, Environmental Sampling Supply Part # LL5035SRNG

6.10.7 Plastic re-usable handle, Environmental Sampling Supply Part # LL5035HNDL

6.11 Specimen container (5 oz.) with lid for collection of soils intended for percent solids analysis, VWR Part # 15704-020 and 15704-019.

6.12 Disposable single use glass purge tubes, 19 x 150 mm glass culture tubes, VWR Part # 45060-19150

6.13 Glass disposable transfer pipettes – 5 ¾"

6.14 Optifix solvent dispenser capable of 10 mL volume (EM Science or equivalent)

6.15 Stainless steel spatulas

6.16 Drying oven capable of maintaining 105 °C

6.17 Refrigerator capable of maintaining 4 °C

6.18 Freezer capable of maintaining temperatures below -15 °C

6.19 Plastic 250 to 500 mL squirt bottles

7 REAGENTS

7.1 Solvents

7.1.1 VOC-free water purchased commercially.

NOTE: Store away from contact with other solvents except methanol.

7.1.2 Methanol: purge & trap grade, stored away from contact with other solvents.

7.2 Solids – not applicable to this method

7.3 Acids/bases – not applicable to this method

7.4 Stock Standards

7.4.1 Stock initial calibration standard solutions - Stock solutions are purchased from vendors as certified concentrations in methanol. The vendor is Absolute Standards. The 54 liquids (Part # 32001), 6 gases (Part #30058), are purchased at 2000 µg/mL. The ketones (Part# 82492) and tetrahydrofuran (Part# 34191) are purchased at 20,000 ug/mL. Additional compounds to complete the target list, MTBE, carbon disulfide, di-isopropyl ether, hexane, 1,1,2-trichloro-1,2,2-trifluoroethane and 1,3,5-trichlorobenzene are purchased individually at 1000 µg/mL. Their respective part numbers are 70209, 70060, 70987, 70962, 70474 and 70409. Table 4 provides a listing of components in each stock solution. Stocks are used only once when the ampoule is opened and then diluted to prepare the working solution.

7.5 Intermediate Standard Solution:

7.5.1 (20/100/200 µg/mL)

7.5.1.1 The working intermediate standard solution (20/100/200 µg/mL) is prepared by diluting the stock standard solutions (See 7.4.1) in a 50 mL volumetric flask with methanol.

7.5.1.2 The working standard is prepared at 20 µg/mL for all targets except the ketones (200 µg/mL) and tetrahydrofuran (100 µg/mL).

7.5.1.3 Fill a 50 mL volumetric flask ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that a syringe needle tip can be submerged in the methanol.

7.5.1.4 Aliquots of the stock are added below the level of methanol in the flask from the syringe needle.

NOTE: Table 5 summarizes the preparation of this standard.

7.5.1.4.1 Using a 500 µL Gastight syringe, aliquot 500 µL of the 2000 µg/mL liquid (32001) and gas stock (30058) to the 50 mL volumetric.

7.5.1.4.2 Using a 1000 µL Gastight syringe, aliquot 1000 µL of the individual standards (70209, 70060, 70987, 70962, and 70474) to the 50 mL volumetric.

7.5.1.4.3 Using a 500 µL Gastight syringe, aliquot 500 µL of ketones (82492) and 250 µl of tetrahydrofuran (34191) to the 50 mL volumetric.

7.5.1.5 Once all components are added to the 50 mL volumetric, make to volume with purge and trap grade methanol.

7.5.1.6 The standard is subdivided into 2 mL GC vials. Make sure to fill each vial full and cap. Store frozen and assign a one month expiration date.

7.6 Calibration Standards: Prepare calibration standards directly in the 10 mL Luer-lock syringe at eight levels immediately prior to loading them onto the purge and trap autosampler (See Step 9.7.3).

7.7 Surrogate

7.7.1 See 7.11, internal standard/surrogate solution

7.8 Laboratory Control Sample (LCS) Stock Solution

7.8.1 The LCS spike mix is also the intermediate standard mix detailed in Section 7.5.

7.9 MS/MSD Spike Mix

7.9.1 The MS/MSD spike mix is also the intermediate standard mix detailed in Section 7.5.

7.10 LOD Spike Mix: The LOD spike mix is also the intermediate standard mix detailed in Section 7.5. Add 0.2µL of the intermediate standard to 10 mLs of reagent water when preparing the water LOD sample. Add 10µL of the intermediate standard to 10mL of methanol when preparing the soil LOD sample.

7.11 Second Source

7.11.1 Stock second source standard solutions: The second source stock standards are acquired from a different commercial vendor than that used for the initial calibration standard. This second source is Restek Corporation. A mega mix (Part #30431) contains the vast majority of the compounds at 2,000 µg/mL. The gases (Part #30042), and oxygenates (MTBE, di-isopropyl ether, Part #30465) are also acquired at 2000 µg/mL. The ketones (Part #30006) are acquired at 5,000 µg/mL. Table 4 provides a listing of components in each stock solution. A hexane stock is not available from Restek and therefore is not included in the

second source standard.

7.11.2 Intermediate second source standard (20/200 µg/mL)

7.11.2.1 Similar to the intermediate calibration standard, the second source standard is prepared at 20 µg/mL for all targets, except the ketones (200 µg/mL).

7.11.2.2 Fill a 25 mL volumetric flask ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that a syringe needle tip can be submerged.

7.11.2.2.1 Using a 500 µL Gastight syringe, aliquot 250 µL of 502.2Mega Mix (30431) to the 25 mL volumetric.

7.11.2.2.2 Using a 1000 µL Gastight syringe aliquot 1,000 µL of the ketone mix (30006) to the 25 mL volumetric.

7.11.2.2.3 Using a 500 µL Gastight syringe aliquot 250 µL of the six gases (30042) and 250 µL of the oxygenates (30465) into the 25 mL volumetric.

NOTE: Once vials are opened and aliquots taken, vials are discarded.

7.11.2.3 Make to volume with methanol, transfer to 2 mL GC vials and cap tightly.

7.11.2.4 Store frozen at all times and assign a one month expiration date.

7.12 Internal Standard/Surrogate

7.12.1 Stock internal standard and surrogate solutions: Stock solutions are acquired commercially as certified solutions in methanol from Restek. The internal standards (Part #30074) and surrogates (Part #30073) are purchased at 2,500 µg/mL. Table 4 provides a listing of the components in each stock solution.

7.12.2 Working internal standard/surrogate solution

7.12.2.1 The working internal standard/surrogate solution is prepared at 25 µg/mL by diluting the stock standard solutions (See 7.12.1) in a 100 mL volumetric flask. With a 2500 µg/mL stock internal or surrogate standard, 500 µL would be added to the flask. Table 5 summarizes preparation of this standard.

7.12.2.2 Fill a 50 mL volumetric ¾ full of purge and trap grade methanol known to be free of volatiles. Make sure the volumetric has enough methanol so that the syringe needle tip can be submerged below the level of the methanol.

7.12.2.3 Using a 1000 µL Gastight syringe aliquot 500 µL of internal standard Mix (#30074) and add to the 50 mL volumetric.

7.12.2.4 Using a 1000 µL Gastight syringe aliquot 500 µL of Surrogate Mix (#30073) and add to the 50 mL volumetric.

NOTE: Discard unused ampoules once aliquot has been taken.

7.12.2.5 Make to volume with purge and trap methanol.

7.12.2.6 Once prepared, the working internal standard/surrogate solution is then portioned into 2 mL GC vials for daily use. Each vial is filled to its neck and capped.

7.12.2.7 Assign an expiration date of three months, enter information into LIMS system and store frozen

7.13 BFB tuning standard (25 µg/mL)

7.13.1 The BFB Tuning Standard is also the Working Internal Standard/Surrogate solution (7.12.2).

7.13.2 Add 2µL of this standard to 10mL of deionized water resulting in 50 ng of BFB delivered to the GC/MS system.

8 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Water Samples

8.1.1 Glass 40-mL VOA vials, containing 0.5 mL of 1:1 HCl with Teflon® lined septa are provided by ECCS for water samples. Hydrochloric acid preserved VOA vials are purchased from a variety of vendors. Once VOA vials are received at ECCS laboratories they are stored in a refrigerator at 4 °C. Water samples preserved with 1:1 HCl have a hold time of 14 days. Water samples not preserved with 1:1 HCl have a hold time of 7 days.

NOTE: HCl is used to eliminate/retard degradation of compounds by bacteria.

NOTE: A temperature blank may be used if temperature reporting is critical.

8.2 Soil Samples

8.2.1 Methanol preserved

8.2.1.1 ECCS supplies a pre-weighed 40 mL VOA vial containing 10 mL of methanol for soil collection. In addition, ECCS supplies a Lock and Load T handle (See 6.11) and disposable sample container for collection of a pre-measured approximately 10 g of soil. The client is responsible to put the nominal 10 g of soil from the Lock and Load syringe into the VOA vial containing purge and trap methanol.. Soil samples received are stored in a refrigerator at 4 °C.

8.2.1.2 An alternative approach used with a mobile laboratory is for the client to collect the soil sample with a Lock and Load disposable syringe and quickly submit the core still contained in the capped syringe to the laboratory. The laboratory immediately extrudes the core into a tared 40 mL VOA vial containing 10mL of purge and trap methanol and measures the weight of the soil. Methanol preserved soil samples have a hold time of 14 days from time of collection.

8.2.2 Soil samples not preserved in methanol are out of control and require a note in the comments of the VOC Soil Weighing Log (See Figure 5) and in the case narrative. These soil samples are extracted as soon as practical.

9 PROCEDURE

9.1 Sample Preparation

9.1.1 Water Samples: Water samples are analyzed directly.

9.1.1.1 Withdraw 10 mL or an appropriate aliquot (based upon the screening analysis) of the water sample from the VOA vial into the 10 mL Luer lock syringe.

9.1.1.2 Tilt the VOA vial while withdrawing the sample to prevent inclusion of air bubbles.

9.1.1.3 Using a 250 µL syringe, add 200 µL of methanol through the Luer tip of the 10 mL syringe containing the unknown sample, Then add 10 µL of the surrogate/IS solution (See 7.12.2) with a 25 µL syringe.

9.1.1.4 Immediately transfer the sample from the 10 mL syringe into the Tekmar ALS 2016 purge tube assigned to that sample.

9.1.2 Preparation of water blanks

9.1.2.1 Fill a 10 mL Luer lock syringe with 10 mL of clean water.

9.1.2.2 . Add 10 µL of surrogate/IS solution (See 7.11.2) to the 10 mL of water.

9.1.2.3 Lock the syringe to the appropriate position on the Purge and Trap autosampler and transfer the blank into the purge tube.

9.1.2.4 Method blank frequency is 1 per purge and trap side/20 samples or less.

9.1.3 Preparation of LCS Samples: LCS samples for waters are identical to CCV standards, so LCS samples are not required for waters.

9.1.3.1 CCVs and LCSs are prepared the same.

9.1.3.2 Fill a 10 mL Luer lock syringe with 10 mL of clean water.

9.1.3.3 Add 10 µL of surrogate/IS solution (See 7.11.2) to the 10 mL of water.

9.1.3.4 Add 2.5 µL of the 20/200/100 µg/mL intermediate standard (See 7.5.1) to the 10 mL in the syringe.

9.1.3.5 Lock the syringe to the appropriate position on the purge and trap autosampler and transfer the CCV/LCS into the purge tube.

9.1.3.6 CCV/LCS frequency is 1 per purge and trap side/10 unknowns or tubes.

9.1.4 Matrix spike water samples: Load a duplicate volume of sample into the 10 mL Luer lock syringe as described in Section 9.1.5. Add 198 µL of methanol followed by 10 µL of the surrogate/IS solution (See 7.12.2). To fortify the sample, add 2.5 µL of the 20/200/100 µg/mL intermediate standard (See 7.5) resulting in an addition of 5.0 µg/L. The ketones and tetrahydrofuran are spiked at 50 and 25 µg/L, respectively. Lock the syringe to the appropriate position on the purge and trap auto-sampler and transfer the sample into the purge tube. Repeat the process for the matrix spike duplicate sample.

9.1.5 Soil Samples: Client supplied soil samples in non-ECCS containers, preserved with methanol – use weight of soil and volume of methanol supplied with the sample.

9.1.5.1 Client supplied soil samples, no methanol

9.1.5.1.1 Accurately weigh about 10 g of soil into a 40 mL VOA vial, record weight and add 10 mL of methanol.

9.1.5.2 Client supplied soil in Lock and Load syringe

9.1.5.2.1 This commonly occurs with use of the mobile laboratory

9.1.5.2.2 A 40 mL VOA vial is tared on a top loading balance and the soil core is extruded into the vial with the weight recorded to XX.XX g.

9.1.5.2.3 Immediately add 10 mL of methanol to the vial immersing the soil.

9.1.5.3 VOA vials supplied by ECCS with the Lock and Load supplies.

9.1.5.3.1 Each VOA vial contains 10 mL of methanol and the entire container was pre-weighed prior to shipment to the client. The total container weight to 0.XX g (Vial, label, and methanol) is recorded on the label with indelible marker.

9.1.5.3.2 Upon receipt at ECCS the vial is reweighted to 0.XX g. The weight is

recorded on the COC and the information is entered into the laboratory information management system (LIMS) either by sample entry or by the chemist prior to analysis.

9.1.5.3.3 The soil weight obtained is the wet weight to be used for sample analysis.

9.1.5.4 A paper record of the determination of soil weight is kept in the VOC Soil Weighing Log (See Figure 5).

9.1.6 Extraction by shaker table/sonication:

9.1.6.1 With clay and dense type soils it may be necessary to break up the soil clods into smaller pieces by mechanical means. A stainless steel spatula is used to manually break up the clods while immersed in methanol and in the VOA vial.

9.1.6.2 Position the ultrasonic bath on the shaker table platform so the long side of the bath is oriented in the same direction as the horizontal movement of the shaker table.

9.1.6.3 Place two foam VOA vial shipping blocks on each end the VOA vial containing the methanol preserved soil samples. Place the blocks and VOA vials into the sonicator lengthwise so the VOA vials are positioned on their sides. Place enough water in the bath to cover the top vials but keep the water level below 2 inches from the top of the sonicator. The water in the bath needs to be clean, free of dust and debris, in order to effectively transfer energy in the bath. It may be necessary to place weight on top of the vials and blocks to keep them submerged. Plastic ice blocks can be used for this purpose.

9.1.6.4 Adjust the variable speed of the shaker table to provide 120 cycles per minute of horizontal shaking without spilling water over the sides of the sonicator.

9.1.6.5 Sonicate and shake the samples for 20 minutes.

NOTE: The sonicator bath needs to be kept cool either by using ice or changing the water.

9.1.6.6 Allow the samples to equilibrate for approximately 20 minutes after sonication prior to opening the vial. Aliquoting extracts and archiving: .Allow the soil to settle from the upper layer of clear methanol extract - usually ½ hour. Alternatively, the vial may be centrifuged at a low speed (<1500 rpm) to separate the clay soils from the extract. If the vial is centrifuged, still allow ½ hour before opening the vial.

9.1.6.7 VOA vials containing soil extracts may be stored in a refrigerator or a

portion may be transferred to a crimp top vial for storage in a freezer.

9.2 Clean-up Methods - not applicable for this method

9.3 Instrument Conditions

9.3.1 GC/MSD pump down: Allow the GC/MS system to stabilize under high vacuum when starting from a vented state. Before analysis, allow at least 4 hours for the MS to stabilize after pump-down.

9.3.2 Purge and trap & GC/MS operating conditions

9.3.2.1 Purge and trap:

Purge Flow:	Helium, 35 mL/min.
Purge Time:	11 minutes
Dry Purge:	2 minutes
Desorb:	1 minute at 250 °C
Bake Time:	8 minutes at 260 °C

9.3.2.2 GC conditions:

Column:	DB-624, 30 m x 0.32 mm, 1.8 µm film
Injector Temp:	200 °C
Transfer Line Temp:	280 °C
Column Head Pressure:	2 PSI
Column Flow:	1.7 mL/min
Split Flow:	120 mL/min
Split Ratio:	82:1

Oven Temperature Program	
Initial Temperature:	45 °C
Initial Hold:	4 minutes
Initial Ramp:	23 °C/ minute
Final Temperature:	240 °C
Final Hold:	2.5 minutes

9.3.2.3 MS conditions

Tune:	maximum sensitivity autotune
EM Voltage:	2000 (200 above autotune)
Scan Range:	45-260 amu
Scan Time:	2 scans/sec

9.3.3 Screening Methods

9.3.3.1 Water samples or soil extracts should be initially screened by one of the two

following methods for potentially high levels of VOCs that might otherwise contaminate the purge and trap system.

9.3.3.2 Screening by purge and trap

9.3.3.2.1 A 100 fold dilution is a good starting point when using the purge and trap to screen water samples or soil extracts.

9.3.3.2.2 Pull up 9.9 mL of clean water into the 10 mL Gastight syringe.

9.3.3.2.3 Using a 100 µL Gastight syringe, aliquot 100 µL of the unknown water sample or 2 µL of the soil extract and add to the 10 mL syringe.

9.3.3.2.4 Add 200 µL of methanol and 10 µL of the surrogate/IS solution (7.12.2) and transfer the sample to the Tekmar 2016 purge and trap autosampler.

9.3.3.3 Direct inject GC/MS screening: See LAM-010 .

9.4 Preventative Maintenance/Troubleshooting

9.4.1 Cleaning the purge and trap system: To minimize the possibility of carryover from occurring between analyses the purge and trap should be cleaned and programmed to run through a "clean cycle" prior to the analysis of actual samples, especially whenever high concentrations of VOCs, above 50 µg/L, have been previously run on the system. Cleaning the purge and trap involves swabbing the outside of the Luer lock threads and purge needle on the purge and trap with a clean "Kimwipe" moistened with reagent water. Reagent water is then used to rinse the 3-way valve and purge needle. The purge and trap "clean cycle" is a two step process. In the first step, no purge tube is attached and for 1.5 minutes excess water is blown out of the purge lines. This step uses Method 2 on the purge and trap. In the second step, a new purge tube is attached and for 5 minutes clean helium gas is purged through the system. This step uses Method 3 on the purge and trap. The tube is never removed after the second step and the sample is loaded to the tube through the 3-way valve. The very short, dummy GCMS acquisition is performed during these two steps so the duet box will switch and alternate between the two purge and trap systems. Table 8 summarizes the purge and trap and GCMS conditions used for this "clean cycle."

9.4.2 The maximum sensitivity autotune report (See Figure 1) for the MSD serves as a useful diagnostic tool. It can be used to indicate when maintenance on the MSD is required.

9.4.2.1 Detection of leaks in the GC/MS system: The presence of a base peak at m/z 28 (N2) that is higher than m/z 69 (base peak of PFTBA) in the tune report indicates the presence of a gross leak in the GC/MS vacuum system. A common source of the leak is a loose transfer line nut sealing the GC capillary column to the MSD transfer line. Tightening this nut often

eliminates a leak.

9.4.2.2 Water in the GC/MS system: The presence of a base peak at m/z 18 (H20) that is higher than m/z 69 in the tune report indicates excessive water remains in the MSD manifold. The MSD vacuum system does not efficiently remove water and this condition indicates that a longer equilibration time is needed prior to initial calibration.

9.4.2.3 Electron multiplier voltage: An electron multiplier voltage higher than 2700 in the tune report indicates that the multiplier needs replacement and/or the MSD source needs cleaning.

9.4.2.4 Peak shape/resolution of PFTBA calibration peaks: The appearance of the PFTBA peaks (m/z 69, 219, 502) used to calibrate the MSD should be symmetric without any shoulders. Isotope masses of these same peaks (m/z 70, 220, 503) should be present and indicated in the auto tune report. Non-symmetric peak shape or the non-detection of isotope masses usually indicates that the MSD source needs to be cleaned.

9.4.3 The response factors of system performance check compounds (SPCCs) calculated during the initial calibration also serve as useful diagnostic tool. SPCCs are used to check that purge and trap and GC conditions are appropriate and that compound degradation is acceptable. Chloromethane will be low if the purge flow is too fast while bromoform will be low if the flow is too slow. 1,1-DCA and especially 1,1,2,2-tetrachloroethane will be low due to degradation from active sites on contaminated transfer lines or valves in the purge and trap system. Clipping the front end of the GC column can be the first action taken to eliminate active sites. Flushing the lines and valves of the purge and trap system with 1:1 HNO_3: water, de-ionized water, and methanol may further remove the active sites. Finally, it is often necessary to totally replace contaminated lines or valves in the purge and trap system when active sites are encountered.

9.5 Retention Time Windows: The retention time window for extracted ion chromatograms in peak identification is 0.6 minutes wide. The GC/MS software then identifies the peak in this window with the relative retention time closest to the actual relative retention time determined from the authentic standard. The window must be this wide in order for the software's integration algorithm to have sufficient time to accurately calculate baselines as part of peak integration. The absolute retention time of target compounds should not vary by more than ± 0.05 minutes when compared to the authentic standard. However, retention times are used, only in part, to qualitatively identify compounds as in conventional GC analysis. Rather, the GC/MS method also utilizes the mass spectra of peaks for qualitative identification of target compounds.

9.6 Instrumental Analysis

9.6.1 Using the results of a few test analyses, adjust the electron multiplier voltage for

the MSD tune file accordingly to achieve approximately 750,000 thousand area counts for the internal standard, 1,4-difluorobenzene, Analyze a low standard at 0.5 µg/L to verify adequate sensitivity once the EM voltage has been adjusted.

9.6.2 The GC/MS system is software-tuned by successfully completing a maximum sensitivity autotune. Perflurotributylamine (PFTBA) is the tuning compound used by the MSD during the maximum sensitivity autotune. A hardcopy report is generated from the tune (See Figure 1). See Section 7.6.2 for interpretation of the tune report. Further manual adjustments to the MSD optics settings may be also be necessary in order to successfully pass the following BFB tuning evaluation.

9.6.2.1 BFB tuning evaluation: Introduce 50 ng of the BFB tuning standard (See 7.13) into the Purge and trap GC/MSD system..

9.6.2.2 The mass spectra acquired from the BFB tuning standard must pass the acceptance criteria provided in Table 9

9.6.2.3 The BFB mass spectra used for tune evaluation may be a single scan or an average using any combination of the three individual MSD scans for BFB including the peak apex and the two scans to the right and/or left of the peak apex. Using a background subtracted BFB mass spectra is preferred using an appropriate background region adjacent to the BFB peak.

9.6.3 Calibration Standards: A separate series of calibration standards must be analyzed on each of the two Tekmar 2016 auto-samplers used with the Tekmar Duet purge and trap configuration. Similarly, separate quantitation methods are used to calculate VOC concentrations for samples analyzed on each of the two auto-samplers.

9.6.3.1 Prepare calibration standards as outlined in Section 9.6.4 to be used for both soil and water samples. The total volume of methanol added to calibration standards is 210 µL. When analyzing waters only, a separate calibration may be performed using less methanol in order to increase accuracy of bromomethane and chloroethane. Prepare a second source verification standard from the intermediate second source standard (See 7.10.2) at 5 µg/L and analyze with each initial calibration on Side A and Side B of the purge and trap.

9.6.4 Accurate use of micro-syringes: Care must be exercised when making a measurement with a micro-syringe that air bubbles are not included and that the syringe barrel does not partially drop during handling thus ejecting a portion of the aliquot. Air bubbles can be eliminated from the syringe by lifting and pumping the syringe plunger quickly a few times prior to measuring the aliquot. Draw the aliquot up slowly for the final measurement. Do not invert the vial containing a standard while filling the syringe as excess standard will run down the outside needle of the syringe resulting in a high bias. When handling an aliquot contained in a syringe, hold the syringe in a manner that a finger is slightly

pushing against the extended plunger of the syringe to prevent its movement.

9.6.5 Standard curve preparation

9.6.5.1 The total volume of methanol for initial calibration standards is 210µL. To prepare the level 1 ICAL (0.5 µg/L) add 200 µL of makeup methanol through the Luer tip into the 10 mL of water in the 10 mL syringe. Using a 10 µL syringe add 10 µL of Surrogate/IS (See 7.12.2) through the Luer tip in to the 10 mL of water. Using a 1 µL syringe add 0.25 µL of spike mix (See 7.5.1) through the Luer tip into the 10 mL of water.

9.6.5.2 Immediately transfer the standard into the Tekmar ALS 2016 tube assigned to that ICAL level.

9.6.5.3 ICAL Levels L-2 through L-8 follow the ICAL grid as listed in Table 6.

9.6.5.4 Analyze each of the calibration standards listed in Section 9.6.4, collect the data and tabulate the area response of the characteristic ions against the concentration for each target compound using internal standard method. Characteristic ions are listed in Table 3. The internal standard selected for the calculation of these ratios should be the internal standard that has a retention time closest to the compound being measured. The internal standard used for each target compound is provided in Table 7. Calculate amount ratios and response ratios for each target compound and at each calibration level relative to the appropriate internal standard.

The amount ratio and response ratios are calculated as follows:

$$Amount\ Ratio = \frac{C(s)}{C(is)}\ Response\ Ratio = \frac{A(s)}{A(is)}$$

Where:

A(s) = Peak area of the target compound or surrogate
A(is) = Peak area of the internal standard
C(s) = Concentration of the target compound or surrogate in µg/L
C(is) = Concentration of the internal standard in µg/L

9.6.5.5 Calculation of response factors (RF) and percent relative standard deviation (% RSD): Calculation of response factors are used to evaluate the initial calibration against specific criteria listed in method 8260 and is also used to quantitate sample concentrations. Individual response factors are calculated by the following formula for each calibration level:

$$Response\ Factor\ (RF) = \frac{Response\ Ratio}{Amount\ Ratio}$$

The % RSD is calculated by the following formula:

$$\% RSD = \left(\frac{RF_{STDEV}}{RF_{AVE}}\right) \times 100$$

Where:
RF_{STDDEV} = Standard deviation of individual response factors
RF_{AVE} = Average of individual response factors

NOTE: Chemstation has software to calculate and generate a response factor report for every ICAL generated. See Figure 4 for example.

9.6.5.6 Calibration check compounds (CCCs): The % RSD of the response factors must be < 30% for individual CCCs in order for the initial calibration to be valid. The CCCs are:

vinyl chloride	1,1-dichloroethene
chloroform,	1,2-dichloropropane
toluene	ethylbenzene

9.6.5.7 The % RSD for other target compounds must also be less than 30%, except for 1,2-dibromo-3-chloropropane and naphthalene which must be less than 40%. In addition, bromomethane, chloroethane, acetone, 2-butanone, tetrahydrofuran, methyl isobutyl ketone, and 2-hexanone are exempt from a % RSD requirement. These compounds must meet a linear calibration criteria of correlation coefficient (r^2) of > 0.990.

9.6.5.7.1 If the correlation coefficient or % RSD does not meet the acceptance criteria for any compound then the integrity of the system is failing and corrective action is needed to eliminate a system leak and/or active sites (See 9.6). Visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites, or poor chromatographic behavior. Polar compounds, like acetone, purge poorly and will always exhibit an erratic response.

9.6.5.8 System performance check compounds (SPCCs) are monitored for a minimum average response factor (RF_{AVE}) from the initial calibration data. Figure 4 provides a data system report listing a summary of response factors. The SPCCs and the minimum average response factor are:

chloromethane	0.10	1,1-dichloroethane	0.10
chlorobenzene	0.30	bromoform	0.10
1,1,2,2-tetrachloroethane	0.30		

9.6.5.9 The average response factor is determined by the data system from the mean of individual response factors by the formula:

9.6.5.10 $$RF_{AVE} = \frac{\Sigma\left(\frac{Response\ Ratio}{Amount\ Ratio}\right)}{N}$$

9.6.5.11 Where: N = Number of standards used in ICAL.

9.6.5.12 The average RF calculated from the calibration data must be greater than the minimum average response factors in order for the initial calibration to be valid.

9.6.5.13 SPCCs are used to check that purge and trap and GC conditions are appropriate and that compound degradation is acceptable. Chloromethane will be low if the purge flow is too fast while bromoform will be low if the flow is too slow. 1,1-Dichloroethane and especially 1,1,2,2-tetrachloroethane will be low due to degradation from active sites on contaminated transfer lines or valves in the purge and trap system.

NOTE: Typically, the first compound to fail is the 1,1,2,2-TCA. If failure occurs, the system may need to be cleaned, non use of purge & trap location, and/or the 1,1,2,2-TCA data must be qualified. If problems persist the unit should be removed from service and maintenance performed as noted in other sections of this method.

9.6.5.14 Use of linear calibration curves: Linear calibration curves are used for select target compounds because they typically have high % RSDs. These compounds include:

bromomethane	chloroethane	acetone
2-butanone	tetrahydrofuran	methyl isobutyl ketone
2-hexanone		

9.6.5.14.1 Bromomethane and chloroethane exhibit erratic response due to methanol quenching of the MSD. The ketones and tetrahydrofuran exhibit erratic purging efficiencies. Plot the response ratio against the amount ratio for each of these target compounds. The curve fit is linear regression with the weight of the inverse of the concentration. The linear regression equation is:

$$Y = m \times x + b$$

Where:
Y = response ratio
m = slope
x = amount ratio
b = intercept
1/x = weighting factor

9.6.5.14.2 The correlation coefficient for these target compounds must be > 0.990.

9.6.6 Updating qualifier ion abundances: The relative responses of qualifier ions for target compounds are updated through the data system with each initial

calibration. The 5 µg/L initial calibration standard is used for this purpose. Mass spectra of peaks found in samples are compared to these qualifier ion responses and the purity of the match is included in the report as a Q value. A Q value above 90 typically indicates the qualitative identification of a target compound in samples. The same qualifier ion ratios are included as a unique window in the Qedit data analysis menu and can further assist the mass spectrometrist during data review.

9.6.7 Second Source Verification: Analyze the second source standard with each initial calibration performed. The second source standard is prepared at 5 ug/l for all target compounds except the ketones at 50 ug/l. Prepare the second source standard by adding 198 µL of makeup methanol to the 10 mL syringe followed by 10 µL of the combined internal standard/surrogate solution (7.12.2). Add 2.5 µL of the intermediate second source (7.11.2) to the 10 mL syringe and load the standard on the 2016 purge and trap autosampler.

9.6.8 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV):

9.6.8.1 The working calibration curve must be verified on each analysis day and every 10 injections and at the end of the run. The concentration of the CCV is 5 µg/L, except for the ketones (50 µg/L) and tetrahydrofuran (25 µg/L). The percent difference between the response factor for the CCCs in the CCV standard and the average response factor determined from the initial calibration data must not vary by more than 20% in order for the calibration to remain valid. If the response factor of any CCC varies by more than 20%, then a new calibration curve must be prepared and affected samples reanalyzed or the data must be appropriately qualified.

$$Percent\ Difference = \left| \frac{(RF_{AVE} - RF_{CCV})}{RF_{AVE}} \times 100 \right|$$

Where:
RF_{AVE} = average response factor from the initial calibration
RF_{CCV} = response factor for the CCV

9.6.8.2 The percent difference must not vary by more than 30% for other target compounds. Initiate corrective action only if a non CCC compound exceeds the 30% limit for any two sequential CCVs. Bromomethane, chloroethane, acetone, 2-butanaone, MEK, tetrahydrofuran, 2-hexanaone and naphthalene are allowed a 40% percent difference.

9.6.8.3 In addition, the SPCCS in the CCV must meet their minimum response factor specified in Section 9.7.5.3 in order for the initial calibration to remain valid.

9.6.8.4 Figure 4 provides a data system report of the % difference between

continuing calibration response factors and the initial calibration average response factors.

9.6.9 Water Sample Analysis: Withdraw 10 mL or an appropriate aliquot (based upon the screening analysis) of the water sample from the VOA vial into the 10 mL Luer lock syringe.

9.6.9.1 Tilt the VOA vial while withdrawing the sample to prevent inclusion of air bubbles.

9.6.9.2 , Then add 10 µL of the surrogate/IS solution (See 7.12.2) with a 25 µL syringe.

9.6.9.3 Immediately transfer the sample from the 10 mL syringe into the Tekmar ALS 2016 purge tube assigned to that sample.

9.6.9.4 Preparation of water blanks

9.6.9.4.1 Fill a 10 mL Luer lock syringe with 10 mL of clean water.

9.6.9.4.2 Add 10 µL of surrogate/IS solution (See 7.12.2) to the 10 mL of water.

9.6.9.4.3 Lock the syringe to the appropriate position on the Purge and Trap autosampler and transfer the blank into the purge tube.

9.6.9.4.4 Method blank frequency is 1 per purge and trap side/20 samples or less.

9.6.9.5 Preparation of LCS Samples: LCS samples for waters are identical to CCV standards, so LCS samples are not required for waters.

9.6.9.6 Water CCV Preparation

9.6.9.6.1 Fill a 10 mL Luer lock syringe with 10 mL of clean water.

9.6.9.6.2 Add 10 µL of surrogate/IS solution (See 7.12.2) to the 10 mL of water.

9.6.9.6.3 Add 2.5 µL of the 20/200/100 µg/mL intermediate standard (See 7.5.1) to the 10 mL in the syringe.

9.6.9.6.4 Lock the syringe to the appropriate position on the purge and trap autosampler and transfer the CCV/LCS into the purge tube.

9.6.9.6.5 CCV frequency is 1 per purge and trap side/10 unknowns or tubes.

9.6.9.7 Matrix spike water samples: Load a duplicate volume of sample into the 10 mL Luer lock syringe as described in Section 9.1.5. Add 10 µL of the surrogate/IS solution (See 7.12.2). To fortify the sample, add 2.5 µL of the 20/200/100 µg/mL intermediate standard (See 7.5.1) resulting in an addition

of 5.0 µg/L. The ketones and tetrahydrofuran are spiked at 50 and 25 µg/L, respectively. Lock the syringe to the appropriate position on the purge and trap auto-sampler and transfer the sample into the purge tube. Repeat the process for the matrix spike duplicate sample.

9.6.10 Soil Aliquot and Analysis

9.6.10.1 Withdraw 10 mL of reagent water into the 10 mL Luer lock syringe.

9.6.10.2 Add 10 µL of the surrogate/IS solution (See 7.12.2) directly into the reagent water in the 10 mL syringe.

9.6.10.3 Using a 250 µL Gastight syringe, remove 200 µL of the soil extract from the 40 mL soil sample VOA vial and add directly into the 10 mL syringe.

9.6.10.4 In all cases, the total volume of methanol in the 10 mL of reagent water prior to loading into the purge and trap is 210 µL.

9.6.10.5 If high levels of VOCs are encountered during a screening process than add the appropriate volume of soil extract followed by addition of methanol so the total volume of methanol equals 210 µL.

9.6.10.6 Immediately transfer the 10 mL of reagent water containing the soil extract in the 10 mL Luer lock syringe into the purge and trap.

9.6.10.7 Blank for soils: Prepare a blank for soils in a 40 mL VOA vial with 10 mL of methanol.

9.6.10.7.1 Extract and sonicate the blank with the soil samples starting with Step 9.1.6. Analysis requires one blank/20 unknowns or less per day.

9.6.10.7.2 The LCS is carried through the procedure as though it were a sample.

9.6.10.8 LCS for Soils

9.6.10.8.1 Prepare a LCS for soils in a 40 mL VOA vial with 10 mL of methanol.

9.6.10.8.2 Add 125 µL of the intermediate initial calibration standard to the 10mL of methanol in the 40 mL VOA vial.

9.6.10.8.3 Extract and sonicate the blank with the soil samples starting with Step 9.1.6. Analysis requires one blank/20 unknowns or less per day.

9.6.10.8.4 The LCS is carried through the procedure as though it were a sample.

9.6.10.9 Matrix spike (MS), matrix spike duplicate (MSD) soil samples:

9.6.10.9.1 Select a soil sample extract for MS/MSD analysis.

9.6.10.9.2 Withdraw 10mL of reagent water into the 10 mL syringe.

9.6.10.9.3 Load a duplicate (200 µL) volume of soil extract from the 40 mL VOA vial into the 10 mL Luer lock syringe.

9.6.10.9.4 Add 10 µL of the surrogate/IS solution (See 7.12.2) directly into the 10 mL syringe through the Luer lock end.

9.6.10.9.5 To fortify the sample by adding 2.5 µL of the 20/200/100 µg/mL intermediate standard (See 7.5.1) directly into the 10 mL syringe through the Luer lock end.

NOTE: Resulting fortified concentrations are 5/25/50 µg/L.

9.6.10.9.6 Repeat steps 9.6.10.9.2 through 9.6.10.9.5 to prepare the matrix spike duplicate sample.

9.6.10.10 Continuing calibration verification (CCV) is required after every 10 unknown samples and at the end of every run.

9.6.10.10.1 CCVs for soils are prepared the same as water CCVs (See 9.6.9.6)

9.6.11 Qualitative analysis

9.6.11.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

9.6.11.2 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

9.6.11.3 The absolute retention time of the sample component is within ± 0.05 minutes of the absolute retention time of the standard component.

9.6.11.4 The relative intensities of the characteristic ions agree with 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20 % and 80 %.)

9.6.11.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

9.6.11.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte background spectra is important.

9.6.11.7 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

9.6.12 Calculations

9.6.12.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the extracted ion current profile of the primary characteristic ion. The concentration in the sample analysis is determined using the average response factor as described in Section 9.6.5.1. Linear regression analysis is used for the select compounds described in Section 9.765.8.

9.6.12.2 Water Sample Calculations: Concentrations of VOCs in water are determined directly from the average response factor and multiplied by any dilution factor:

$$\mu g/L\ water\ sample = \mu g/L\ measured \times DF$$

Where: DF = Dilution factor, based on 10 mL of sample in the purge tube (e.g. If 10 mL of sample is used, the DF =1; likewise, if 2 mL of sample was used with 8 mL water, the DF = 5.)

9.6.12.3 Soil sample calculations: Concentrations of VOCs in soil are determined on a dry-weight basis by the following formula:

$$\mu g/kg\ soil\ sample = (\mu g/L\ measured \times 0.01L) \times \left(\frac{V_m/V_p}{W_s}\right) \div FS$$

Where:
V_m = Volume of methanol used to extract samples in mL, normally 10 mL

V_p = Volume of methanol extract purged in mL, normally 0.200 mL
W_s = Wet weight of soil extracted in kg, nominally 0.010 kg
FS = Fraction of dry solids of in the soil sample, approximately 0.85

9.6.12.3.1 Soil sample correction factor by LIMS: The correction factor for calculating soils from uploaded GC/MS data is established in the bench sheet by setting the initial sample volume at 10 mL and the final sample volume to 500 mL. This multiplier of 50 converts µg/L results from the uploaded GC/MS data to µg/kg results for methanol extracted soils Exact soil weights are entered into the LIMs batch when calculating soil results. The fraction of dry solids in samples is determined in a separate, % solids batch and this fraction is used by the LIMS when calculating soil results.

9.6.12.4 Where applicable, the concentration of any non-target analytes identified in the sample should be estimated by the same formula, except the areas A(x) and A(is) should be from the total ion chromatograms, and the response ratio for the compound should be assumed to be 1. The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

10 QUALITY CONTROL

10.1 Refer to Method 8000 for general quality control procedures for chromatography methods.

10.2 The MSD must successfully complete both a maximum sensitivity autotune and a BFB tune evaluation (See 9.6.1 and Table 9).

10.3 Initial calibration must consist of at least 5 points from the available 7 calibration standards. The % RSD of response factors must be less than 30% for individual CCCs (See 9.6.5.2). The % RSD of the remaining target compound must be less than 30% except as noted in Section 9.6.5.3.

10.4 An analytical batch consists of 20 or fewer samples. Batch quality control samples should be analyzed with each set with the following frequency:

Blanks - One per 20 or fewer samples, minimum one per day
LCSs - One per 20 or fewer samples, minimum one per day
MS/MSDs - One MS/MSD per 20 or fewer samples, minimum one set per day

10.5 Method blanks consist of an aliquot of laboratory reagent water or methanol 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 procedure. If target analytes or interferences are present at concentrations that impact the analytical results for samples, the samples (including quality control samples) should be re-extracted or appropriately qualified in accordance with GEN-015, Qualification of

Data.

10.6 LCSs are prepared and processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. An LCS is not required for water analysis as it is the same as a CCV. Soil LCS control limits for precision and accuracy are established on at least a yearly basis through the use of at least 20 data points. If the recovery of any of the target analytes is outside control limits, the samples (including quality control samples) should be re-extracted or appropriately qualified in accordance with GEN-015, Qualification of Data.

10.7 MS/MSD samples consist of duplicate aliquots of sample spiked with the target analytes., MS/MSD control limits for precision and accuracy are established on at least a yearly basis through the use of at least 20 data points. MS/MSD control limits are advisory. If the recovery or RPD of any of the target analytes is outside control limits, data should be appropriately qualified in accordance with GEN-015, Qualification of Data.

10.8 The working calibration curve must be verified on each working day by the injection of one or more CCV standards. The percent difference between the response factors for the CCCs in the CCV standard must be within 20% of the average response factor calculated from the initial calibration (See 9.6.8.). The percent difference of the remaining target compounds in the CCV must not vary by more than 30% for each of any two sequential CCVs. The response factor determined for SPCCs in the CCV must also exceed their minimum response factor (See 9.6.5.4). If any CCC or SPCC in a CCV fails, a new calibration curve must be prepared or data appropriately qualified in accordance with GEN-015, Qualification of Data.

10.9 Surrogates are added to every sample and QC sample. Surrogate control limits are generated on at least a yearly basis. If a surrogate recovery is outside of control limits, the sample should be re-extracted and re-analyzed, if possible. If not, the data should be appropriately qualified in accordance with GEN-015, Qualification of Data.

10.10 A second source calibration verification standard must be analyzed with every initial calibration. The accepted limits are 80-120% for all analytes. If an SCV fails, immediate corrective action is required before proceeding with sample analysis. Affected data should be qualified according to GEN-015, Qualification of Data.

10.11 Internal standards are added to every injection vial. The response of the internal standards must not vary by < 50% or > 200% from the initial response of the mid-point standard in the initial calibration.

11 PERFORMANCE DATA

11.1 Limit of detection s (LODs) have been determined for waters and soils from the analysis of seven replicates fortified at 0.50 µg/L and 25 µg/kg, respectively. The LODs are presented in Table 1.

11.2 Demonstration of capability (DOC) was performed for water samples by analyzing 4

replicates spiked at 5 µg/L. The DOC summary for waters is listed in Table 2. DOC was also performed for soils at 250 µg/kg. The DOC summary for soils is provided in Table 2.

12 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

12.1 Contingencies for out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and filed with the Quality Manager. Analytical results shall be qualified as necessary.

13 WASTE MANAGEMENT / POLLUTION PREVENTION

13.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

14 REFERENCES

14.1 Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Method 8260B, SW-846, Test Methods for Evaluating Solid Wastes, Update III Revision 2, December 1996.

14.2 Purge and Trap of Aqueous Samples, Method 5030B, SW-846, Test Methods for Evaluating Solid Wastes, Update III Revision 2, December 1996.

14.3 Determinative Chromatographic Separations, Method 8000B, SW-846, Test Methods for Evaluating Solid wastes, Update III, Revision 2, December 1996.

14.4 Analytical Detection Limit Guidance & Laboratory Guide for Determining Method Detection Limits, Wisconsin Department of Natural Resources Laboratory. Certification Program, April 1996, PUBL-TS-056-96

TABLE 1

LODS AND REPORTING LIMITS

WATER SAMPLES

Compound	LOD (µg/L)	Reporting Limit (µg/L)
Dichlorodifluoromethane	0.092	0.50
Chloromethane	0.12	1.0
Vinyl chloride	0.078	0.50
Bromomethane	2.2	5.0
Chloroethane	0.75	5.0
Trichlorofluoromethane	0.12	0.50
1,1,2-Trichlorotrifluoroethane	0.099	0.50
1,1-Dichloroethene	0.15	0.50
Acetone	7.7	20
Carbon disulfide	0.073	0.50
Methylene chloride	0.049	0.50
Methyl-tert-Butyl ether	0.10	0.50
trans-1,2-Dichloroethene	0.13	0.50
n-Hexane	0.095	0.50
1,1-Dichloroethane	0.15	0.50
Diisopropyl ether	0.12	0.50
2,2-Dichloropropane	0.092	0.50
cis-1,2-Dichloroethene	0.15	0.50
2-Butanone (MEK)	6.6	20
Tetrahydrofuran	3.4	10
Bromochloromethane	0.12	0.50
Chloroform	0.11	0.50
1,1,1-Trichloroethane	0.085	0.50
Carbon tetrachloride	0.087	0.50
1,1-Dichloropropene	0.10	0.50
Benzene	0.064	0.50
1,2-Dichloroethane	0.13	0.50
Trichloroethene	0.096	0.50
1,2-Dichloropropane	0.11	0.50
Dibromomethane	0.11	0.50
Bromodichloromethane	0.13	0.50
cis-1,3-Dichloropropene	0.087	0.50
Methyl isobutyl ketone (MIBK)	6.0	20
Toluene	0.12	0.50
trans-1,3-Dichloropropene	0.18	0.50
1,1,2-Trichloroethane	0.072	0.50
Tetrachloroethene	0.10	0.50
1,3-Dichloropropane	0.10	0.50

TABLE 1 (CONT.)

LODS AND REPORTING LIMITS

WATER SAMPLES

Compound	LOD (µg/L)	Reporting Limit (µg/L)
2-Hexanone	7.5	20
Chlorodibromomethane	0.076	0.50
1,2-Dibromoethane	0.087	0.50
Chlorobenzene	0.071	0.50
1,1,1,2-Tetrachloroethane	0.14	0.50
Ethylbenzene	0.10	0.50
m+p-Xylenes	0.10	0.50
o-Xylene	0.097	0.50
Styrene	0.075	0.50
Bromoform	0.12	0.50
Isopropylbenzene	0.066	0.50
1,1,2,2-Tetrachloroethane	0.092	0.50
Bromobenzene	0.092	0.50
1,2,3-Trichloropropane	0.17	1.0
n-Propylbenzene	0.083	0.50
2-Chlorotoluene	0.093	0.50
1,3,5-Trimethylbenzene	0.072	0.50
4-Chlorotoluene	0.068	0.50
tert-Butylbenzene	0.098	0.50
1,2,4-Trimethylbenzene	0.096	0.50
sec-Butylbenzene	0.066	0.50
1,3-Dichlorobenzene	0.081	0.50
p-Isopropyltoluene	0.082	0.50
1,4-Dichlorobenzene	0.061	0.50
n-Butylbenzene	0.071	0.50
1,2-Dichlorobenzene	0.087	0.50
1,2-Dibromo-3-chloropropane	0.19	0.50
1,2,4-Trichlorobenzene	0.076	1.0
Hexachlorobutadiene	0.12	1.0
Naphthalene	0.53	5.0
1,2,3-Trichlorobenzene	0.067	1.0

Waters analyzed in GCIS-1629 and GCIS-1636, March 2007

TABLE 1-1

LODS AND REPORTING LIMITS

SOIL SAMPLES

Compound	LOD (µg/kg)	Reporting Limit (µg/kg)
Dichlorodifluoromethane	4.2	25
Chloromethane	8.4	25
Vinyl chloride	5.0	25
Bromomethane	890	1000
Chloroethane	160	500
Trichlorofluoromethane	5.8	25
1,1,2-Trichlorotrifluoroethane	3.9	25
1,1-Dichloroethene	6.2	25
Acetone	1300	1500
Carbon disulfide	3.4	25
Methylene chloride	4.4	25
Methyl-tert-Butyl ether	9.6	25
trans-1,2-Dichloroethene	5.2	25
n-Hexane	4.4	25
1,1-Dichloroethane	2.4	25
Diisopropyl ether	7.8	25
2,2-Dichloropropane	5.7	25
cis-1,2-Dichloroethene	3.9	25
2-Butanone (MEK)	1000	1500
Tetrahydrofuran	600	1500
Bromochloromethane	4.8	25
Chloroform	5.8	25
1,1,1-Trichloroethane	4.2	25
Carbon tetrachloride	4.3	25
1,1-Dichloropropene	3.4	25
Benzene	2.5	25
1,2-Dichloroethane	6.0	25
Trichloroethene	4.6	25
1,2-Dichloropropane	5.0	25
Dibromomethane	11	25
Bromodichloromethane	4.8	25
cis-1,3-Dichloropropene	4.8	25
Methyl isobutyl ketone (MIBK)	840	1500
Toluene	4.1	25
trans-1,3-Dichloropropene	5.7	25
1,1,2-Trichloroethane	5.0	25
Tetrachloroethene	4.9	25
1,3-Dichloropropane	8.0	25

TABLE 1-1 (CONT.)

LODS AND REPORTING LIMITS

SOIL SAMPLES

Compound	LOD (µg/kg)	Reporting Limit (µg/kg)
2-Hexanone	950	1500
Chlorodibromomethane	6.0	25
1,2-Dibromoethane	6.0	25
Chlorobenzene	2.1	25
1,1,1,2-Tetrachloroethane	4.8	25
Ethylbenzene	1.2	25
m+p-Xylenes	4.5	25
o-Xylene	3.6	25
Styrene	4.2	25
Bromoform	9.4	25
Isopropylbenzene	2.2	25
1,1,2,2-Tetrachloroethane	11	25
Bromobenzene	4.4	25
1,2,3-Trichloropropane	16	50
n-Propylbenzene	1.9	25
2-Chlorotoluene	2.8	25
1,3,5-Trimethylbenzene	3.3	25
4-Chlorotoluene	3.4	25
tert-Butylbenzene	3.0	25
1,2,4-Trimethylbenzene	3.1	25
sec-Butylbenzene	1.9	25
1,3-Dichlorobenzene	3.2	25
p-Isopropyltoluene	3.7	25
1,4-Dichlorobenzene	2.0	25
n-Butylbenzene	4.1	25
1,2-Dichlorobenzene	2.9	25
1,2-Dibromo-3-chloropropane	22	25
1,2,4-Trichlorobenzene	3.5	50
Hexachlorobutadiene	6.2	50
Naphthalene	13	250
1,2,3-Trichlorobenzene	5.0	50

Soils analyzed in GCIS-1637 and GCIS-1639, March 2007

TABLE 2

DEMONSTRATION OF CAPABILITY

WATER

Compound	Spike Amount (µg/L)	Mean Recovery	RSD (%)
Dichlorodifluoromethane	5	102	3.74
Chloromethane	5	99	2.44
Vinyl chloride	5	102	2.30
Bromomethane	5	165	13.8
Chloroethane	5	139	2.54
Trichlorofluoromethane	5	108	4.12
1,1,2-Trichlorotrifluoroethane	5	102	4.26
1,1-Dichloroethene	5	101	4.29
Acetone	50	92	8.46
Carbon disulfide	5	99	2.17
Methylene chloride	5	107	3.60
Methyl-tert-Butyl ether	5	91	4.47
trans-1,2-Dichloroethene	5	101	2.52
n-Hexane	5	100	1.40
1,1-Dichloroethane	5	104	2.01
Diisopropyl ether	5	96	4.72
2,2-Dichloropropane	5	90	3.19
cis-1,2-Dichloroethene	5	99	2.66
2-Butanone (MEK)	50	89	9.27
Tetrahydrofuran	25	84	7.25
Bromochloromethane	5	102	2.30
Chloroform	5	103	2.46
1,1,1-Trichloroethane	5	102	1.85
Carbon tetrachloride	5	104	3.78
1,1-Dichloropropene	5	101	2.14
Benzene	5	101	3.27
1,2-Dichloroethane	5	104	3.00
Trichloroethene	5	100	0.18
1,2-Dichloropropane	5	98	1.55
Dibromomethane	5	96	1.74
Bromodichloromethane	5	99	1.78
cis-1,3-Dichloropropene	5	92	2.77
Methyl isobutyl ketone (MIBK)	50	85	1.89
Toluene	5	97	1.15
trans-1,3-Dichloropropene	5	93	1.56

TABLE 2 (CONT.)

DEMONSTRATION OF CAPABILITY

WATER

Compound	Spike Amount (µg/L)	Mean Recovery	RSD (%)
1,1,2-Trichloroethane	5	97	4.04
Tetrachloroethene	5	103	2.08
1,3-Dichloropropane	5	97	3.45
2-Hexanone	50	84	5.02
Chlorodibromomethane	5	97	2.91
1,2-Dibromoethane	5	94	3.16
Chlorobenzene	5	101	0.90
1,1,1,2-Tetrachloroethane	5	100	0.81
Ethylbenzene	5	102	1.12
m+p-Xylenes	10	101	1.16
o-Xylene	5	98	0.37
Styrene	5	97	2.07
Bromoform	5	92	2.43
Isopropylbenzene	5	99	1.23
1,1,2,2-Tetrachloroethane	5	87	1.00
Bromobenzene	5	98	3.02
1,2,3-Trichloropropane	5	90	2.15
n-Propylbenzene	5	101	2.57
2-Chlorotoluene	5	101	2.00
1,3,5-Trimethylbenzene	5	99	1.81
4-Chlorotoluene	5	100	1.51
tert-Butylbenzene	5	98	3.61
1,2,4-Trimethylbenzene	5	100	2.13
sec-Butylbenzene	5	101	1.69
1,3-Dichlorobenzene	5	100	1.28
p-Isopropyltoluene	5	100	1.11
1,4-Dichlorobenzene	5	99	1.49
n-Butylbenzene	5	102	2.16
1,2-Dichlorobenzene	5	98	1.97
1,2-Dibromo-3-chloropropane	5	84	1.43
1,2,4-Trichlorobenzene	5	95	1.04
Hexachlorobutadiene	5	107	2.34
Naphthalene	5	78	3.96
1,2,3-Trichlorobenzene	5	94	2.21

Waters analyzed in GCIS-1626, March 2007

TABLE 2-1 (CONT.)

DEMONSTRATION OF CAPABILITY

SOIL

Compound	Spike Amount (µg/kg)	Mean Recovery	RSD (%)
Dichlorodifluoromethane	250	81	2.39
Chloromethane	250	95	2.73
Vinyl chloride	250	93	3.11
Bromomethane	250	99	8.16
Chloroethane	250	90	10.1
Trichlorofluoromethane	250	102	4.35
1,1,2-Trichlorotrifluoroethane	250	100	1.62
1,1-Dichloroethene	250	98	2.43
Acetone	2500	104	8.17
Carbon disulfide	250	96	2.32
Methylene Chloride	250	104	2.35
Methyl-tert-Butyl Ether	250	94	3.56
trans-1,2-Dichloroethene	250	99	2.79
n-Hexane	250	97	1.28
1,1-Dichloroethane	250	102	2.21
Diisopropyl Ether	250	95	1.02
2,2-Dichloropropane	250	97	2.96
cis-1,2-Dichloroethene	250	99	2.31
2-Butanone (MEK)	2500	99	7.45
Tetrahydrofuran	250	97	7.43
Bromochloromethane	250	101	1.67
Chloroform	250	101	3.12
1,1,1-Trichloroethane	250	101	1.41
Carbon Tetrachloride	250	105	2.93
1,1-Dichloropropene	250	102	2.06
Benzene	250	103	1.55
1,2-Dichloroethane	250	102	1.90
Trichloroethene	250	100	1.77
1,2-Dichloropropane	250	98	1.58
Dibromomethane	250	98	1.61
Bromodichloromethane	250	98	1.99
cis-1,3-Dichloropropene	250	97	2.62
Methyl Isobutyl Ketone	2500	98	4.91
Toluene	250	95	1.90
trans-1,3-Dichloropropene	250	97	2.41
1,1,2-Trichloroethane	250	98	2.96
Tetrachloroethene	250	101	1.41

TABLE 2-1 (CONT.)

DEMONSTRATION OF CAPABILITY

SOIL

Compound	Spike Amount (µg/kg)	Mean Recovery	RSD (%)
1,3-Dichloropropane	250	101	1.38
2-Hexanone	2500	96	6.67
Chlorodibromomethane	250	98	3.63
1,2-Dibromoethane	250	96	2.74
Chlorobenzene	250	99	0.41
1,1,1,2-Tetrachloroethane	250	98	1.77
Ethylbenzene	250	100	0.79
m+p-Xylenes	500	100	1.90
o-Xylene	250	97	1.25
Styrene	250	98	0.76
Bromoform	250	98	4.53
Isopropylbenzene	250	99	1.19
1,1,2,2-Tetrachloroethane	250	91	2.66
Bromobenzene	250	95	1.16
1,2,3-Trichloropropane	250	94	1.65
n-Propylbenzene	250	97	1.36
2-Chlorotoluene	250	97	2.02
1,3,5-Trimethylbenzene	250	96	2.55
4-Chlorotoluene	250	99	0.66
tert-Butylbenzene	250	95	1.91
1,2,4-Trimethylbenzene	250	96	1.87
sec-Butylbenzene	250	98	1.61
1,3-Dichlorobenzene	250	97	1.79
p-Isopropyltoluene	250	96	1.00
1,4-Dichlorobenzene	250	97	0.97
n-Butylbenzene	500	97	1.46
1,2-Dichlorobenzene	250	97	1.51
1,2-Dibromo-3-chloropropane	250	93	7.69
1,2,4-Trichlorobenzene	250	94	2.17
Hexachlorobutadiene	250	99	1.41
Naphthalene	250	86	2.96
1,2,3-Trichlorobenzene	250	92	2.30

Soils analyzed in GCIS-1639, March 2007

TABLE 3

ABSOLUTE RETENTION TIME ORDER AND C HARACTERISTIC IONS

Compound	Absolute RT (min)	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Dichlorodifluoromethane	1.33	85	87
Chloromethane	1.47	50	52
Vinyl chloride	1.55	62	64
Bromomethane	1.80	94	96
Chloroethane	1.90	64	66
Trichlorofluoromethane	2.11	151	101, 153
1,1,2-Trichloro-1,2,2-trifluoroethane	2.55	101	103, 151, 153
1,1-Dichloroethene	2.59	96	61, 63
Acetone	2.68	58	
Carbon disulfide	2.77	76	78
Methylene chloride	3.03	84	49, 86,
Methyl-tert-Butyl ether	3.19	73	57
trans-1,2-Dichloroethene	3.21	96	61, 98
n-Hexane	3.36	57	55, 86
1,1-Dichloroethane	3.58	63	65, 83
Diisopropyl ether	3.55	87	59, 69, 102
2,2-Dichloropropane	4.01	77	97
cis-1,2-Dichloroethene	4.03	96	61, 98
2-Butanone (MEK)	4.04	72	57
Tetrahydrofuran	4.22	72	71
Bromochloromethane	4.22	128	49, 130
Chloroform	4.25	83	85
Dibromofluoromethane (SURR)	4.38	113	
1,1,1-Trichloroethane	4.38	97	61, 99
Pentafluorobenzene (IS)	4.40	168	
Carbon tetrachloride	4.48	117	119
1,1-Dichloropropene	4.49	75	77, 110
Benzene	4.65	78	
1,2-Dichloroethane	4.72	62	98
1,4-Difluorobenzene (IS)	4.94	114	
Trichloroethene	5.12	95	97, 130, 132
1,2-Dichloropropane	5.33	63	112
Dibromomethane	5.42	93	95, 174
Bromodichloromethane	5.50	83	85, 127
cis-1,3-Dichloropropene	5.83	75	77
Methyl isobutyl ketone (MIBK)	5.92	58	85, 100
Toluene-d8 (SURR)	6.00	98	

TABLE 3 (CONT.)

ABSOLUTE RETENTION TIME ORDER AND CHARACTERISTIC IONS

Compound	Absolute RT (min)	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Toluene	6.05	92	91
trans-1,3-Dichloropropene	6.25	75	77
1,1,2-Trichloroethane	6.39	83	85, 97
Tetrachloroethene	6.43	164	129, 131, 166
1,3-Dichloropropane	6.52	76	78
2-Hexanone	6.53	58	85, 100
Chlorodibromomethane	6.67	129	127
1,2-Dibromoethane	6.78	107	109, 188
Chlorobenzene-d5 (IS)	7.09	117	
Chlorobenzene	7.11	112	77, 114
1,1,1,2-Tetrachloroethane	7.16	131	119, 133
Ethylbenzene	7.15	91	106
m+p-Xylenes	7.24	106	91
o-Xylene	7.55	106	91
Styrene	7.56	104	78
Bromoform	7.74	173	175, 254
Isopropylbenzene	7.80	105	120
4-Bromofluorobenzene (SURR)	7.96	95	174, 176
1,1,2,2-Tetrachloroethane	8.07	83	85, 131
Bromobenzene	8.09	156	77, 158
1,2,3-Trichloropropane	8.12	75	77
n-Propylbenzene	8.11	91	120
2-Chlorotoluene	8.22	91	126
1,3,5-Trimethylbenzene	8.24	105	120
4-Chlorotoluene	8.30	91	126
tert-Butylbenzene	8.48	119	91, 134
1,2,4-Trimethylbenzene	8.53	105	120
sec-Butylbenzene	8.65	105	134
1,3-Dichlorobenzene	8.79	146	111, 148
p-Isopropyltoluene	8.75	119	91, 134
1,4-Dichlorobenzene-d4 (IS)	8.85	152	115, 150
1,4-Dichlorobenzene	8.79	146	111, 148
n-Butylbenzene	9.07	91	92, 134
1,2-Dichlorobenzene	9.16	146	111, 148
1,2-Dibromo-3-chloropropane	9.77	75	155, 157
1,2,4-Trichlorobenzene	10.38	180	145, 182
Hexachlorobutadiene	10.44	225	223, 227
Naphthalene	10.58	128	
1,2,3-Trichlorobenzene	10.77	180	145, 182

TABLE 4

STOCK STANDARD SOLUTIONS

INITIAL CALIBRATION

8260B Liquids, Absolute Part #32001, 2000 µg/mL		
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1-Dichloroethane	1,1-Dichloroethene	1,1-Dichloropropene
1,2,3-Trichlorobenzen	1,2,3-Trichloropropane	1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane	1,2-Dibromoethane
1,2-Dichlorobenzene	1,2-Dichloroethane	1,2-Dichloropropane
1,3,5-Trimethylbenzene	1,3-Dichlorobenzene	1,3-Dichloropropane
1,4-Dichlorobenzene	2,2-Dichloropropane	2-Chlorotoluene
4-Chlorotoluene	Benzene	Bromobenzene
Bromochloromethane	Bromodichloromethane	Bromoform
cis-1,3-Dichloropropene	Carbon tetrachloride	Chloroform
cis-1,2-Dichloroethene	Chlorodibromomethane	Dibromomethane
Ethylbenzene	Hexachlorobutadiene	Isopropylbenzene
m+p-Xylenes	Methylene chloride	Methyl-tert-Butyl ether
Naphthalene	n-Butylbenzene	n-Propylbenzene
o-Xylene	p-Isopropyltoluene	sec-Butylbenzene
Styrene	trans-1,3-Dichloropropene	tert-Butylbenzene
Tetrachloroethene	Toluene	trans-1,2-Dichloroethene
Trichloroethene		

8260B Gases, Absolute Part # 30058, 2000 µg/mL		
Bromomethane	Chloroethane	Chloromethane
Dichlorodifluoromethane	Trichlorofluoromethane	Vinyl chloride

8260B Ketones, Absolute Part # 82492, 20,000 µg/mL		
Acetone	2-Butanone	2-Hexanone
4-Methyl-2-pentanone		

Single Component Solutions
Methyl-tert-Butyl ether, Absolute Part # 70209, 1000 µg/mL
Carbon disulfide, Absolute Part # 70060, 1000 µg/mL
Diisopropyl ether, Absolute Part # 70987, 1000 µg/mL
n-Hexane, Absolute Part # 70962, 1000 µg/mL
Tetrahydrofuran, Absolute Part # 34191, 20,000 µg/mL
1,1,2-Trichlorotrifluoroethane, Absolute Part # 70474, 1000 µg/mL
1,3,5-Trichlorobenzene, Absolute Part #70409, 1000ug/ml

TABLE 4-1

STOCK STANDARD SOLUTIONS

SECOND SOURCE

502.2 Mega Mix Standard, Restek Part #30431, 2000 µg/mL		
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1,2-Trichlorotrifluoroethane	1,1-Dichloroethane	1,1-Dichloroethene
1,1-Dichloropropene	1,2,3-Trichlorobenzene	1,2,3-Trichloropropane
1,2,4-Trichlorobenzene	1,2,4-Trimethylbenzene	1,2-Dibromo-3-chloropropane
1,2-Dibromoethane	1,2-Dichlorobenzene	1,2-Dichloroethane
1,2-Dichloropropane	1,3,5-Trimethylbenzene	1,3-Dichlorobenzene
1,3-Dichloropropane	1,4-Dichlorobenzene	2,2-Dichloropropane
2-Chlorotoluene	4-Chlorotoluene	Benzene
Bromobenzene	Bromochloromethane	Bromodichloromethane
Bromoform	cis-1,3-Dichloropropene	
Carbon tetrachloride	Chloroform	cis-1,2-Dichloroethene
Chlorodibromomethane	Dibromomethane	Ethylbenzene
Hexachlorobutadiene	Isopropylbenzene	m+p-Xylenes
Methylene chloride	1,1,1,2-tetrachloroethane	Naphthalene
n-Butylbenzene	n-Propylbenzene	o-Xylene
p-Isopropyltoluene	sec-Butylbenzene	Styrene
trans-1,3-Dichloropropene	tert-Butylbenzene	Tetrachloroethene
chlorobenzene	Toluene	trans-1,2-Dichloroethene
Trichloroethene		

502.2 Calibration Mix #1 (gases), Restek Part # 30042, 2000 µg/mL		
Bromomethane	Chloroethane	Chloromethane
Dichlorodifluoromethane	Trichlorofluoromethane	Vinyl chloride

VOA Calibration Mix #1 (ketones), Restek Part # 30006, 5000 µg/mL		
Acetone	2-Butanone	2-Hexanone
4-Methyl-2-pentanone		

California Oxygenates Mix, Restek Part # 30465, 2000 µg/mL	
Methyl tert-butyl ether	Diisopropyl ether

TABLE 4-2

STOCK STANDARD SOLUTIONS

INTERNAL STANDARDS AND SURROGATES

8260 Internal Standard Mix, Restek Part # 30074, 2500 µg/mL	
Chlorobenzene-d5	1,4-Dichlorobenzene-d4
1,4-Difluorobenzene	Pentafluorobenzene

8260 Surrogate Mix, Restek Part # 30073, 2500 µg/mL		
4-Bromofluorobenzene	Dibromofluoromethane	Toluene-d8

TABLE 5

INTERMEDIATE STANDARD SOLUTION PREPARATION

INITIAL CALIBRATION

Description	Absolute Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
54 Liquids	32001	2000	500	50	20
6 Gases	30058	2000	500	50	20
4 Ketones	82492	20000	500	50	200
MTBE	70209	1000	1000	50	20
Carbon disulfide	70060	1000	1000	50	20
Diisopropyl ether	70987	1000	1000	50	20
Hexane	70962	1000	1000	50	20
Tetrahydrofuran	74191	20000	250	50	100
1,1,2-Trichlorotrifluoroethane	70474	1000	500	50	20

NOTES: (1) Solvent Purge and trap methanol.
(2) Compounds may be added or deleted for project/site specific requirements.

TABLE 5-1

INTERMEDIATE STANDARD SOLUTION PREPARATION

SECOND SOURCE

Description	Restek Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
Mega Mix	30633	2000	250	25	20
6 Gases	30042	2000	250	25	20
4 Ketones	30006	5000	1000	25	200
Oxygenates	30465	2000	250	25	20

TABLE 5-2

INTERMEDIATE STANDARD SOLUTION PREPARATION

INTERNAL STANDARDS AND SURROGATES

Description	Restek Part #	Stock Conc. (µg/mL)	Volume Added (µL)	Final Volume (mL)	Final Conc. (µg/mL)
8260 Internal Standards	30074	2500	500	50	25
8260 Surrogate Standards	30073	2500	500	50	25

TABLE 6

INITIAL CALIBRATION STANDARDS

PREPARATION IN 10 ML OF REAGENT WATER

Calibration Level	**L-1**	**L-2**	**L-3**	**L-4**	**L-5**	**L-6**	**L-7**
µg/L Final in 10 mL	0.5/2.5/5.0	1.0/5.0/10.0	2.0/10.0/20	5/25/50	10/50/100	25/125/2500	50/250/5000
Volume of Makeup Methanol (µL) (optional for soils)	200	200	199	197	1965	188	175
Volume of IS/Surr. Solution (µL)(5.12.2)	10	10	10	10	10	10	10
Volume of Standard Solution (µL) (7.5.1)	0.25	0.50	1.0	2.5	5.0	12.5	25

NOTE: Calculation Formula: µg/L

$$\frac{A \times B}{V \times 1000 ng/ug}$$

Where:
A = Volume Standard Solution in µL added to 10 mL
B = Concentration of analyte in Standard Solution in µg/mL
V = Volume in syringe (usually 10 mL) in L (0.01 L)

TABLE 7

INTERNAL STANDARDS AND CORRESPONDING TARGET COMPOUNDS

Pentafluorobenzene		
Dichlorodifluoromethane	Chloromethane	Vinyl chloride
Bromomethane	Chloroethane	Trichlorofluoromethane
1,1,2-Trichlorotrifluoroethane	1,1-Dichloroethene	Acetone
Carbon disulfide	Methylene chloride	Methyl-tert-butyl ether
trans-1,2-Dichloroethene	n-Hexane	1,1-Dichloroethane
Diisopropyl Ether	2,2-Dichloropropane	cis-1,2-Dichloroethene
2-Butanone (MEK)	Tetrahydrofuran	Bromochloromethane
Chloroform	Dibromofluoromethane (SURR)	1,1,1-Trichloroethane
Carbon tetrachloride	1,1-Dichloropropene	Benzene
1,2-Dichloroethane		

1,4-Difluorobenzene		
Trichloroethene	1,2-Dichloropropane	Dibromomethane
Bromodichloromethane	cis-1,3-Dichloropropene	Methyl isobutyl ketone (MIBK)
Toluene-d8 (SURR)	Toluene	trans-1,3-Dichloropropene
1,1,2-Trichloroethane		

Chlorobenzene-d5		
Tetrachloroethene	1,3-Dichloropropane	2-Hexanone
Chlorodibromomethane	1,2-Dibromoethane	Ethylbenzene
m+p-Xylenes	o-Xylene	Styrene
Bromoform	Isopropylbenzene	Bromofluorobenzene (SURR)

1,4-Dichlorobenzene-d4		
1,1,2,2-Tetrachloroethane	Bromobenzene	1,2,3-Trichloropropane
n-Propylbenzene	2-Chlorotoluene	1,3,5-Trimethylbenzene
4-Chlorotoluene	tert-Butylbenzene	1,2,4-Trimethylbenzene
sec-Butylbenzene	1,3-Dichlorobenzene	p-Isopropyltoluene
1,4-Dichlorobenzene	n-Butylbenzene	1,2-Dichlorobenzene
1,2-Dibromo-3-chloropropane	1,2,4-Trichlorobenzene	Hexachlorobutadiene
Naphthalene	1,2,3-Trichlorobenzene	

TABLE 8

CONDITIONS FOR THE PURGE AND TRAP "CLEAN CYCLE"

Step 1, No Purge Tube Attached	
Purge and Trap:	Method "2"
Purge Flow:	Helium, 35 mL/min.
Purge Time:	1.5 minutes
Dry Purge:	0 minutes
Desorb:	0.2 minutes at 0 °C
Bake Time:	0 minutes at 0 °C

GC/MS Conditions: Method and Sequence "Purge"	
Oven Temperature Program	
Initial Temperature:	45 °C
Initial Hold:	0.5 minutes
EM Voltage:	1800

Step 2, Purge Tube Attached	
Purge and Trap:	Method "3"
Purge Flow:	Helium, 35 mL/min.
Purge Time:	5.0 minutes
Dry Purge:	0 minutes
Desorb:	0.2 minutes at 0 °C
Bake Time:	0 minutes at 0 °C

GC/MS Conditions: Method and Sequence "Purge"	
Oven Temperature Program	
Initial Temperature:	45 °C
Initial Hold:	0.5 minutes
EM Voltage:	1800

TABLE 9

BFB MASS INTENSITY CRITERIA

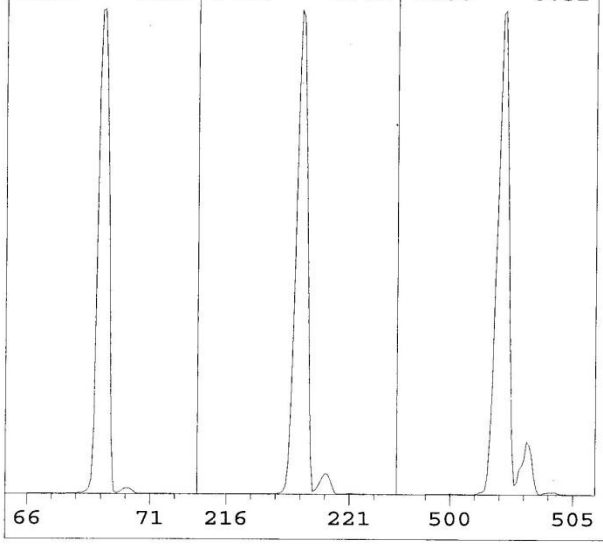
m/z	Required Intensity (Relative Abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base Peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5-9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

FIGURE 1

MAXIMUM SENSITIVITY AUTOTUNE REPORT

HP5971 Maximum Sensitivity Autotune
Instrument: GC/MS Instrument #1
Tue Feb 12 12:26:47 2008 D:\HPCHEM\1\5971\ATUNE.U

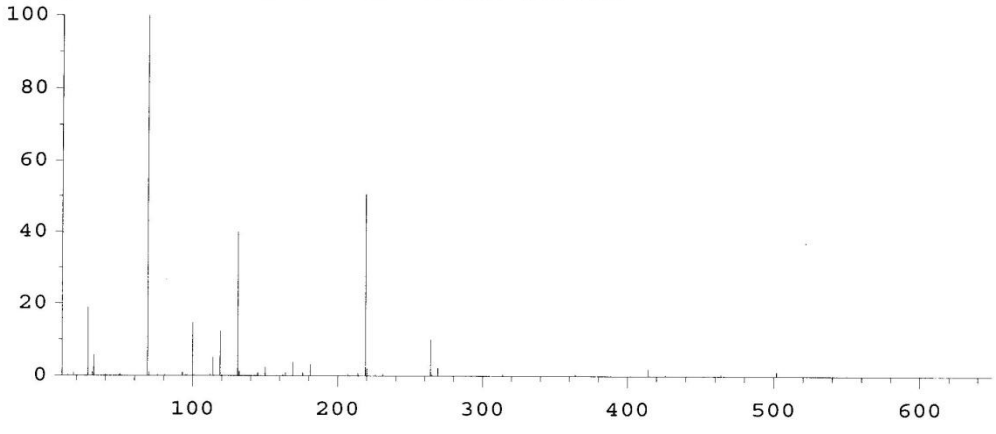
Mass	69.10	Mass	219.00	Mass	502.20
Ab	2108606	Ab	1099103	Ab	28982
Pw50	0.52	Pw50	0.51	Pw50	0.52



EMVolts	1553	AmuGain	597
Xray	8.6	AmuOffs	84
Emission	ON	Wid219	0.026
MS Temp	150	TTI	OFF
Vacuum	38	DC Pol	NEG
Samples	16	Repeller	19.64
Averages	1	IonFocus	36.0
StepSize	0.10	EntLens	4.52
MassGain	185	EntOffs	14.81
MassOffs	2	Filament	2

PFTBA OPEN

Scan: 10.00 - 650.00 Samples: 16 Thresh: 150
227 peaks Base: 69.00 Abundance: 1781248



Mass	Abund	Rel Abund	Iso Mass	Iso Abund	Iso Ratio
69.00	1781248	100.00	70.00	19560	1.10
218.95	904128	50.76	219.95	39224	4.34
502.05	23968	1.35	503.05	2416	10.08

FIGURE 2

TOTAL ION CHROMATOGRAM OF A CCV AND SUMMARY REPORT

Quantitation Report (Not Reviewed)

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D
Acq On : 12 Feb 2008 2:50 pm
Sample : CCS, 5UG/L
Misc :
MS Integration Params: RTEINT.P
Quant Time: Feb 13 13:29 2008

Vial: 4
Operator: cps
Inst : GC/MS Ins
Multiplr: 1.00
Quant Results File: VOCA0212.RES

Quant Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration
DataAcq Meth : 8260

CPS 2/13/08

Internal Standards	R.T.	QIon	Response	Conc	Units		Dev(Min)
1) Pentafluorobenzene	4.53	168	1646282	25.00	ug/l		0.00
30) 1,4-Difluorobenzene	5.08	114	2428392	25.00	ug/l		0.00
40) Chlorobenzene-D5	7.24	117	2190468	25.00	ug/l		0.00
56) 1,4-Dichlorobenzene-D4	8.99	152	1125532	25.00	ug/l		0.00
System Monitoring Compounds							
24) Dibromofluoromethane	4.52	113	738831	22.01	ug/l		0.00
Spiked Amount 25.000			Recovery =	88.04%			
37) Toluene-D8	6.14	98	2578616	25.03	ug/l		0.00
Spiked Amount 25.000			Recovery =	100.12%			
55) Bromofluorobenzene	8.11	95	881882	24.04	ug/l		0.00
Spiked Amount 25.000			Recovery =	96.16%			
Target Compounds							Qvalue
2) Dichlorodifluoromethane	1.42	85	133423	4.94	ug/l		99
3) Chloromethane	1.58	50	90602	4.93	ug/l		89
4) Vinyl Chloride	1.66	62	77674	4.60	ug/l		97
5) Bromomethane	1.91	96	9209	3.82	ug/l	#	13
6) Chloroethane	1.99	64	55564	6.36	ug/l		95
7) Trichlorofluoromethane	2.21	101	117100	3.32	ug/l	#	93
8) 1,1,2-Trichlorotrifluoroet	2.66	101	143241	4.79	ug/L		96
9) 1,1-Dichloroethene	2.70	96	135926	4.99	ug/l		93
10) Acetone	2.80	58	44047	46.14	ug/L		100
11) Carbon Disulfide	2.89	76	461673	4.85	ug/L		99
12) Methylene Chloride	3.16	84	129724	4.90	ug/l		97
13) Methyl-t-butyl Ether	3.31	73	151105	4.10	ug/L		97
14) t-1,2-Dichloroethene	3.34	96	137303	4.49	ug/l		92
15) n-Hexane	3.48	57	169714	5.19	ug/L		100
16) 1,1-Dichloroethane	3.72	63	197595	4.66	ug/l		99
17) Diisopropyl Ether	3.67	87	88123	4.97	ug/L		96
18) 2.2- Dichloropropane	4.14	77	88433	3.14	ug/l		96
19) c-1,2-Dichloroethene	4.16	96	155263	4.58	ug/l		97
20) 2-Butanone(MEK)	4.18	72	64360	45.48	ug/l		99
21) Tetrahydrofuran	4.35	72	36739	23.98	ug/L		99
22) Bromochloromethane	4.36	128	73342	4.21	ug/l		97
23) Chloroform	4.39	83	207405	4.63	ug/l		97
25) 1,1,1-Trichloroethane	4.52	97	165855	4.43	ug/l		99
26) Carbon tetrachloride	4.60	117	150110	4.49	ug/l		95
27) 1.1-Dichloropropene	4.63	75	175512	4.87	ug/l		99
28) Benzene	4.79	78	522438	4.83	ug/l		99
29) 1,2-Dichloroethane	4.86	62	89431	4.54	ug/l		95
31) Trichloroethene	5.25	95	149765	4.76	ug/l		99

(#) = qualifier out of range (m) = manual integration
004A.D VOCA0212.M Wed Feb 13 13:29:44 2008 Page 1

FIGURE 2 (CONT.)

TOTAL ION CHROMATOGRAM OF A CCV AND SUMMARY REPORT

Quantitation Report (Not Reviewed)

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Feb 13 13:29 2008 Quant Results File: VOCA0212.RES

Quant Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration
DataAcq Meth : 8260

	Compound	R.T.	QIon	Response	Conc	Unit		Qvalue
32)	1,2-Dichloropropane	5.47	63	122527	4.95	ug/l		96
33)	Dibromomethane	5.56	93	76899	4.57	ug/l		96
34)	Bromodichloromethane	5.65	83	152464	4.72	ug/l		100
35)	c-1,3-Dichloropropene	5.97	75	181341	4.86	ug/l		99
36)	Methyl Isobutyl Ketone (MI	6.07	58	235719	51.79	ug/l		98
38)	Toluene	6.19	92	387541	5.13	ug/l		96
39)	1,1,2-Trichloroethane	6.53	83	83438	4.72	ug/l		98
41)	t-1,3-Dichloropropene	6.39	75	127746	4.69	ug/l	#	89
42)	Tetrachloroethene	6.57	164	155189	5.04	ug/l		96
43)	1,3-Dichloropropane	6.67	76	160881	4.92	ug/l		96
44)	2-Hexanone	6.67	58	222153	53.74	ug/l	#	95
45)	Dibromochloromethane	6.82	129	127664	4.65	ug/l		96
46)	1,2-Dibromoethane	6.93	107	110160	4.78	ug/l	#	98
47)	Chlorobenzene	7.26	112	439888	5.17	ug/l		98
48)	1,1,1,2-Tetrachloroethane	7.32	131	141899	4.92	ug/l		97
49)	Ethylbenzene	7.30	91	682203	5.14	ug/l		98
50)	m+p-Xylene	7.38	106	571758	10.35	ug/l		96
51)	o-Xylene	7.70	106	275747	5.18	ug/l		99
52)	Styrene	7.71	104	449245	5.19	ug/l		99
53)	Bromoform	7.90	173	83417	4.66	ug/l	#	98
54)	Isopropylbenzene	7.94	105	611509	5.28	ug/l		100
57)	1,1,2,2-Tetrachloroethane	8.22	83	105229	4.98	ug/l		94
58)	Bromobenzene	8.25	156	189176	5.01	ug/l		98
59)	1,2,3-Trichloropropane	8.28	75	82167	4.94	ug/l		86
60)	n-Propyl benzene	8.26	91	811071	5.32	ug/l		99
61)	2-Chlorotoluene	8.37	91	501878	5.13	ug/l		97
62)	1,3,5-Trimethylbenzene	8.39	105	524641	5.18	ug/l		97
63)	4-Chlorotoluene	8.45	91	450035	5.19	ug/l		100
64)	t-Butyl benzene	8.63	119	516364	5.46	ug/l		95
65)	1,2,4-Trimethylbenzene	8.68	105	535928	5.19	ug/l		99
66)	sec-Butyl benzene	8.80	105	792268	5.33	ug/l		98
67)	1,3-Dichlorobenzene	8.95	146	359797	5.11	ug/l		99
68)	p-Isopropyl toluene	8.90	119	643468	5.31	ug/l		99
69)	1,4-Dichlorobenzene	9.02	146	347429	5.05	ug/l		96
70)	n-Butyl benzene	9.21	91	603802	5.33	ug/l		98
71)	1,2-Dichlorobenzene	9.31	146	307081	5.14	ug/l		99
72)	1,2-Dibromo-3-chloropropan	9.92	157	20317	4.76	ug/l		98
73)	1,2,4-Trichlorobenzene	10.52	180	231459	5.23	ug/l		96
74)	Hexachlorobutadiene	10.59	225	137900	5.45	ug/l		95
75)	Naphthalene	10.74	128	313529	4.72	ug/l	#	97
76)	1,2,3-Trichlorobenzene	10.93	180	180977	4.83	ug/l		98

(#) = qualifier out of range (m) = manual integration
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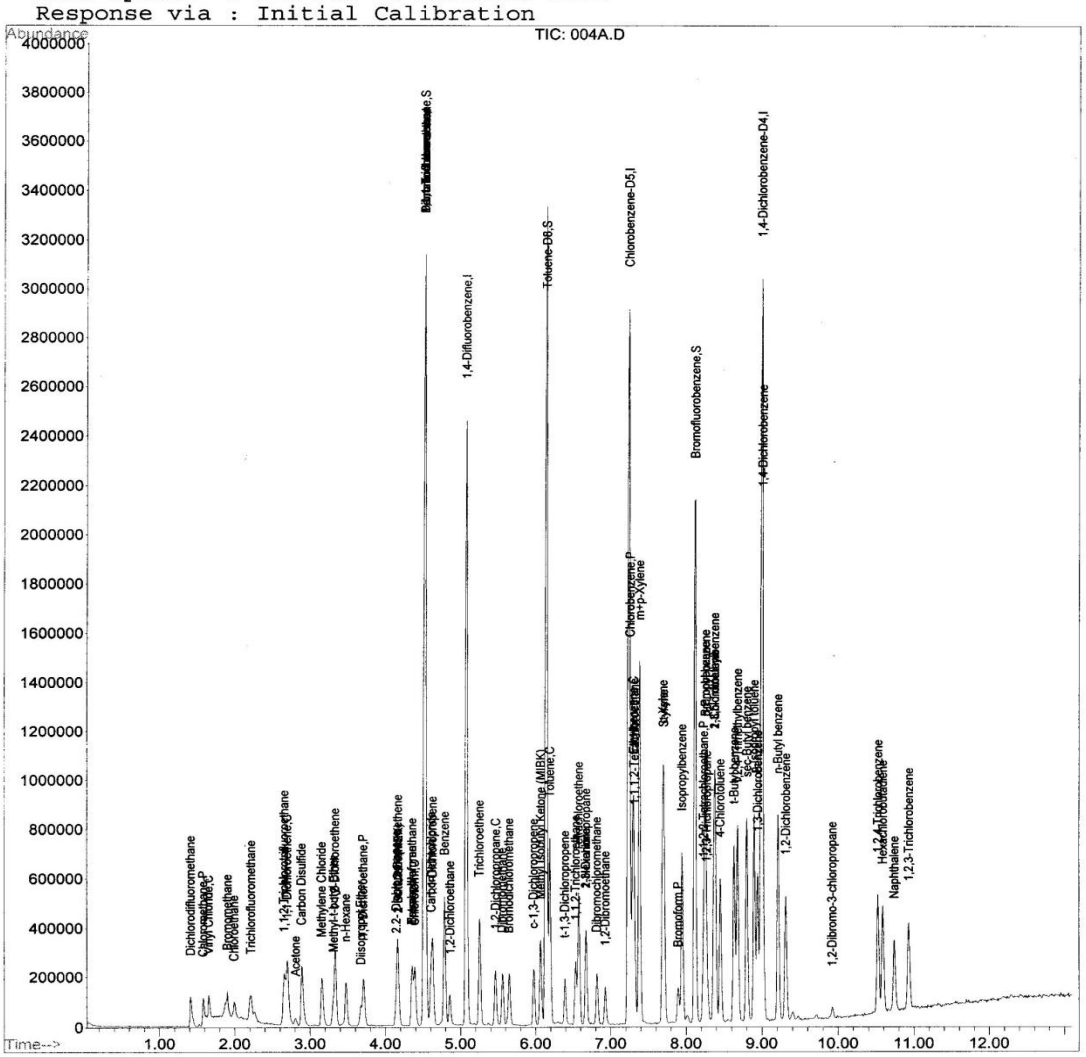
FIGURE 2 (CONT.)

TOTAL ION CHROMATOGRAM OF A CCV AND SUMMARY REPORT

Quantitation Report

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P
Quant Time: Feb 13 13:29 2008 Quant Results File: VOCA0212.RES

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration



004A.D VOCA0212.M Wed Feb 13 13:29:45 2008 Page 3

FIGURE 3

DATA SYSTEM SUMMARY OF % DIFFERENCE AND RESPONSE FACTOR FOR CONTINUING CALIBRATION VERIFICATION

Evaluate Continuing Calibration Report

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Multiple Level Calibration

pass cps 2/13/08

Min. RRF : 0.000 Min. Rel. Area : 50% Max. R.T. Dev 0.06min
Max. RRF Dev : 20% Max. Rel. Area : 150%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
1	I	Pentafluorobenzene	1.000	1.000	0.0	113	0.00
2		Dichlorodifluoromethane	0.410	0.405	1.2	112	0.00
3	P	Chloromethane	0.279	0.275	1.4	111	0.02
4	C	Vinyl Chloride	0.257	0.236	8.2	113	0.00
5		Bromomethane	0.037	0.028	24.3#	102	0.00
6		Chloroethane	0.138	0.169	-22.5#	120	0.01
7		Trichlorofluoromethane	0.536	0.356	33.6#	76	0.00
8		1,1,2-Trichlorotrifluoroeth	0.454	0.435	4.2	111	0.00
9	C	1,1-Dichloroethene	0.413	0.413	0.0	115	0.00
10		Acetone	0.015	0.013	13.3	112	0.00
11		Carbon Disulfide	1.445	1.402	3.0	112	0.00
12		Methylene Chloride	0.406	0.394	3.0	112	0.00
13		Methyl-t-butyl Ether	0.560	0.459	18.0	92	0.00
14		t-1,2-Dichloroethene	0.464	0.417	10.1	101	0.00
15		n-Hexane	0.497	0.515	-3.6	120	0.02
16	P	1,1-Dichloroethane	0.644	0.600	6.8	107	0.02
17		Diisopropyl Ether	0.269	0.268	0.4	114	0.00
18		2.2- Dichloropropane	0.427	0.269	37.0#	71	0.00
19		c-1,2-Dichloroethene	0.514	0.472	8.2	102	0.00
20		2-Butanone(MEK)	0.022	0.020	9.1	103	0.00
21		Tetrahydrofuran	0.023	0.022	4.3	108	0.00
22		Bromochloromethane	0.265	0.223	15.8	98	0.00
23	C	Chloroform	0.681	0.630	7.5	106	0.00
24	S	Dibromofluoromethane	0.510	0.449	12.0	101	0.00
25		1,1,1-Trichloroethane	0.568	0.504	11.3	98	0.02
26		Carbon tetrachloride	0.507	0.456	10.1	102	0.00
27		1.1-Dichloropropene	0.547	0.533	2.6	110	0.00
28		Benzene	1.644	1.587	3.5	108	0.00
29		1,2-Dichloroethane	0.299	0.272	9.0	99	0.02
30	I	1,4-Difluorobenzene	1.000	1.000	0.0	111	0.00
31		Trichloroethene	0.324	0.308	4.9	107	0.00
32	C	1,2-Dichloropropane	0.255	0.252	1.2	113	0.00
33		Dibromomethane	0.173	0.158	8.7	100	0.00
34		Bromodichloromethane	0.333	0.314	5.7	104	0.00
35		c-1,3-Dichloropropene	0.384	0.373	2.9	110	0.00
36		Methyl Isobutyl Ketone (MIB	0.046	0.049	-6.5	119	0.00
37	S	Toluene-D8	1.060	1.062	-0.2	112	0.00
38	C	Toluene	0.805	0.798	0.9	114	0.00
39		1,1,2-Trichloroethane	0.182	0.172	5.5	107	0.00

(#) = Out of Range
004A.D VOCA0212.M Thu Feb 14 13:00:30 2008 Page 1

FIGURE 3 (CONT.)

DATA SYSTEM SUMMARY OF % DIFFERENCE AND RESPONSE FACTOR FOR CONTINUING CALIBRATION VERIFICATION

Evaluate Continuing Calibration Report

Data File : D:\HPCHEM\1\DATA\GC-1926\004A.D Vial: 4
Acq On : 12 Feb 2008 2:50 pm Operator: cps
Sample : CCS, 5UG/L Inst : GC/MS Ins
Misc : Multiplr: 1.00
MS Integration Params: RTEINT.P

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Multiple Level Calibration

Min. RRF : 0.000 Min. Rel. Area : 50% Max. R.T. Dev 0.06min
Max. RRF Dev : 20% Max. Rel. Area : 150%

		Compound	AvgRF	CCRF	%Dev	Area%	Dev(min)
41		t-1,3-Dichloropropene	0.311	0.292	6.1	99	0.00
42		Tetrachloroethene	0.351	0.354	-0.9	109	0.00
43		1,3-Dichloropropane	0.373	0.367	1.6	107	0.00
44		2-Hexanone	0.046	0.051	-10.9	122	0.00
45		Dibromochloromethane	0.313	0.291	7.0	99	0.00
46		1,2-Dibromoethane	0.263	0.251	4.6	108	0.00
47	P	Chlorobenzene	0.971	1.004	-3.4	114	0.00
48		1,1,1,2-Tetrachloroethane	0.329	0.324	1.5	113	0.00
49	C	Ethylbenzene	1.514	1.557	-2.8	114	0.00
50		m+p-Xylene	0.631	0.653	-3.5	112	0.00
51		o-Xylene	0.607	0.629	-3.6	114	0.00
52		Styrene	0.988	1.025	-3.7	115	0.00
53	P	Bromoform	0.205	0.190	7.3	100	0.00
54		Isopropylbenzene	1.323	1.396	-5.5	117	0.00
55	S	Bromofluorobenzene	0.419	0.403	3.8	108	0.00
56	I	1,4-Dichlorobenzene-D4	1.000	1.000	0.0	110	0.00
57	P	1,1,2,2-Tetrachloroethane	0.469	0.467	0.4	111	0.00
58		Bromobenzene	0.838	0.840	-0.2	114	0.00
59		1,2,3-Trichloropropane	0.370	0.365	1.4	131	0.00
60		n-Propyl benzene	3.387	3.603	-6.4	114	0.00
61		2-Chlorotoluene	2.174	2.230	-2.6	113	0.00
62		1,3,5-Trimethylbenzene	2.248	2.331	-3.7	112	0.00
63		4-Chlorotoluene	1.924	1.999	-3.9	115	0.00
64		t-Butyl benzene	2.102	2.294	-9.1	116	0.00
65		1,2,4-Trimethylbenzene	2.295	2.381	-3.7	115	0.00
66		sec-Butyl benzene	3.301	3.520	-6.6	119	0.00
67		1,3-Dichlorobenzene	1.563	1.598	-2.2	115	0.00
68		p-Isopropyl toluene	2.694	2.859	-6.1	114	0.00
69		1,4-Dichlorobenzene	1.528	1.543	-1.0	109	0.00
70		n-Butyl benzene	2.515	2.682	-6.6	119	0.00
71		1,2-Dichlorobenzene	1.326	1.364	-2.9	113	0.00
72		1,2-Dibromo-3-chloropropane	0.095	0.090	5.3	108	0.00
73		1,2,4-Trichlorobenzene	0.984	1.028	-4.5	117	0.00
74		Hexachlorobutadiene	0.563	0.613	-8.9	122	0.00
75		Naphthalene	1.475	1.393	5.6	109	0.00
76		1,2,3-Trichlorobenzene	0.832	0.804	3.4	109	0.00

(#) = Out of Range SPCC's out = 0 CCC's out = 0
004A.D VOCA0212.M Thu Feb 14 13:00:31 2008 Page 2

FIGURE 4

CALCULATION PAGE FOR AVERAGE RESPONSE FACTORS AND STATISTICS

Response Factor Report GC/MS Ins

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration

Pass CPS 2/13/08

Calibration Files
1 =012A.D 2 =013A.D 3 =014A.D
4 =015A.D 5 =016A.D 6 =017A.D

		Compound	1	2	3	4	5	6	Avg	%RSD
1)	I	Pentafluorobenzene	----------ISTD----------							
2)		Dichlorodifluoromet	0.403	0.402	0.407	0.411	0.425	0.413	0.410	1.87
3)	P	Chloromethane	0.277	0.286	0.279	0.282	0.284	0.274	0.279	2.17
4)	C	Vinyl Chloride	0.262	0.268	0.273	0.236	0.263	0.252	0.257	5.25
5)		Bromomethane	0.034	0.037	0.047	0.031	0.040	0.035	0.037	14.37
6)		Chloroethane	0.106	0.167		0.159		0.128	0.138	18.11
7)		Trichlorofluorometh	0.581	0.493	0.572	0.529	0.538	0.534	0.536	5.90
8)		1,1,2-Trichlorotrif	0.486	0.470	0.477	0.443	0.437	0.436	0.454	5.01
9)	C	1,1-Dichloroethene	0.421	0.441	0.423	0.408	0.417	0.385	0.413	4.38
10)		Acetone	0.022	0.014	0.015	0.013	0.014	0.014	0.015	19.65
11)		Carbon Disulfide	1.602	1.491	1.460	1.418	1.427	1.371	1.445	5.94
12)		Methylene Chloride	0.443	0.402	0.389	0.397	0.417	0.399	0.406	4.53
13)		Methyl-t-butyl Ethe	0.555	0.594	0.542	0.568	0.551	0.546	0.560	3.16
14)		t-1,2-Dichloroethen	0.484	0.476	0.460	0.469	0.459	0.454	0.464	2.60
15)		n-Hexane	0.562	0.472	0.482	0.485	0.495	0.482	0.497	6.05
16)	P	1,1-Dichloroethane	0.684	0.640	0.676	0.633	0.641	0.614	0.644	4.13
17)		Diisopropyl Ether	0.277	0.269	0.285	0.266	0.260	0.257	0.269	3.51
18)		2.2- Dichloropropan	0.465	0.450	0.432	0.426	0.407	0.405	0.427	5.53
19)		c-1,2-Dichloroethen	0.525	0.520	0.519	0.522	0.514	0.498	0.514	2.05
20)		2-Butanone(MEK)	0.025	0.025	0.022	0.022	0.021	0.020	0.022	8.39
21)		Tetrahydrofuran	0.021	0.026	0.022	0.023	0.023	0.023	0.023	6.07
22)		Bromochloromethane	0.244	0.287	0.285	0.259	0.260	0.259	0.265	5.91
23)	C	Chloroform	0.686	0.692	0.702	0.677	0.693	0.659	0.681	2.55
24)	S	Dibromofluoromethan	0.508	0.516	0.511	0.506	0.504	0.505	0.510	1.07
25)		1,1,1-Trichloroetha	0.633	0.533	0.569	0.583	0.560	0.548	0.568	5.80
26)		Carbon tetrachlorid	0.521	0.502	0.509	0.509	0.510	0.496	0.507	1.57
27)		1.1-Dichloropropene	0.579	0.536	0.558	0.552	0.539	0.532	0.547	3.09
28)		Benzene	1.726	1.695	1.617	1.671	1.663	1.585	1.644	3.80
29)		1,2-Dichloroethane	0.279	0.298	0.306	0.312	0.307	0.293	0.299	3.68
30)	I	1,4-Difluorobenzene	----------ISTD----------							
31)		Trichloroethene	0.345	0.326	0.338	0.320	0.321	0.305	0.324	4.15
32)	C	1,2-Dichloropropane	0.272	0.255	0.260	0.248	0.248	0.248	0.255	3.43
33)		Dibromomethane	0.187	0.174	0.174	0.176	0.168	0.167	0.173	4.42
34)		Bromodichloromethan	0.338	0.336	0.339	0.336	0.334	0.319	0.333	2.24
35)		c-1,3-Dichloroprope	0.409	0.398	0.367	0.380	0.371	0.386	0.384	3.81
36)		Methyl Isobutyl Ket	0.043	0.045	0.045	0.045	0.047	0.049	0.046	4.09
37)	S	Toluene-D8	1.050	1.060	1.071	1.054	1.062	1.064	1.060	0.64
38)	C	Toluene	0.967	0.813	0.827	0.778	0.773	0.741	0.805	9.85
39)		1,1,2-Trichloroetha	0.176	0.194	0.184	0.180	0.176	0.182	0.182	3.38
40)	I	Chlorobenzene-D5	----------ISTD----------							
41)		t-1,3-Dichloroprope	0.331	0.283	0.301	0.327	0.308	0.312	0.311	5.20

(#) = Out of Range ### Number of calibration levels exceeded format ###
VOCA0212.M Wed Feb 13 13:17:30 2008 Page 1

FIGURE 4 (CONT.)

CALCULATION PAGE FOR AVERAGE RESPONSE FACTORS AND STATISTICS

Response Factor Report GC/MS Ins

Method : D:\HPCHEM\1\METHODS\VOCA0212.M (RTE Integrator)
Title : ECCS 8260 LIST
Last Update : Wed Feb 13 13:09:09 2008
Response via : Initial Calibration

Calibration Files
1 =012A.D 2 =013A.D 3 =014A.D
4 =015A.D 5 =016A.D 6 =017A.D

		Compound	1	2	3	4	5	6	Avg	%RSD
42)		Tetrachloroethene	0.374	0.369	0.336	0.361	0.343	0.341	0.351	4.68
43)		1,3-Dichloropropane	0.362	0.401	0.368	0.383	0.372	0.368	0.373	3.91
44)		2-Hexanone	0.045	0.043	0.045	0.046	0.047	0.050	0.046	4.84
45)		Dibromochloromethan	0.306	0.321	0.307	0.326	0.307	0.311	0.313	2.46
46)		1,2-Dibromoethane	0.272	0.261	0.261	0.259	0.268	0.258	0.263	1.95
47)	P	Chlorobenzene	0.999	1.017	0.965	0.983	0.980	0.943	0.971	3.74
48)		1,1,1,2-Tetrachloro	0.367	0.335	0.323	0.319	0.333	0.314	0.329	5.85
49)	C	Ethylbenzene	1.643	1.528	1.535	1.523	1.528	1.469	1.514	5.32
50)		m+p-Xylene	0.707	0.633	0.642	0.646	0.614	0.605	0.631	6.78
51)		o-Xylene	0.634	0.612	0.608	0.613	0.604	0.602	0.607	2.71
52)		Styrene	1.006	0.974	0.972	0.993	1.022	0.992	0.988	2.28
53)	P	Bromoform	0.221	0.190	0.203	0.211	0.203	0.200	0.205	4.72
54)		Isopropylbenzene	1.394	1.338	1.310	1.327	1.329	1.313	1.323	3.29
55)	S	Bromofluorobenzene	0.406	0.420	0.413	0.416	0.437	0.426	0.419	2.41
56)	I	1,4-Dichlorobenzene-D	----------	ISTD	----------					
57)	P	1,1,2,2-Tetrachloro	0.504	0.487	0.481	0.465	0.424	0.466	0.469	5.40
58)		Bromobenzene	0.988	0.820	0.826	0.814	0.820	0.796	0.838	8.03
59)		1,2,3-Trichloroprop	0.423	0.393	0.384	0.308	0.359	0.361	0.370	9.72
60)		n-Propyl benzene	3.616	3.439	3.531	3.480	3.311	3.248	3.387	5.42
61)		2-Chlorotoluene	2.402	2.231	2.192	2.167	2.105	2.086	2.174	5.55
62)		1,3,5-Trimethylbenz	2.375	2.284	2.345	2.292	2.183	2.143	2.248	4.50
63)		4-Chlorotoluene	1.995	2.014	1.929	1.918	1.887	1.863	1.924	3.15
64)		t-Butyl benzene	2.162	2.091	2.092	2.184	2.042	2.068	2.102	2.45
65)		1,2,4-Trimethylbenz	2.521	2.289	2.397	2.279	2.197	2.209	2.295	5.47
66)		sec-Butyl benzene	3.563	3.347	3.407	3.254	3.219	3.192	3.301	4.51
67)		1,3-Dichlorobenzene	1.756	1.639	1.591	1.527	1.490	1.481	1.563	6.85
68)		p-Isopropyl toluene	2.729	2.656	2.756	2.776	2.679	2.668	2.694	2.38
69)		1,4-Dichlorobenzene	1.674	1.567	1.555	1.564	1.464	1.445	1.528	5.71
70)		n-Butyl benzene	2.564	2.451	2.722	2.485	2.460	2.480	2.515	3.97
71)		1,2-Dichlorobenzene	1.380	1.380	1.363	1.333	1.296	1.258	1.326	3.88
72)		1,2-Dibromo-3-chlor	0.077	0.107	0.095	0.092	0.098	0.097	0.095	9.71
73)		1,2,4-Trichlorobenz	1.069	1.019	1.035	0.966	0.937	0.921	0.984	5.84
74)		Hexachlorobutadiene	0.580	0.597	0.549	0.555	0.560	0.547	0.563	3.32
75)		Naphthalene	1.630	1.562	1.443	1.412	1.357	1.441	1.475	6.29
76)		1,2,3-Trichlorobenz	0.898	0.833	0.881	0.814	0.785	0.799	0.832	5.07

(#) = Out of Range ### Number of calibration levels exceeded format ###
VOCA0212.M Wed Feb 13 13:17:31 2008 Page 2

FIGURE 5

Log Page Number_____

VOC SOIL WEIGHING LOG

Preparation Date:_____ Received Date:_____

Prepared By:_____ Repipettor Checked: Received By:_____

Balance ID:_____ Yes_____ Balance ID:_____

ECCS Sample Number	Sample ID	Initial Jar Wt. (g)	Final Jar Wt. (g)	Initial MEOH (ml)	Wt. Soil (g)	Final MEOH (ml)

	Manufacturer	Lot Number	Density @ 20°C
Methanol, P&T Grade			0.791 g/ml

Comments:_____

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

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QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures

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Any printed documents in use within a Pace Analytical Services, Inc. laboratory have been reviewed and approved by the persons listed on the cover page. They can only be deemed official if proper signatures are present.

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1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

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

1.1. Introduction to PASI

1.1.1. Pace Analytical Services, Inc. (PASI) is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. PASI offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, industrial hygiene testing, explosives, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. PASI has implemented a consistent Quality System in each of its laboratories and service centers. In addition, the company utilizes an advanced data management system that is highly efficient and allows for flexible data reporting. Together, these systems ensure data reliability and superior on-time performance. This document defines the Quality System and QA/QC protocols.

1.1.2. Our goal is to combine our expertise in laboratory operations with customized solutions to meet the specific needs of our customers.

1.2. Statement of Purpose

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

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. PASI management is committed to maintaining the highest possible standard of service for our customers by following a documented quality system. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the PASI network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work. This daily focus on quality is applied with initial project planning, continued through all field and laboratory activities, and is ultimately included in the final report generation.

1.3.3. PASI management demonstrates its commitment to quality by providing the resources, including facilities, equipment, and personnel to ensure the adherence to these documented policies and procedures and to promote the continuous improvement of the quality system. All PASI personnel must comply with all current applicable state, federal, and industry standards, and are required to perform all tests in accordance with stated methods and customer requirements.

1.4. Core Values

1.4.1. **Integrity-** Pace personnel are required to abide by the PASI Code of Ethics and all Pace employees must go through Data Integrity/Ethics training upon initial orientation and as an annual refresher.

1.4.2. **Value Employees-** Pace management views employees as our most important asset and communicates to them the relevance and importance of their activities within their job functions and how they contribute to the achievement of the objectives of the quality management system.

1.4.3. **Know Our Customers-** Pace makes every effort to know our customers and address their sampling and analytical needs. More information on this item can be found in section 2.0.

1.4.4. **Honor Commitments-** Pace labs focus on making solid commitments with regards to quality, capacity, and agreed upon turnaround time to our customers.

1.4.5. **Flexible Response To Demand-** Pace labs are equipped with both the material and personnel resources to enable them to be responsive to the demands of customers when situations or projects need change.

1.4.6. **Pursue Opportunities-** Pace is committed to pursuing opportunities for the growth of the company by constantly exploring markets and areas where we can expand.

1.4.7. **Continuously Improve-** Pace has committed much time and effort into establishing a continuous improvement program where company personnel meet on a regular basis to share ideas in cost reduction, production improvement and standardization in order to develop best practices. This information, as well as company financial and production metrics, are tracked, evaluated, and shared with each Pace facility.

1.5. Code of Ethics

1.5.1. PASI's fundamental ethical principles are as follows:

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

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

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

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

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

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

1.5.4. Any Pace employee can contact corporate management to report an ethical concern by calling the anonymous hotline at 612-607-6431.

1.6. Standards of Conduct

1.6.1. Data Integrity

1.6.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at PASI are the cornerstones of the company. Lack of data integrity is an assault on our most basic values putting PASI and its employees at grave financial and legal risk and will not be tolerated. Therefore, employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

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

1.6.2. Confidentiality

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

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

1.6.3. Conflict of Interest

1.6.3.1. PASI employees must avoid situations that might involve a conflict of interest or could appear questionable to others. The employee must be careful in two general areas:

1.6.3.1.1. Participation in activities that conflict or appear to conflict with the employees' PASI responsibilities.

1.6.3.1.2. Offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally. This includes bribes, gifts, kickbacks, or illegal payments.

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

1.6.4. Compliance

1.6.4.1. All employees are required to read, understand, and comply with the various components of the standards listed in this document. As confirmation that they understand their responsibility, each employee is required to sign an acknowledgment form annually that then becomes part of the employee's permanent record. Employees will be held accountable for complying with the Quality Systems as summarized in the Quality Assurance Manual.

1.7. Laboratory Organization

1.7.1. The PASI Corporate Office centralizes company-wide accounting, business development, financial management, human resources development, information systems, marketing, quality,

safety, and training activities. PASI's Director of Quality is responsible for assisting the development, implementation and monitoring of quality programs for the company. See Attachment IIB for the Corporate Organizational structure.

1.7.2. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office.

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

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

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

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

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

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

1.7.8. Appropriate support groups are present in each laboratory. The actual organizational structure for PASI – Green Bay is listed in Attachment IIA. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its accrediting authorities and revise the organizational chart in the Quality Assurance Manual (QAM) within 30 days. For changes in Department Managers or Supervisors or other laboratory personnel, no notifications will be sent to the laboratory's accrediting agencies; changes to the organizational chart will be updated during or prior to the annual review process. Changes or additions in these key personnel will also be noted by additional signatures on the QAM, as applicable. In any case, the QAM will remain in effect until the next scheduled revision.

1.8. Laboratory Job Descriptions

1.8.1. Senior General Manager

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

1.8.2. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.3. Quality Manager

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Quality. They may also report to a Senior Quality Manager within the same facility;
- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors Quality Assurance/Quality Control activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Quality office). The Quality Manager is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;

- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews and maintains records of proficiency testing results;
- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors correctives actions;
- Maintains the currency of the Quality Manual.

1.8.4. Quality Analyst

- Assists the SQM/QM in the performance of quality department responsibilities as delegated by the SQM/QM;
- Assists in monitoring QA/QC data;
- Assists in internal audits;
- Assists in maintaining training records;
- Assists in maintaining the document control system;

1.8.5. Technical Director

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

1.8.6. Administrative Business Manager

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

- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.7. Client Services Manager

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

1.8.8. Project Manager

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

1.8.9. Project Coordinator

- Responsible for preparation of project specifications and provides technical/project support;
- Coordinates project needs with other department sections and assists with proposal preparation;
- Prepares routine proposals and invoicing;
- Responsible for scanning, copying, assembling and binding final reports;
- Other duties include filing, maintaining forms, process outgoing mail, maintaining training database and data entry.

1.8.10. Department Manager/Supervisor

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

1.8.11. Group Supervisor/Leader

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

1.8.12. Laboratory Analyst

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

1.8.13. Laboratory Technician

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

1.8.14. Sample Management Personnel

- Signs for incoming samples and verifies the data entered on the Chain of custody forms;

- Enters the sample information into the Laboratory Information Management System (LIMS) for tracking and reporting;
- Stages samples according to EPA requirements;
- Assists Project Managers and Coordinators in filling bottle orders and sample shipments.

1.8.15. Systems Administrator or Systems Manager

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

1.8.16. Safety/Chemical Hygiene Officer

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

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

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

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through a web-based Learning Management System. After a new employee has been instructed in matters of human resources, they are given instructional materials for the LMS and a password for access.

1.9.2. A new hire training checklist is provided to the new employee that lists training items for the employee to work through either independently on LMS or with their supervisor or trainer. The training items that can be completed independently include:

- Reading through applicable Standard Operating Procedures;
- Reviewing the Quality Manual and Chemical Hygiene Plan;
- Core training modules such as quality control indicators, basic laboratory skills, etc.;
- Quality Systems training including traceability of measurements, method calibration, calibration verification, accuracy, precision and uncertainty of measurements, corrective actions, documentation, and root cause analysis;

- Data Integrity/Ethics training.

1.9.3. The new employee's Department Supervisor provides the employee with a basic understanding of the role of the laboratory within the structure of PASI and the basic elements of that individual's position. Supervised training uses the following techniques:

- Hands-on training
- Training checklists/worksheets
- Lectures and training sessions
- Method-specific training
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Proficiency testing programs.
- On-line courses

1.9.4. Group Supervisors/Leaders are responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's training file indicates what procedures an analyst or a technician is capable of performing, either independently or with supervision. The files also include documentation of continuing capability, which are fully detailed in Section 3.4. Training documentation files for each person are maintained by the Quality Office either in hardcopy format or within the LMS.

1.9.5. All procedures and training records are maintained and available for review during laboratory audits. These procedures are reviewed/updated periodically by laboratory management. Additional information can be found in SOP S-ALL-Q-020 **Training and Employee Orientation** or its equivalent revision or replacement.

1.10. Data Integrity System

1.10.1. The data integrity system at PASI provides assurances to management that a highly ethical approach is being applied to all planning, training and implementation of methods. Data integrity is crucial to the success of our company and Pace Analytical is committed to creating and maintaining a culture of quality throughout the organization. To accomplish this goal, PASI has implemented a data integrity system that encompasses the following four requirements:

1.10.1.1. A data integrity training program: standardized training is given to each new employee and a yearly refresher is presented to all employees. Key topics addressed by this training include:

1.10.1.1.1. Need for honesty and transparency in analytical reporting
1.10.1.1.2. Process for reporting data integrity issues
1.10.1.1.3. Specific examples of unethical behavior and improper practices
1.10.1.1.4. Documentation of non-conforming data that is still useful to the data user
1.10.1.1.5. Consequences and punishments for unethical behavior
1.10.1.1.6. Examples of monitoring devices used by management to review data and systems

1.10.1.2. Signed data integrity documentation for all employees: this includes a written quiz following the Ethics training session and written agreement to abide by the Code of Ethics and Standards of Conduct explained in the employee manual.

1.10.1.3. In-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.10.1.4. Documentation of any review or investigation into possible data integrity infractions. This documentation, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.10.2. PASI management makes every effort to ensure that personnel are free from any undue pressures that affect the quality of their work including commercial, financial, over scheduling, and working condition pressures.

1.10.3. Corporate management also provides all PASI facilities a mechanism for confidential reporting of data integrity issues that ensures confidentiality and a receptive environment in which all employees are comfortable discussing items of ethical concern. The anonymous message line is monitored by the Corporate Director of Quality who will ensure that all concerns are evaluated and, where necessary, brought to the attention of executive management and investigated. Any Pace employee can contact corporate management to report an ethical concern by calling the anonymous hotline at 612-607-6431.

1.11. Laboratory Safety

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

1.12. Security and Confidentiality

1.12.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by PASI staff. Keyless door lock combinations and computer access codes/logins are changed periodically. Posted signs direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors, including PASI staff from other facilities, must sign the Visitor's Logbook maintained by the receptionist. A staff member will accompany them during the duration of their stay on the premises unless the SGM/GM/AGM/OM, SQM/QM, or Technical Director specify otherwise. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out. The last staff member to leave their department for the day must ensure that all outside access points to that area are secure.

1.12.2. Additional security is provided where necessary, (e.g., specific secure areas for sample, data, and customer report storage), as requested by customers, or cases where national security is of concern. These areas are lockable within the facilities, or are securely offsite. Access is limited to specific individuals or their designees. Security of sample storage areas is the responsibility of the Sample Custodian. Security of samples and data during analysis and data reduction is the responsibility of Group Supervisors. Security of customer report archives is the responsibility of the Client Services Manager. These secure areas are locked whenever these individuals or their designees are not present in the facility.

1.12.3. Access to designated laboratory sample storage locations is limited to authorized personnel only. Provisions for lock and key access are provided. No samples are to be removed without proper authorization. If requested by customer or contract, samples are not to be removed from secure storage areas without filling out an associated internal chain of custody.

1.12.4. Standard business practices of confidentiality are applied to all documents and information regarding customer analyses. Specific protocols for handling confidential documents are described in PASI SOPs. Additional protocols for sample identification by internal laboratory identification numbers only are implemented as required under contract specific Quality Assurance Project Plans (QAPPs).

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

1.13. Communications

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

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

2.0. SAMPLE CUSTODY

2.1. Sampling Support

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

2.2. Field Services

2.2.1. Pace Analytical has a large Field Services Division which is based in their Minneapolis facility as well as limited field service capabilities in some of our other facilities. Field Services provides comprehensive nationwide service offerings including:

- Stack Testing
- Ambient Air
- CEM Certification Testing
- Air Quality Monitoring
- Onsite Analytical Services- FTIR and GC
- Real-time Process Diagnostic/Optimization Testing
- Wastewater, Groundwater and Drinking Water Monitoring
- Storm Water and Surface Water Monitoring
- Soil and Waste Sampling
- Mobile Laboratory Services

2.2.2. Field Services operates under the PASI Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in Standard Operating Procedures and Procedure Manuals.

2.3. Project Initiation

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

2.3.2. The laboratory maintains records of all such reviews, including discussions with customers. Routine analytical project documentation of quotes, notes, dates, initials, and/or recordings is maintained in a project folder by project management. Conditions for new and more complex contracts are determined by the SGM/GM/AGM/OM and sales representatives. Quality Management is consulted on technical requirements and operations staff provides input on volume capacities. Evidence of these reviews is maintained in the form of awarded Request for Proposals (RFPs), signed quotes or contracts, and a Customer Relationship Management (CRM) database. If a review identifies a potential mismatch between customer requirements and laboratory capabilities and/or capacities, Pace will specify its level of commitment by listing these exceptions to the requirements within the RFP, quote or contract.

2.3.3. Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-GB-C-012 **Review of Analytical Requests** or its equivalent revision or replacement.

2.4. Chain of Custody

2.4.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis. PASI has implemented Standard Operating Procedures to ensure that sample custody traceability and responsibility objectives are achieved for every project.

2.4.2. Field personnel or client representatives must complete a chain of custody for all samples that are received by the laboratory. The importance of completeness of COCs is stressed to the samplers and is critical to efficient sample receipt and to insure the requested methods are used to analyze the correct samples.

2.4.3. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.4.4. The sampler is responsible for providing the following information on the chain of custody form:

- Customer project name
- Project location or number
- Field sample number/identification
- Date and time sampled
- Sample matrix
- Preservative
- Requested analyses
- Sampler signature
- Relinquishing signature
- Date and time relinquished
- Sampler remarks as needed
- Custody Seal Number if present
- Regulatory Program Designation
- The state where the samples were collected to ensure all applicable state requirements are met
- Turnaround time requested
- Purchase order number

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

2.4.6. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.5. Sample Acceptance Policy

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

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

2.5.3. All samples must:

- Have unique customer identification that is clearly marked on durable waterproof labels affixed to the sample containers that match the chain of custody.
- Have clear documentation on the chain of custody related to the location of the sampling site with the time and date of sample collection.
- Have the sampler's name and signature.
- Have all requested analyses clearly designated on the COC.
- Have clear documentation of any special analytical or data reporting requirements.
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be correctly preserved unless the method allows for laboratory preservation.
- Be received within holding time. Any samples with hold times that are exceeded will not be processed without prior customer approval.
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval.
- Be received within appropriate temperature ranges - not frozen but ≤6°C (See Note 1), unless program requirements or customer contractual obligations mandate otherwise (see Note 2). The cooler temperature is recorded directly on the COC and the SCUR. Samples that are delivered to the laboratory immediately after collection are considered acceptable if there is evidence that the chilling process has been started. For example, by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data associated with any deviations from the above sample acceptance policy requirements will be appropriately qualified.

Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be read and recorded to

±0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ±0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤6°C limit

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

Note 3: Biological Tissue Samples should be received frozen at ≤0°C or on ice ≤6°C.

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

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

2.5.5. Samples for drinking water analysis that are improperly preserved, or are received past holding time, are rejected at the time of receipt, with the exception of VOA samples that are tested for pH at the time of analysis.

2.5.6. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.6. Sample Log-in

2.6.1. After sample inspection, all sample information on the chain of custody is entered into the Laboratory Information Management System (LIMS). This permanent record documents receipt of all sample containers including:

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

2.6.2. All samples received are logged into the LIMS within one working day of receipt. Sample login may be delayed due to customer clarification of analysis needed, corrective actions for sample receipt non-conformance, or other unusual circumstances. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 08:00 as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

2.6.3. The Laboratory Information Management System (Epic Pro) automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of BB-XXXXX-YYY. The BB represents the laboratory identification within Pace's laboratory network. The 5 digit "X" number represents the project number followed by a 3 digit sample number. The project number is a sequential number that is assigned as a new project is created. The sample number corresponds to the number of samples submitted by the client. In addition to the unique sample ID, there is a sample container ID that consists of the sample number, the container type (ex. BP1U), and bottle 1 of Y, where Y represents the total number of containers of that particular type. Together the sample LIMs number and sample container ID number create a unique barcode encryption that can be linked to the sample analysis requested by the client. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the customer's field identification; it will be a permanent reference number for all future interactions.

2.6.4. Current division codes are noted below. These division codes are used primarily for accounting purposes and LIMS sample identifications. More division codes may be added without updating this document.

10 = Minnesota/Montana
92 = Asheville and Charlotte
60 = Kansas
50 = Indianapolis
12 = Virginia/Duluth MN
51 = Columbus
75 = Dallas
35 = Florida
20 = Gulf Coast
30 = Pittsburgh
40 = Green Bay
17 = Pace Life Sciences
65 = Schenectady, NY
36 = South Florida

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

2.6.6. Samples with hold times that are near expiration date/time may be sent directly to the laboratory for analysis at the discretion of the Project Manager and/or SGM/GM/AGM/OM.

2.6.7. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.7. Sample Storage

2.7.1. **Storage Conditions**

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

2.7.1.2. Storage blanks, consisting of two 40mL aliquots of reagent water, are stored with volatile samples and are used to measure cross-contamination acquired during storage. If applicable, laboratories must have documented procedures and criteria for evaluating storage blanks, appropriate to the types of samples being stored.

2.7.1.3. Additional information can be found in S-GB-Q-028 **Monitoring Temperature Controlled Units.**

2.7.2. **Temperature Monitoring**

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

2.7.2.2. The temperature of each refrigerated storage area is maintained at ≤6°C unless state or program requirements differ. The temperature of each freezer storage area is maintained at <-10°C unless state or program requirements differ. The temperature of each storage area is checked and documented each day of use (each calendar day). If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

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

Additional information can be found in S-GB-Q-028 **Monitoring Temperature Controlled Units.**

2.7.3. **Hazardous Materials**

2.7.3.1. Pure product or potentially heavily contaminated samples must be tagged as "hazardous" or "lab pack" and stored separately from other samples.

2.7.4. **Foreign/Quarantined Soils**

2.7.4.1. Depending on the soil disposal practices of the laboratory, foreign soils and soils from USDA regulated areas are adequately segregated to enable proper sample disposal. The USDA requires these samples to be incinerated or sterilized by an approved treatment procedure. Additional information regarding USDA regulations and sample handling can be found in applicable local laboratory SOPs.

2.7.4.2. Additional information on sample storage can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement and in S-GB-W-001 **Waste Handling and Management**.

2.8. Sample Protection

2.8.1. PASI laboratory facilities are operated under controlled access protocols to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted at all times.

2.8.2. Samples are removed from storage areas by designated personnel and returned to the storage areas, if necessary, immediately after the required sample quantity has been taken.

2.8.3. Upon customer request, additional and more rigorous chain of custody protocols for samples and data can be implemented. For example, some projects may require internal chain-of-custody protocols.

2.8.4. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.9. Subcontracting Analytical Services

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

2.9.2. Prior to subcontracting samples to a laboratory outside Pace Analytical, the potential sub-contract laboratory will be pre-qualified by verifying that the subcontractor meets the following criteria:

- All certifications required for the proposed subcontract are in effect,
- Sufficient professional liability and other required insurance coverage is in effect, and
- Is not involved in legal action by any federal, state, or local government agency for data integrity issues and has not been convicted in such investigation at any time during the past 5 years.

2.9.3. The contact and preliminary arrangements are made between the PASI Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract laboratory agreement include:

- Method of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Laboratory certification requirement
- Price per analysis
- Turn-around time requirements

2.9.4. Chain-of-custody forms are generated for samples requiring subcontracting to other laboratories. Sample receiving personnel re-package the samples for shipment, create a transfer chain of custody form and record the following information:

- Pace Analytical Laboratory Number
- Matrix
- Requested analysis
- Special instructions regarding turnaround, required detection or reporting limits, or any unusual information known about the samples or analytical procedure.
- Signature in "Relinquished By"

2.9.5. All subcontracted sample data reports are sent to the PASI Project Manager. Pace will provide a copy of the subcontractor's report to the client when requested.

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

2.9.7. Additional information can be found in S-GB-C-009 **Subcontracting Samples** or its equivalent revision or replacement.

2.10. Sample Retention and Disposal

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

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

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

2.10.4. Additional information can be found in S-GB-W-001 **Waste Handling and Management** and S-GB-C-010 **Sample Management** or their equivalent revisions or replacements.

3.0. ANALYTICAL CAPABILITIES

3.1. Analytical Method Sources

3.1.1. PASI laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. The latest valid editions of methodologies are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 is a representative listing of general analytical protocol references. PASI discloses in writing to its customers and regulatory agencies any instances in which modified methods are being used in the analysis of samples.

3.1.2. In the event of a customer-specific need, instrumentation constraint or regulatory requirement, PASI laboratories reserve the right to use valid versions of methods that may not be the most recent edition available.

3.2. Analytical Method Documentation

3.2.1. The primary form of PASI laboratory documentation of analytical methods is the Standard Operating Procedure (SOP). SOPs contain pertinent information as to what steps are required by an analyst to successfully perform a procedure. The required contents for the SOPs are specified in the company-wide SOP for Preparation of SOPs (S-ALL-Q-001).

3.2.2. The SOPs may be supplemented by other training materials that further detail how methods are specifically performed. This training material will undergo periodic, documented review along with the other Quality System documentation.

3.3. Analytical Method Validation

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

3.4. Demonstration of Capability (DOC)

3.4.1. Analysts complete an initial demonstration of capability (IDOC) study prior to performing a method or when there is a change in instrument type, personnel, or test method, or at any time that a method has not been performed by the laboratory or analyst in a 12-month period. The mean recovery and standard deviation of each analyte, taken from 4 replicates of a quality control standard is calculated and compared to method criteria (if available) or established laboratory criteria for evaluation of acceptance. Each laboratory maintains copies of all demonstrations of capability, including those that fail acceptance criteria and corresponding raw data for future reference and must

document the acceptance criteria prior to the analysis of the DOC. Demonstrations of capability are verified on an annual basis.

3.4.2. For Continuing Demonstrations of Capability, the laboratories may use Performance Testing (PT) samples in lieu of the 4-replicate approach listed above. For methods or procedures that do not lend themselves to the "4-replicate" approach, the demonstration of capability requirements will be specified in the applicable SOP. Drinking Water DOCs must be done at or below the MCL.

3.4.3. Additional information can be found in SOP S-ALL-Q-020 **Training and Employee Orientation** or its equivalent revision or replacement.

3.5. Regulatory and Method Compliance

3.5.1. PASI understands that expectations of our customers commonly include the assumption that laboratory data will satisfy specific regulatory requirements. Therefore PASI attempts to ascertain, prior to beginning a project, what applicable regulatory jurisdiction, agency, or protocols apply to that project. This information is also required on the chain of custody submitted with samples.

3.5.2. PASI makes every effort to detect regulatory or project plan inconsistencies, based upon information from the customer, and communicate them immediately to the customer in order to aid in the decision making process. PASI will not be liable if the customer chooses not to follow PASI recommendations.

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

4.0. QUALITY CONTROL PROCEDURES

Quality control data is analyzed and where they are found to be outside pre-defined criteria, planned action is taken to correct the problem in order to prevent incorrect results from being reported. Quality control samples are to be processed in the same manner as client samples.

4.1. Method Blank

4.1.1. A method blank is used to evaluate contamination in the preparation/analysis system and is processed through all preparation and analytical steps with its associated samples.

4.1.2. A method blank is processed at a minimum frequency of one per preparation batch In the case of a method that has no separate preparation step, a method blank is processed with no more than 20 samples of a specific matrix performed by the same analyst, using the same method, standards, and reagents.

4.1.3. The method blank consists of a matrix similar to the associated samples that is known to be free of analytes of interest. Method blanks are not applicable for certain analyses, such as pH, conductivity, flash point and temperature

4.1.4. Method blanks are not applicable for certain analyses, such as pH, conductivity, flash point and temperature.

4.1.5. Each method blank is evaluated for contamination. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the reporting limit and is greater than 1/10 of the amount of that analyte found in any associated sample. Some labs, due to client requirements, etc., may have to evaluate their method blanks down to ½ the reporting limit as opposed to the reporting limit itself. Corrective actions for blank contamination may include the re-preparation and re-analysis of all samples (where possible) and quality control samples. Data qualifiers must be applied to results that are considered affected by contamination in a method blank.

4.1.6. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.2. Laboratory Control Sample

4.2.1. The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis.

4.2.2. An LCS is processed at a minimum frequency of one per preparation batch. In the case of a method that has no separate preparation step, an LCS will be processed with no more than 20 samples of a specific matrix performed by the same analyst, using the same method, standards, and reagents.

4.2.3. The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes.

4.2.4. The LCS contains **all** analytes specified by a specific method or by the customer or regulatory agency, which may include full list of target compounds, with certain exceptions. These exceptions may include analyzing only specific Aroclors when PCB analysis is requested or not spiking with all EPA Appendix IX compounds when a full Appendix IX list of compounds is requested. However,

the lab must ensure that all target components in its scope of accreditation are included in the spike mixture for the LCS over a two (2) year period. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

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

4.2.5. The LCS is evaluated against the method default or laboratory-derived acceptance criteria. For those methods that require laboratory-derived limits, method default control limits may be used until the laboratory has a minimum of 20, but preferably greater than 30, data points from which to derive internal acceptance criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any associated sample containing an 'out-of-control' compound must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier. When the acceptance criteria for the LCS are exceeded high, and there are associated samples that are non-detects, then those non-detects can be reported with data qualifiers, or when the acceptance criteria are exceeded low, those associated sample results may be reported if they exceed the maximum regulatory limit/decision level with data qualifiers.

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

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

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

4.2.8. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

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

4.3.1. A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch unless the MS is actually used as the LCS.

4.3.2. A **Matrix Spike/Matrix Spike Duplicate** (MS/MSD) set is processed at a frequency specified in a particular method or as determined by a specific customer request. This frequency will be specified in the applicable method SOP or customer QAPP. In the absence of such requirements, an MS/MSD set is routinely analyzed once per every 20 samples per matrix per method.

4.3.3. The MS and MSD consist of the sample matrix that is then spiked with known concentrations of target analytes. Laboratory personnel spike customer samples that are specifically designated as MS/MSD samples or, when no designated samples are present in a batch, randomly select samples to spike that have adequate sample volume or weight. Spiked samples are prepared and analyzed in the same manner as the original samples and are selected from different customers if possible.

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

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

4.3.6. A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method.

4.3.7. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.4. Sample Duplicate

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

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

4.4.3. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.5. Surrogates

4.5.1. Surrogates are compounds that reflect the chemistry of target analytes and are typically added to samples for organic analyses to monitor the effect of the sample matrix on compound recovery.

4.5.2. Surrogates are added to each customer sample (for organics), method blank, LCS, MS, and calibration standard prior to extraction or analysis. The surrogates are evaluated against the method or laboratory derived acceptance criteria or against project-specific acceptance criteria specified by the client, if applicable. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error. An exception to this would be samples that have high surrogate values but no reportable hits for target compounds. These samples would be reported, with a qualifier, because the implied high bias would not affect the final results. For methods with multiple surrogates, documentation regarding acceptance and associated compounds will be found in the individual method SOPs.

4.5.3. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.6. Internal Standards

4.6.1. Internal Standards are method-specific analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, sample, and calibration standard at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. At a minimum, the laboratory will follow method specific guidelines for the treatment of internal standard recoveries as they are related to the reporting of data.

4.6.2. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.7. Field Blanks

4.7.1. Field blanks are blanks prepared at the sampling site in order to monitor for contamination that may be present in the environment where samples are collected. These field quality control samples are often referenced as field blanks, rinsate blanks, or equipment blanks. The laboratory analyzes these field blanks as normal samples and informs the customer if there are any target compounds detected above the reporting limits.

4.8. Trip Blanks

4.8.1. Trip blanks are blanks that originate from the laboratory as part of the sampling event and are used to monitor for contamination of samples during transport. These blanks accompany the empty sample containers to the field and then accompany the collected samples back to the laboratory. These blanks are routinely analyzed for volatile methods where ambient background contamination is likely to occur.

4.9. Limit of Detection (LOD)

4.9.1. PASI laboratories are required to use a documented procedure to determine a limit of detection for each analyte of concern in each matrix reported. All sample processing steps of the preparation and analytical methods are included in this determination including any clean ups. For any test that does not have a valid LOD, sample results below the limit of quantitation (LOQ) cannot be reported.

4.9.2. The LOD is initially established for the compounds of interest for each method in a clean matrix with no target analytes present and no interferences at a concentration that would impact the results. The LOD is then determined every time there is a change in the test method that affects how the test is performed or when there has been a change in the instrument that affects the sensitivity. If required by customer, method or accreditation body, the LOD will be re-established annually for all applicable methods.

4.9.3. Unless otherwise noted, the method used by PASI laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. Where required by regulatory program or customer, the above referenced procedure will be followed.

4.9.4. Where specifically stated in the published method, LODs or MDLs will be performed at the listed frequency.

4.9.5. The validity of the LOD must be shown by detection (a value above zero) of the analytes in a QC sample in each quality system matrix. The QC sample must contain the analyte at no more than 3X the LOD for a single analyte test and 4X the LOD for multiple analyte tests. This verification must be performed on each instrument used for sample analysis and reporting of data. The validity of the LOD must be verified as part of the LOD determination process. This verification must be done prior to the use of the LOD for sample analysis.

4.9.6. An LOD study is not required for any analyte for which spiking solutions or quality control samples are not available such as temperature.

4.9.7. The LOD, if required, shall be verified annually for each quality system matrix, technology and analyte. In lieu of performing full LOD (MDL) studies annually, the laboratory can verify the LOD (MDL) on an annual basis, providing this verification is fully documented and does not contradict other customer or program requirements that the laboratory must follow. The requirements of this verification are:

- The spike concentration of the verification must be no more than 3X times the LOD for single analyte tests and 4X the LOD for multiple analyte tests.
- The laboratory must verify the LOD on each instrument used for the reporting of sample data.
- The laboratory must be able to identify all target analytes in the verification standard (distinguishable from noise).

4.9.8. Additional information can be found in SOP S-GB-Q-020 **Determination of LOD and LOQ** or its equivalent revision or replacement.

4.10. Limit of Quantitation (LOQ)

4.10.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For PASI laboratories, this LOQ is referred to as the RL, or Reporting Limit. This RL is based on the lowest

calibration standard concentration that is used in each initial calibration. Results below this level are not allowed to be reported without qualification since the results would not be substantiated by a calibration standard. For methods with a determined LOD, results can be reported out below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

4.10.2. The LOQ must be higher than the LOD.

4.10.3. To verify the LOQ, the laboratory will prepare a sample in the same matrix used for the LCS. The sample will be spiked with each target analyte at a concentration equivalent to the RL or 2X the RL. This sample must undergo the routine sample preparation procedure including any routine sample cleanup steps. The sample is then analyzed and the recovery of each target analyte determined. The recovery for each target analyte must meet the laboratories current control limits for an LCS. The annual LOQ verification is not required if the LOD was determined or verified annually on that instrument.

4.10.4. Additional information can be found in SOP S-GB-Q-020 **Determination of LOD and LOQ** or its equivalent revision or replacement.

4.11. Estimate of Analytical Uncertainty

4.11.1. PASI laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, PASI laboratories base this estimation on the recovery data obtained from the Laboratory Control Spikes. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-GB-Q-010 **Estimation of Measurement Uncertainty** or its equivalent revision or replacement.

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

4.12. Proficiency Testing (PT) Studies

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

4.12.2. The laboratory initiates an investigation whenever PT results are deemed 'unacceptable' by the PT provider. All findings and corrective actions taken are reported to the SQM/QM or their designee. A corrective action plan is initiated and this report is sent to the appropriate state accreditation agencies for their review. Additional PTs will be analyzed and reported as needed for certification purposes.

4.12.3. PT samples are treated as typical customer samples, utilizing the same staff, methods, equipment, facilities, and frequency of analysis. PT samples are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

4.12.4. Comparison of analytical results with anyone participating in the same PT study is prohibited prior to the close of the study.

4.12.5. Additional information can be found in SOP S-GB-Q-021 **Proficiency Testing Program** or its equivalent revision or replacement.

4.13. Rounding and Significant Figures

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

4.13.2. Data is compared to the reporting limits and MDLs to determine if qualifiers are needed before the rounding step occurs.

4.13.3. **Rounding:** PASI-Green Bay follows the odd / even guidelines for rounding numbers:

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

4.13.4. **Significant Digits**

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

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

4.14. Retention Time Windows

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

4.14.2. One process for the production of retention time windows: Make 3 injections of all single component or multi-component analytes over a 72-hour period. Record the retention time in minutes for each analyte and surrogate to 3 decimal places. Calculate the mean and standard deviation of the three absolute retention times for each target analyte and surrogate. For multi-component analytes, choose 3-5 major peaks and calculate the mean and standard deviation for each of the peaks. If the standard deviation of the retention times of a target analyte is 0.000, the lab may use a default standard deviation of 0.01. The width of the retention time window for each analyte and surrogate and major peak in a multi-component analyte is defined as +/- 3 times the standard deviation of the mean absolute retention time established during that 72-hour period or 0.03 minutes, whichever is greater.

4.14.3. The center of the retention time window is established for each analyte and surrogate by using the absolute retention times from the CCV at the beginning of the analytical shift. For samples run with an initial calibration, use the retention time of the mid-point standard of the initial calibration curve.

4.14.4. For more information, please reference the local facility's analytical SOPs.

5.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

5.1. Document Management

5.1.1. Additional information can be found in SOP S-GB-Q-029 **Document Control and Management** or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

5.1.2. Pace Analytical Services, Inc. has an established procedure for managing documents that are part of the quality system. The list of managed documents includes, but is not limited to, Standard Operating Procedures (both technical and non-technical), Quality Assurance Manuals, quality policy statements, training documents, work-processing documents, charts, posters, memoranda, notices, forms, software, and any other procedures, tables, plans, etc. that have a direct bearing on the quality system (including applicable data records and non-technical documents).

5.1.3. A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. This establishes that there are no invalid or obsolete documents in use in the facility. All documents are reviewed periodically and revised if necessary. Obsolete documents are systematically discarded or archived for audit or knowledge preservation purposes.

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

5.1.5. SOPs, specifically, are available to all laboratory staff via the Learning Management System (LMS) which is a secure repository that is accessed through an internet portal. As a local alternative to the hard copy system of controlled documents, secured electronic copies of controlled documents may be maintained on the laboratory's local server. These document files must be read-only for all personnel except the Quality Department and system administrator. Other requirements for this system are as follows:

- Electronic documents must be readily accessible to all facility employees.
- Electronic documents (i.e. pdf's) must be locked from printing. All hardcopy SOPs must be obtained from the Quality Department.

5.1.6. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for PASI. The base QAM template is distributed by the Corporate Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template and submit those modifications to the Corporate Director of Quality for review. Once approved and signed by both the CEO and the Director of Quality; the SGM/GM/AGM/OM, the SQM/QM, and any Technical Directors sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis by all of the PASI SQMs/QMs and revised accordingly by the Director of Quality.

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

5.1.7.1. SOPs fall into two categories: company-wide documents and facility specific documents. Company-wide SOPs start with the prefix S-ALL- and local SOPs start with the individual facility prefix.

5.1.7.2. The purpose of the company-wide SOPs is to establish policies and procedure that are common and applicable to all PASI facilities. Company-wide SOPs are document-controlled by the corporate quality office and signed copies are distributed to all of the SQMs/QMs. The local management personnel sign the company-wide SOPs. The SQM/QM is then in charge of distribution to employees, external customers, or regulatory agencies and maintaining a distribution list of controlled document copies.

5.1.7.3. Local PASI facilities are responsible for developing facility-specific SOPs applicable to their respective facility. The local facility develops these facility-specific SOPs based on the corporate-wide SOP template. This template is written to incorporate a set of minimum method requirements and PASI best practice requirements. The local facilities may add to or modify the corporate-wide SOP template provided there are no contradictions to the minimum method or best practice requirements. Facility-specific SOPs are controlled by the applicable SQM/QM according to the corporate document management policies.

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

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

5.1.7.6. Additional information can be found in SOP S-GB-Q-017 **Preparation of SOPs** or its equivalent revision or replacement.

5.1.8. **Other Documentation**

5.1.8.1. Insert additional procedures and protocols relevant for any other controlled documents employed within the laboratory.

5.2. Document Change Control

5.2.1. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After revisions are approved, a revision number is assigned and the previous version of the document is officially retired. Copies may be kept for audit or knowledge preservation purposes.

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

5.3. Management of Change

5.3.1. The process for documenting necessary changes within the laboratory network are not typically handled using the corrective or preventive action system as outlined in section 9.0. Management of Change is a proactive approach to dealing with change to minimize the potential negative impact of systematic change in the laboratory and to ensure that each change has a positive desired outcome. This process will primarily be used for the implementation of large scale projects and information system changes as a means to apply consistent systems or procedures within the laboratory network. The request for change is submitted by the initiator and subsequently assigned to an individual or team for development and planning. The final completion of the process culminates in final approval and verification that the procedure was effectively implemented. Additional information can be found in SOP S-GB-Q-016 **Management of Change** or its equivalent revision or replacement.

6.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

Each PASI facility is equipped with sufficient instrumentation and support equipment to perform the relevant analytical testing or field procedures performed by each facility. Support equipment includes chemical standards, thermometers, balances, disposable and mechanical pipettes, etc. This section details some of the procedures necessary to maintain traceability and to perform proper calibration of instrumentation and support equipment. See Attachment III for a list of equipment currently used at the Green Bay PASI facility.

6.1. Standards and Traceability

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

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

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

6.1.4. All prepared standard or reagent containers include the PASI identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials. This ensures traceability back to the standard preparation logbook.

6.1.5. For containers that are too small to accommodate labels that list all of the above information associated with a standard, the minimum required information will be PASI standard ID, concentration, and expiration date. This assures that no standard will be used past its assigned expiration date.

6.1.6. If a second source standard is required to verify an existing calibration or spiking standard, this standard must be obtained from a different manufacturer or from a different lot unless client specific QAPP requirements state otherwise.

6.1.7. Additional information concerning standards and reagent traceability can be found in the SOP S-GB-Q-026 **Standard and Reagent Management and Traceability** or its equivalent revision or replacement.

6.2. General Analytical Instrument Calibration Procedures (Organic and Inorganic)

6.2.1. All support equipment and instrumentation are calibrated or checked before use to ensure proper functioning and verify that the laboratory's requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards

traceable to recognized national standards or reference standards whose values have been statistically validated.

6.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in the narrative. Any specific method requirement for number and type of calibration standards supersedes the general requirement. Instrument and method specific calibration criteria are explained within the specific analytical standard operating procedures for each facility.

6.2.3. Results from all calibration standards analyzed must be included in constructing the calibration curve with the following exceptions:

6.2.3.1. The lowest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The reporting limit must be adjusted to the lowest concentration included in the calibration curve;

6.2.3.2. The highest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done an individual analyte basis. The upper limit of quantitation must be adjusted to the highest concentration included in the calibration curve;

6.2.3.3. Multiple points from either the high end or the low end of the calibration curve may be excluded as long as the remaining points are contiguous in nature and the minimum number of levels remains as established by method or standard operating procedure. The reporting limit or quantitation range, whichever is appropriate, must be adjusted accordingly;

6.2.3.4. Results from a concentration level between the lowest and highest calibration levels can only be excluded from an initial calibration curve for a documentable and acceptable cause with approval from the responsible department supervisor and the local SQM/QM or their designee. An acceptable cause is defined as an obvious sample introduction issue that resulted in no response or a documented response that is less than the lowest standard used in the ICAL. A suspected incorrectly prepared standard is not considered to be an acceptable cause. The results for all analytes are to be excluded and the point must be replaced by re-analysis. Re-analysis of this interior standard must occur within the same 12-hour tune time period for GC/MS methodologies and within 8 hours of the initial analysis of that standard for non-GC/MS methodologies. All samples analyzed prior to the re-analyzed calibration curve point must be re-analyzed after the calibration curve is completed and re-processed against the final calibration curve.

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

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

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

6.2.7. **General Organic Calibration Procedures**

6.2.7.1. Calibration standards are prepared at a minimum of five concentrations for organic analyses (unless otherwise stipulated in the method).

6.2.7.2. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Curves that do not meet the appropriate criteria require corrective action that may include re-running the initial calibration curve. Rounding to meet initial calibration criteria is not allowed, that is, 15.3 cannot be rounded down to meet a $\leq$ 15% RSD requirement. This also applies to linear and non-linear fit requirements. All initial calibrations are verified with an initial calibration verification standard (ICV) obtained from a second manufacturer or second lot from the same manufacturer if that lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

6.2.7.3. The calibration curve is periodically verified by the analysis of a mid-level continuing calibration verification (CCV) standard during the course of sample analysis. This standard is from the same source as the initial calibration unless otherwise specified in the source method to be from an alternate source material. Rounding to meet continuing calibration criteria is not allowed. Continuing calibration verification is performed at the beginning and end of each analytical batch except if an internal standard is used, then only one verification at the beginning of the batch is needed, whenever it is expected that the analytical system may be out of calibration, if the time period for calibration has expired, or for analytical systems that have specific calibration verification requirements. This verification standard must meet acceptance criteria in order for sample analysis to proceed.

6.2.7.4. In the event that the CCV does not meet the acceptance criteria, a second CCV may be injected as part of the diagnostic evaluation and corrective action investigation. If the second CCV is acceptable, the analytical sequence may be continued. If both CCVs fail, the analytical sequence is terminated and corrective action is initiated. Sample analysis cannot begin until after documented corrective action has been completed and either two consecutive passing CCVs have been analyzed or the instrument has successfully passed a new initial calibration. All samples analyzed since the last compliant CCV are re-analyzed for methodologies utilizing external calibration.

6.2.7.5. When instruments are operating unattended, autosamplers may be programmed to inject consecutive CCVs as a preventative measure against CCV failure with no corrective action. In this case, both CCVs must be evaluated to determine potential impact to the results. A summary of the decision tree and necessary documentation are listed below:

- If both CCVs meet the acceptance criteria, the analytical sequence is allowed to continue without corrective action. The 12 hour clock begins with the injection of the second CCV.
- If the first CCV does not meet the acceptance criteria and the second CCV is acceptable, the analytical sequence is continued and the results are reported.
- If the first CCV meets the acceptance criteria and the second CCV is out of control, the samples after the out of control CCV must be re-analyzed in a compliant analytical sequence.
- If both CCVs are out of control, all samples since the last acceptable CCV must be re-analyzed in a compliant analytical sequence.

6.2.7.6. Some analytical methods require that samples be bracketed by passing CCVs analyzed both before and after the samples. This is specific to each method but, as a general rule, all external calibration methods require bracketing CCVs. Most internal standard calibrations do not require bracketing CCVs.

6.2.7.7. Some analytical methods require verification based on a time interval; some methods require a frequency based on an injection interval. The type and frequency of the calibration verifications is dependent on both the analytical method and possibly on the quality program associated with the samples. The type and frequency of calibration verification will be documented in the method specific SOP employed by each laboratory.

6.2.8. General Inorganic Calibration Procedures

6.2.8.1. The instrument is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. A calibration blank is also included. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Rounding to meet initial calibration criteria is not allowed. This also applies to linear and non-linear fit requirements. The number of calibration standards used depends on the specific method criteria or customer project requirements, although normally a minimum of three standards is used.

6.2.8.2. The ICP and ICP/MS can be standardized with a zero point and a single point calibration if:

- Prior to analysis, the zero point and the single point calibration are analyzed and a linear range has been established,
- Zero point and single point calibration standards are analyzed with each batch
- A standard corresponding to the LOQ is analyzed with the batch and meets the established acceptance criteria
- The linearity is verified at the frequency established by the method or manufacturer.

6.2.8.3. All initial calibrations are verified with an initial calibration verification standard (ICV) obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

6.2.8.4. During the course of analysis, the calibration curve is periodically verified by the analysis of calibration verification standards (CCV). A calibration verification standard is analyzed within each analytical batch at method/program specific intervals to verify that the initial calibration is still valid. The CCV is also analyzed at the end of the analytical batch.

6.2.8.5. A calibration blank is also run with each calibration verification standard to verify the cleanliness of the system. All reported results must be bracketed by acceptable CCVs. Instrument and method specific calibration acceptance criteria are explained within the specific analytical standard operating procedures for each facility.

6.2.8.6. Interference check standards are also analyzed per method requirements and must meet acceptance criteria for metals analyses.

6.3. Support Equipment Calibration Procedures

6.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until repaired. The laboratory maintains records to demonstrate the correction factors applied to working thermometers.

6.3.2. On each day the equipment is used, balances, ovens, refrigerators (those used to keep samples and standards at required temperatures), freezers, and water baths are checked in the expected use range with NIST traceable references in order to ensure the equipment meets laboratory specifications and these checks are documented appropriately.

6.3.3. **Analytical Balances**

6.3.3.1. Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Calibration weights are ASTM Class 1 or other class weights that have been calibrated against a NIST standard weight and are re-certified every 5 years at a minimum against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the Quality department.

6.3.4. **Thermometers**

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

6.3.4.2. Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures. Each thermometer is individually numbered and assigned a correction factor based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

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

6.3.5. **pH/Electrometers**

6.3.5.1. The meter is calibrated before use each day, using fresh buffer solutions. Additional information regarding pH/Electrometers can be found in SOP S-GB-I-071 *Measurement of pH in Water, Soil and Waste* or its equivalent revision or replacement.

6.3.6. **Spectrophotometers**

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

6.3.7. **Mechanical Volumetric Dispensing Devices**

6.3.7.1. Mechanical volumetric dispensing devices including bottle top dispensers (those that are critical in measuring a required amount of reagent), pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis. Glass microliter syringes are checked for accuracy prior to initial use.

6.3.7.2. Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-GB-Q-030 **Support Equipment** or its equivalent revision or replacement.

6.4. Instrument/Equipment Maintenance

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

6.4.2. The Operations Manager and/or department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

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

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

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

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

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

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

6.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily.

7.0. CONTROL OF DATA

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

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

7.1. Analytical Results Processing

7.1.1. When analytical, field, or product testing data is generated, it is either recorded in a bound laboratory logbook (e.g., Run log or Instrument log) or copies of computer-generated printouts that are appropriately labeled and filed. These logbooks and other laboratory records are kept in accordance with each facility's Standard Operating Procedure for documentation storage and archival. If the laboratory chooses to minimize or eliminate its paper usage, these records can be kept on electronic media. In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues.

7.1.2. The primary analyst is responsible for initial data reduction and review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets and LIMS fields.

7.1.3. The primary analyst then compiles the initial data package for verification. This compilation must include sufficient documentation for data review. It may include standard calibrations, chromatograms, manual integration documentation, electronic printouts, chain of custody forms, and logbook copies.

7.1.4. Some agencies or customers require different levels of data reporting. For these special levels, the primary analyst may need to compile additional project information, such as initial calibration data or extensive spectral data, before the data package proceeds to the verification step.

7.2. Data Verification

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

7.2.2. Analysts performing the analysis and subsequent data reduction have primary responsibility for quality of the data produced. The primary analyst initiates the data verification process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. Data review checklists, either hardcopy or electronic, are used to document the data review process. The primary analyst is responsible for the initial input of the data into the LIMS. The primary analyst and reviewer must be clearly identified on all applicable data review checklists.

7.2.3. The completed data package is then sent to a designated qualified reviewer (this cannot be the primary analyst). The following criteria have been established to qualify someone as a data reviewer. To perform secondary data review, the reviewer must:

7.2.3.1. Have a current Demonstration of Capability (DOC) study on file and have an SOP acknowledgement form on file for the method/procedure being reviewed; or, See Note

7.2.3.2. Have a DOC on file for a similar method/technology (i.e., GC/MS) and have an SOP acknowledgment form on file for the method/procedure being reviewed; or, See Note

7.2.3.3. Supervise or manage a Department and have an SOP acknowledgment form on file for the method/procedure being reviewed; or,

7.2.3.4. Have significant background in the department/methods being reviewed through education or experience and have an SOP acknowledgment form on file for the method/procedure being reviewed.

7.2.4. **Note:** Secondary reviewer status must be approved personally by the SQM/QM or SGM/GM/AGM/OM in the event that this person has no prior experience on the specific method or general technology.

7.2.5. This reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer validates the data entered into the LIMS and documents approval of manual integrations.

7.2.6. Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data (or designating the review of data electronically). The Operations or Project Manager examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution.

7.2.7. Additional information regarding data review procedures can be found in SOP S-ALL-Q-037 **Data Review** or its equivalent revision or replacement, as well as in SOP S-GB-Q-024 **Manual Integration** or its equivalent revision or replacement.

7.2.8. The Data Checker program will process validated data for a given project against a set of pre-determined requirements and known chemistry relationships. The program creates a report that includes a series of warnings and errors for any requirement or condition determined to be suspect or incorrect. These warnings and error counts are displayed on the "Project Inquiry by Client" screen and on the final Data Checker reports. For projects that have any number of warnings or errors, the Data Checker report will provide a message that identifies the source and condition of the error or warning.

7.2.9. Some reports and/or data packages may be reviewed by the QM or SQM or designee based on program requirements (e.g., DoD) or client requirements. In this case a thorough review for completeness and accuracy may include a compilation of raw data and QC summaries in addition to the final report to produce a full deliverable package. See SOP S-GB-Q-022 (or equivalent replacement), **Audits and Inspections**, for full Quality department final report and raw data review requirements.

7.3. Data Reporting

7.3.1. Data for each analytical fraction pertaining to a particular PASI project number are delivered to the Project Manager for assembly into the final report. All points mentioned during technical and QC reviews are included in a case narrative if there is potential for data to be impacted.

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

7.3.2.1. A title which designates the report as “Final Report”, “Laboratory Results”, “Certificate of Results”, etc.;

7.3.2.2. Name and address of laboratory (or subcontracted laboratories, if used);

7.3.2.3. Phone number and name of laboratory contact to where questions can be referred;

7.3.2.4. A unique identification number for the report. The pages of the report shall be numbered and a total number of pages shall be indicated;

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

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

7.3.2.7. Identification of any sample that did not meet acceptable sampling requirements of the relevant governing agency, such as improper sample containers, holding times missed, sample temperature, etc.;

7.3.2.8. Date and time of collection of samples, date of sample receipt by the laboratory, dates of sample preparation and analysis, and times of sample preparation and analysis when the holding time for either is 72 hours or less;

7.3.2.9. Identification of the test methods used;

7.3.2.10. Identification of sampling procedures if sampling was conducted by the laboratory;

7.3.2.11. Deviations from, additions to, or exclusions from the test methods. These can include failed quality control parameters, deviations caused by the matrix of the sample, etc., and can be shown as a case narrative or as defined footnotes to the analytical data;

7.3.2.12. Identification of whether calculations were performed on a dry or wet-weight basis;

7.3.2.13. Reporting limits used;

7.3.2.14. Final results or measurements, supported by appropriate chromatograms, charts, tables, spectra, etc.;

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

7.3.2.16. Date report was issued;

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

7.3.2.18. If necessary, a statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory;

7.3.2.19. Identification of all test results provided by a subcontracted laboratory or other outside source;

7.3.2.20. Identification of results obtained outside of quantitation levels.

In addition to the requirements listed above, final reports shall also contain the following items when necessary for the interpretation of results:

7.3.2.21. Deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions;

7.3.2.22. Where relevant, a statement of compliance/non-compliance with requirements and/or specifications (e.g., the TNI standard);

7.3.2.23. Where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit;

7.3.2.24. Where appropriate and needed, opinions and interpretations, which may include opinions on the compliance/non-compliance of the results with requirements, fulfillment of contractual requirements, recommendations on how to use the results, and guidance to be used for improvement;

7.3.3. Additional items may be required per Client QAPPs or different state regulations.

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

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

7.3.6. The following positions are the only approved signatories for PASI final reports:

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

7.4. Data Security

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

7.5. Data Archiving

7.5.1. All records compiled by PASI are maintained legible and retrievable and stored secured in a suitable environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. These records may include, but are not limited to, customer data reports, calibration and maintenance of equipment, raw data from instrumentation, quality control documents, observations, calculations, and logbooks. These records are retained in order to provide for possible historical reconstruction including sampling, receipt, preparation, analysis, and personnel involved. TNI-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the SQM/QM or a designated Data Archivist.

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

7.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained by the acquiring entity for a minimum of five years. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

7.6. Data Disposal

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

8.0. QUALITY SYSTEM AUDITS AND REVIEWS

8.1. Internal Audits

8.1.1. Responsibilities

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

8.1.2. Scope and Frequency of Internal Audits

8.1.2.1. The complete internal audit process consists of the following four sections:

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

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

8.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible. Examples of system-wide elements that can be audited include:

- Quality Systems documents, such as Standard Operating Procedures, training documents, Quality Assurance Manual, and all applicable addenda
- Data records and non-technical documents
- Personnel and training files.
- General laboratory safety protocols.
- Chemical handling practices, such as labeling of reagents, solutions, and standards as well as all associated documentation.
- Documentation concerning equipment and instrumentation, calibration/maintenance records, operating manuals.
- Sample receipt and management practices.
- Analytical documentation, including any discrepancies and corrective actions.

- General procedures for data security, review, documentation, reporting, and archiving.
- Data integrity issues such as proper manual integrations.

8.1.2.4. When the operations of a specific department are evaluated, a number of additional functions are reviewed including:

- Detection limit studies
- Internal chain of custody documentation
- Documentation of standard preparations
- Quality Control limits and Control charts

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

8.1.2.6. A representative number of data audits are completed annually. Findings from these data audits are handled in the same manner as those from other internal and external audits.

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

8.1.3. Internal Audit Reports and Corrective Action Plans

8.1.3.1. Additional information can be found in SOP S-GB-Q-022 **Internal and External Audits** or its equivalent revision or replacement.

8.1.3.2. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the SQM/QM writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

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

8.1.3.4. Once completed, the internal audit report is issued jointly to the SGM/GM/AGM/OM and the manager(s)/supervisor(s) of the audited operation at a minimum. The responsible manager(s)/supervisor(s) responds within 14 days with a proposed plan to correct all of the deficiencies cited in the audit report. The SQM/QM may grant additional time for responses to large or complex deficiencies (not to exceed 30 days). Each response must include timetables for completion of all proposed corrective actions.

8.1.3.5. The SQM/QM reviews the audit responses. If the response is accepted, the SQM/QM uses the action plan and timetable as a guideline for verifying completion of the corrective action(s). If the SQM/QM determines that the audit response does not adequately address the correction of cited deficiencies, the response will be returned for modification.

8.1.3.6. To complete the audit process, the SQM/QM performs a re-examination of the areas where deficiencies were found to verify that all proposed corrective actions have been implemented. An audit deficiency is considered closed once implementation of the necessary corrective action has been audited and verified. This is usually within 60-90 days after implementation. If corrective action cannot be verified, the associated deficiency remains open until that action is completed.

8.2. External Audits

8.2.1. PASI laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

8.2.2. Audit teams external to the company review the laboratory to assess the effectiveness of systems and degree of technical expertise. The SQM/QM and other QA staff host the audit team and assist in facilitation of the audit process. Generally, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. In some cases, items of concern are discussed during a debriefing convened at the end of the on-site review process.

8.2.3. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM. The SGM/GM/AGM/OM provides the necessary resources for staff to develop and implement the corrective action plans. The SQM/QM collates this information and provides a written response to the audit team. The response contains the corrective action plan and expected completion dates for each element of the plan. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

8.3. Quarterly Quality Reports

8.3.1. The SQM/QM is responsible for preparing a quarterly report to management summarizing the effectiveness of the laboratory Quality Systems. This status report will include:

- Overview of quality activities for the quarter
- Certification status
- Proficiency Testing study results
- SOP revision activities
- Internal audit (method/system) findings
- Manual integration audit findings (Mintminer)
- Raw Data and Final Report review findings
- MDL activities
- Other significant Quality System items

8.3.2. The Corporate Director of Quality utilizes the information from each laboratory to make decisions impacting the quality program compliance of the company as a whole. Each

SGM/GM/AGM/OM utilizes the quarterly report information to make decisions impacting Quality Systems and operational systems at a local level.

8.3.3. Additional information can be found in SOP S-ALL-Q-014 **Quality System Review** or its equivalent revision or replacement.

8.4. Annual Managerial Review

8.4.1. A managerial review of Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements.

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

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

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

8.5. Customer Service Reviews

8.5.1. As part of the annual managerial review listed previously, the sales staff is responsible for reporting on customer feedback, including complaints. The acquisition of this information is completed by performing surveys.

8.5.2. The sales staff continually receives customer feedback, both positive and negative, and reports this feedback to the laboratory management in order for them to evaluate and improve their management system, testing activities and customer service.

8.5.3. In addition, the labs must be willing to cooperate with customers or their representatives to clarify customer requests and to monitor the laboratory's performance in relation to the work being performed for the customers. This cooperation may include providing the customer reasonable access to relevant areas of the lab for the witnessing of tests being performed; or the preparation of samples or data deliverables to be used for verification purposes.

8.5.4. Customer service is an important aspect to Pace's overall objective of providing a quality product. Good communication should be provided to the customer's throughout projects. The lab should inform the customer of any delay or major deviations in the performance of analytical tests.

9.0. CORRECTIVE ACTION

Additional information can be found in SOP S-GB-Q-027 **Corrective and Preventive Actions** or its equivalent revision or replacement.

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

9.1. Corrective Action Documentation

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

- Internal Laboratory Non-Conformance Trends
- PE/PT Sample Results
- Internal and External Audits
- Data or Records Review (including non-technical records)
- Client Complaints
- Client Inquiries
- Holding Time violations

9.1.2. Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g., matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

9.1.3. The person who discovers the deficiency or non-conformance initiates the corrective action documentation on the Non-Conformance Corrective/ Preventive Action report and/or LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

9.1.4. In the event that the laboratory is unable to determine the cause, laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within LabTrack or on the Corrective/Preventive Action Report.

9.1.5. After all the documentation is completed, the routing of the Corrective/Preventive Action Report and /or LabTrack will continue from the person initiating the corrective action, to their immediate supervisor or the applicable Project Manager and finally to the SQM/QM, if applicable, who may be responsible for final review and signoff of corrective/preventive actions.

9.1.6. In the event that analytical testing or results do not conform to documented laboratory policies or procedures, customer requirements, or standard specifications, the laboratory shall investigate the significance of the non-conformance and document appropriate corrective actions. The proper level of laboratory management will review any departure from these requirements for technical suitability. These departures are permitted only with the approval of the SGM/GM/AGM/OM or the SQM/QM. Where necessary, Project Management will notify the customer of the situation and will advise of any ramifications to data quality (with the possibility of work being recalled). The procedures for handling non-conforming work are detailed in SOP S-GB-Q-027 **Corrective and Preventive Actions** or its equivalent revision or replacement.

9.2. Corrective Action Completion

9.2.1. Internal Laboratory Non-Conformance Trends

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

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting error

9.2.2. PE/PT Sample Results

9.2.2.1. Any PT result assessed as “not acceptable” requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

9.2.3. **Internal and External Audits**

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

9.2.4. **Data Review**

9.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data reviews (e.g., by the SQM/QM), errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

9.2.5. **Client Complaints**

9.2.5.1. Project Managers are responsible for issuing corrective action forms, when warranted, for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor for investigation. After potential corrective actions have been determined, the Project Manager reviews the corrective action form to ensure all customer needs or concerns are being adequately addressed.

9.2.6. **Client Inquiries**

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

9.2.7. **Holding Time Violations**

9.2.7.1. In the event that a holding time has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the SQM/QM must be made aware of all holding time violations.

9.2.7.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the hold time excursion and the ultimate resolution is then documented and included in the customer project file. The SQM/QM includes a list of all missed holding times in their Quarterly Report to the corporate quality office.

9.3. Preventive Action Documentation

9.3.1. Pace laboratories can take advantage of several available information sources in order to identify needed improvements in all of their systems including technical, managerial, and quality. These sources may include:

- Management Continuous Improvement Plan (CIP) metrics which are used by all production departments within Pace. When groups compare performance across the company, ways to improve systems may be discovered. These improvements can be made within a department or laboratory-wide.

- Annual managerial reviews- part of this TNI-required and NVLAP-required review is to look at all processes and procedures used by the laboratory over the past year and to determine ways to improve these processes in the future.
- Quality systems reviews- any frequent checks of quality systems (monthly logbook reviews, etc.) can uncover issues that can be corrected or adjusted before they become a larger issue.

9.3.2. When improvement opportunities are identified or if preventive action is required, the laboratory can develop, implement, and monitor preventive action plans.

10.0. GLOSSARY

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

3P Program	The Pace Analytical continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all PASI labs.
Acceptance Criteria	TNI and DoD- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI and DoD- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
Accrediting Authority	DoD- The Territorial, State or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.
Accrediting (or Accreditation) Body	DoD- Authoritative body that performs accreditation.
Accuracy	TNI and DoD- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI and DoD- 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	DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- The evaluation process used to measure the performance or effectiveness of a system and its elements against specific criteria. Note: In this standard (DoD), assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	DoD- A process in which a sample is converted to free atoms.

Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. DoD- A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.
Batch	TNI and DoD- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. South Carolina- same definition as TNI except 24 hours should be changed to 8 hours.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Blind Sample	DoD- A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.
Calibration	TNI and DoD- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. DoD- The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	DoD- A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration. DoD- A substance or reference material used to calibrate an instrument.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. DoD- A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.
Chain of Custody	DoD- An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of custody Form (COC)	TNI and DoD- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	DoD- Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Confirmation	TNI and DoD- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures.
Conformance	DoD- An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.

Congener	DoD- A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a CVS in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Data Audit	DoD- A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e. that they meet specified acceptance criteria).
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.

Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form. DoD- 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	DoD- 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.
Demonstration of Capability	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate acceptable accuracy.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate is 1%.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form.
Document Control	DoD- 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.
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	DoD- The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	DoD- A solvent used to carry the components of a mixture through a stationary phase.
Elute	DoD- To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	DoD- A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	DoD- The process of measuring or collecting environmental data.

Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.
False Positive	DoD- An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition. Note: For DoD, the finding must be linked to a specific requirement.
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.

Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The time elapsed from the time of sampling to the time of extraction or analysis, or from extraction to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	DoD- One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP-AES that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.

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

Laboratory Duplicate	DoD- Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
LabTrack	Database used by Pace Analytical to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A training database used by Pace Analytical to train their employees. This system is a self-paced system which is capable of tracking all employee training requirements and documentation.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. DoD- The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate is 1%.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.
Laboratory Information Management System (LIMS)	A computer system that is used to maintain all sample information from sample receipt, through preparation and analysis and including sample report generation.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.
Management	DoD- Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	DoD- System to establish policy and objectives and to achieve those objectives.
Manager (however named)	DoD- The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI and DoD- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.

Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. DoD- A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI and DoD- 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	TNI and DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI and DoD- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	DoD- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	DoD- 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.
MintMiner	Program used by Pace Analytical to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.

Negative Control	DoD- Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	DoD- An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Performance Audit	DoD- The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	DoD- Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI and DoD- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI- Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. DoD- Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing	TNI and DoD- A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Proficiency Testing Program	TNI and DoD- 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 Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria. DoD- A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.
Protocol	TNI and DoD- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client. DoD- An integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	DoD- A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. DoD- The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of the users.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. DoD- 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.
Quality Manual	TNI and DoD- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions are to be used for purposes of batch and quality control requirements: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard. The quantitation range lies within the calibration range.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. DoD- Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank)	DoD- A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. DoD- A 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 Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location. DoD- A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.
Reference Toxicant	DoD- The toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e. statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A client-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (or otherwise named)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	DoD- Denotes a mandatory specification; often designated by the term "shall".
Retention Time	DoD- The time between sample injection and the appearance of a solute peak at the detector.
Sample	DoD- Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by Pace Analytical sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).

Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selective Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. DoD- The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.
Sensitivity	TNI and DoD- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	DoD- Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	DoD- Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio	DoD- 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.
Spike	DoD- A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI and DoD- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.

Standard (Chemical)	DoD- Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	DoD- A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. DoD- 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 Reference Material (SRM)	DoD- A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Supervisor	DoD- The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes specifically named by a client (also called project-specific analytes).
Technical Director	DoD- Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	DoD- A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.

Test Method	DoD- An adoption of a scientific technique for performing a specific measurement as documented in a laboratory SOP or as published by a recognized authority.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. DoD- The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	DoD- A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty Measurement	The parameter associated with the result of a measurement that characterized the dispersion of the values that could be reasonably attributed to the measurand (i.e. the concentration of an analyte).
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification	TNI and DoD- Confirmation by examination and objective 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 adjustment, to repair, 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 instrument's individual record.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).
Work Cell	DoD- A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented.

11.0. REFERENCES

11.1. "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136.

11.2. "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.

11.3. "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.

11.4. U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis.

11.5. U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis.

11.6. "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.

11.7. "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.

11.8. "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.

11.9. "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.

11.10. "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (September 1986).

11.11. Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.

11.12. Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.

11.13. Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.

11.14. Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.

11.15. Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.

11.16. Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, 1988.

11.17. National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards. Most recent version.

11.18. ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.

11.19. Department of Defense Quality Systems Manual (QSM), version 4.2, October 25, 2010.

11.20. TNI (The NELAC Institute) Standards; most recent version.

12.0. REVISIONS

The PASI Corporate Quality Office files both a paper copy and electronic version of a Microsoft Word document with tracked changes detailing all revisions made to the previous version of the Quality Assurance Manual. This document is available upon request. All revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual 16.0	Section 2.6.5: added VM/Duluth. Sections 2.7.1.3 and 2.7.2.2: added SOT references. Section 4.1.2: added parenthetical phrase directing the reader to the glossary section. Section 4.1.3: added language from old section 4.1.4 and deleted language in order to match current practices. Section 4.1.4: .reworded for clarity. Also added last sentence in red text to allow labs to insert additional method blank requirements. Sections 4.1.7, 4.2.9, 4.4.4, and 6.2.7.8: revised wording per updated Ohio VAP requirements. Sections 4.5.2 and 4.6.1: added 'calibration standard' to list of QC items that require the addition of surrogates and internals. Also added red letter text for additional lab-specific information. Section 4.10.3: fixed LOQ verification language to match TNI standard (V1M4, section 1.5.2.2.c). Old section 4.12.2: deleted. Covered in reference in current section 4.12.5. Section 6.2.3: moved language that had been in the 'organic calibration only' section to this general calibration section. The language in this section applies to both organic and inorganic tests. Section 6.2.7.3: added clarification statement regarding the calibration verification standard. Section 6.3.7.1: reworded for clarity and added red letter text for calibration of micro-liter syringes. Section 7.2.5: added language specifying secondary reviewer documents approval of manual integrations. Section 7.2.7: added reference to the Manual Integration SOP. Section 7.2.8: added new red-letter text language to match Data Checker SOP. Section 7.2.9: added new red-letter text language to comply with DoD QSM 4.2. Section 8.3.1: deleted items in order to match current SOP S-ALL-Q-014. Added red-letter text to the following sections for Ohio VAP labs: 2.5.2.1, 4.5.2.1, 4.6.3, and 7.6.2. Attachment VI: added red letter text under title to satisfy AZ state requirement. Attachment VIII, Analyte Chart: changed holding times expressed as '6 Months' to '180 Days' to match actual practice as defined by LIMS acodes. Attachment VIII, Analyte Chart: added explanation under the header to explain the holding times expressed in the chart.	05Feb2013

ATTACHMENT I- QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

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

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

PERCENT DIFFERENCE (%D)

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

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

PERCENT DRIFT

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

RELATIVE PERCENT DIFFERENCE (RPD)

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

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

CORRELATION COEFFICIENT (R)

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

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

ATTACHMENT I- QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

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

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

AVERAGE ($\overline{X}$)

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

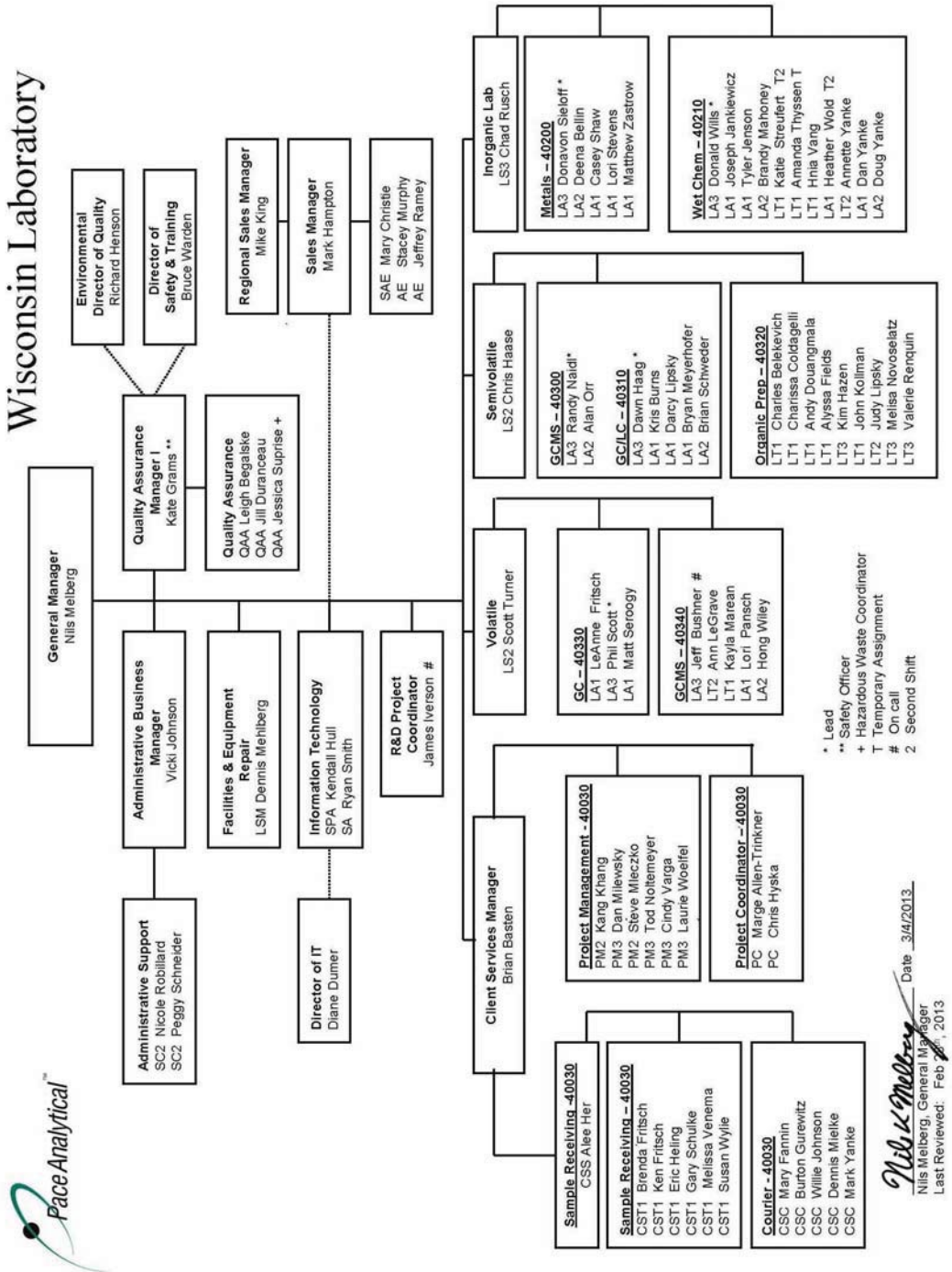
where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

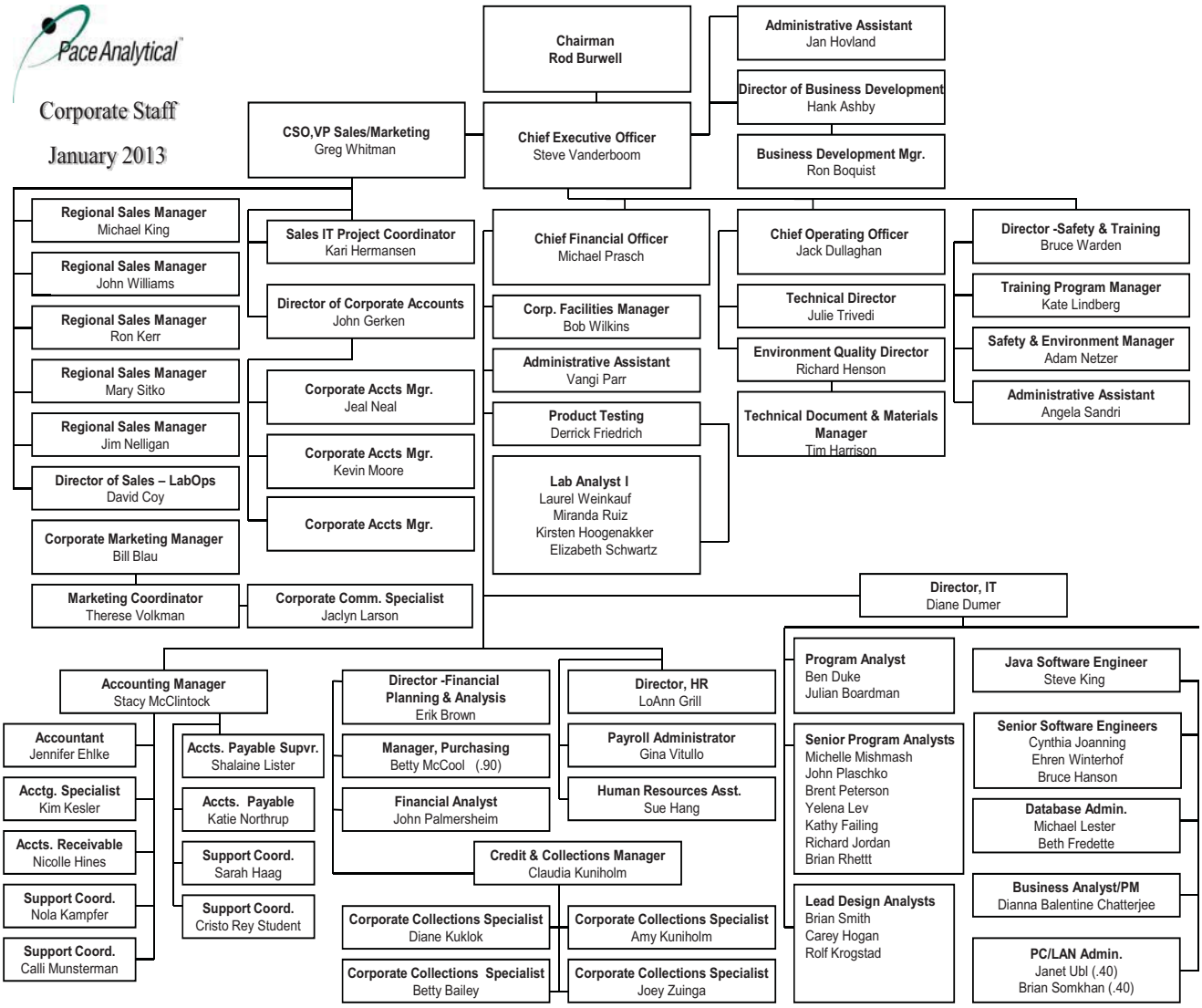
$$RSD = \frac{S}{\overline{X}} * 100$$

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

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



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



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

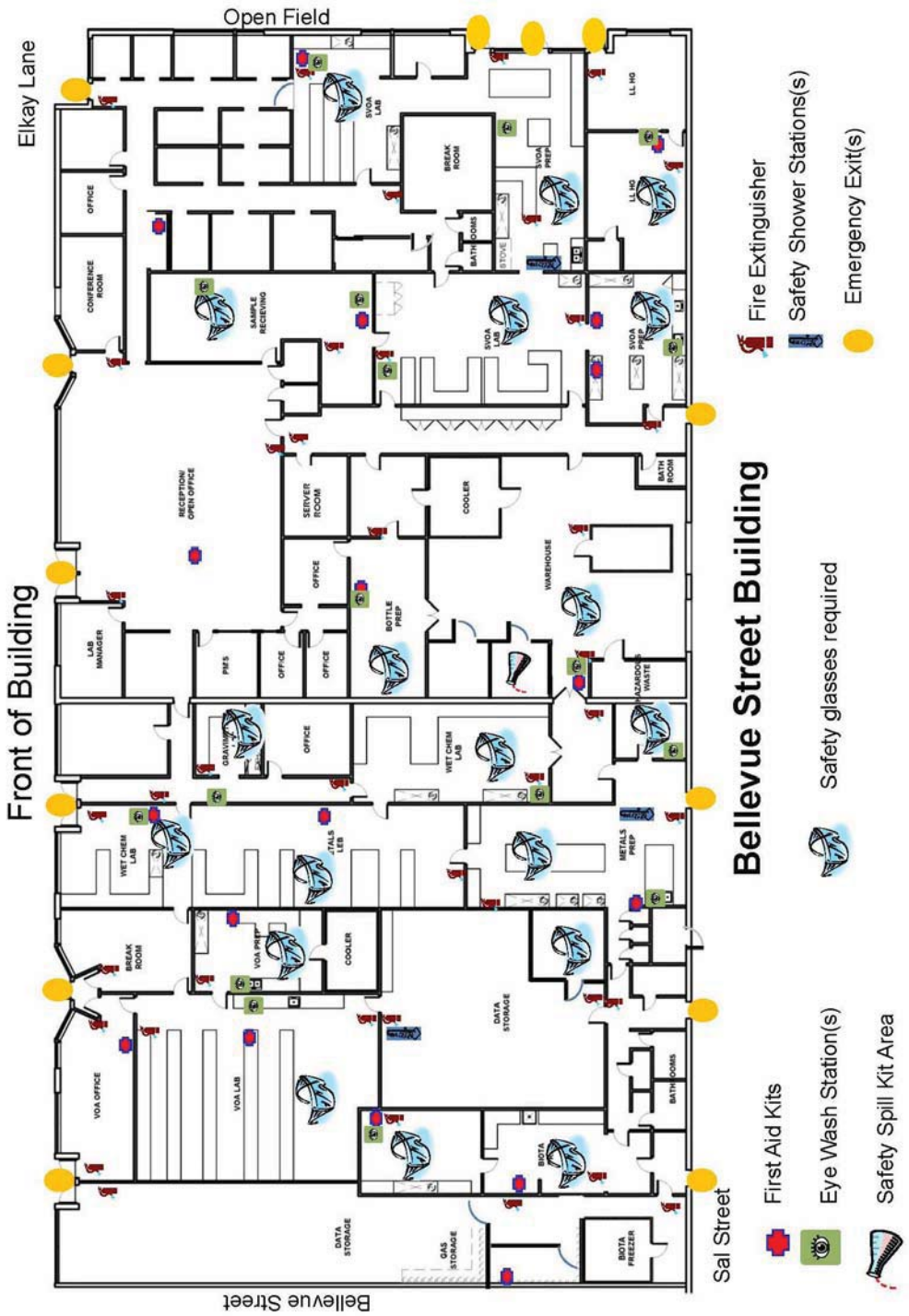
DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
QA	Hot Wire Anemometer	40HWA02	Control Company	4330	-	Air Flow Velocity
SVOA	GC	40GCS1	Hewlett-Packard	5890	FID	WI-DRO/8015
SVOA	GC	40GCS3	Hewlett-Packard	5890	FID	WI-DRO/8015
SVOA	GC	40GCS4	Hewlett-Packard	5890	FID	WI-DRO/8015SCREEN
SVOA	GC	40GCS5	Hewlett-Packard	5890	FID	WI-DRO/8015 ALCOHOL
SVOA	GC	40GCS6	Hewlett-Packard	5890	ECD/ECD	8081
SVOA	GC	40GCS7	Hewlett-Packard	6890	ECD/ECD	
SVOA	GC	40GCS8	Hewlett-Packard	6890	ECD/ECD	8081
SVOA	GC	40GCS9	Hewlett-Packard	6890	ECD/ECD	8082
SVOA	GC	40GCSA	Hewlett-Packard	5890	ECD/ECD	Screener
SVOA	GC	40GCSB	Hewlett-Packard	6890	ECD/ECD	8082
SVOA	GC	40GCSC	Hewlett-Packard	6890	ECD/ECD	8082
SVOA	GC	40GCSE	Hewlett-Packard	5890	ECD/ECD	8082
SVOA	GC	40GCSF	Hewlett-Packard	7890	FID	WI-DRO/8015
SVOA	GC/MS	40MSS1	Hewlett-Packard	5890	HP 5972	8270C/625
SVOA	GC/MS	40MSS2	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS3	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS4	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS5	Hewlett-Packard	5890	HP 5972	8270 SCREEN
SVOA	GC/MS	40MSS6	Hewlett-Packard	5890	HP 5972	8270C
SVOA	GC/MS	40MSS7	Hewlett-Packard	7890A	HP 5975	8270C
SVOA	Separatory Funnel Extractor	40SFE1	Lab Line	-	-	SW846 3510C
SVOA	Separatory Funnel Extractor	40SFE2	Lab Line	-	-	SW846 3510C
SVOA	Separatory Funnel Extractor	40SFE3	Lab Line	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC1	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC2	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC3	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC4	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC5	Zymark	-	-	SW846 3510C
SVOA	Sonicator	40SON3	Lab Line	9334	-	
SVOA	Precision Water Bath	40WB01	Precision	-	-	
SVOA	Water Baths	40WB02	-	-	-	
SVOA	Tube Heaters	-	Kontes	-	-	
SVOA	Accuprep MPS GPC Cleanup System (John)	40GPC2	J2 Scientific	-	-	SW846 3640A
SVOA	Accuprep MPS GPC Cleanup System (Jane)	40GPC1	J2 Scientific	-	-	SW846 3640A
SVOA	Mars Xtraction Microwave System	40MIC1	CEM	907501	-	SW846 3546
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX1	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX2	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX3	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX4	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX5	Gerhardt Soxtherm	SE-416	-	SW846 3540C

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX6	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX7	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX8	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Sonifier Cell Disruptors with Horns	Sonicator #2	-	-	-	SW846 3550B
SVOA	Sonifier Cell Disruptors with Horns	40SON1	Misonix	3000		SW846 3550B
SVOA	Sonifier Cell Disruptors with Horns	40SON2	Misonix	S-4000		SW846 3550B
SVOA	Environ Shaker	40SKR1	LabLine	3527	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT1	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT2	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT3	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT4	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT5	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT6	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT7	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT8	-	-	-	-
SVOA	Extraction Mixer	40EXT1	-	-	-	-
SVOA	Extraction Mixer	40EXT2	-	-	-	-
SVOA	Extraction Mixer	40EXT3	-	-	-	-
SVOA	Extraction Mixer	40EXT4	-	-	-	-
SVOA	Extraction Mixer	40EXT5	-	-	-	-
VOA	GC/MS	40MSV1	Hewlett-Packard	5890	HP 5972	SW846 8260B/5030
VOA	GC/MS	40MSV2	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV3	Agilent	6850	Agilent 5975	SW846 8260B/624/5030B
VOA	GC/MS	40MSV5	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV7	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV8	Agilent	6850	Agilent 5975B	SW846 8260B/624/5030B
VOA	GC/MS	40MSVA	Hewlett-Packard	7890	Agilent 5975C	SW846 8260B/624/5030B
VOA	GC/MS	40MSVB	Hewlett-Packard	7890A	Agilent 5975C	SW846 8260B/624/5030B
VOA	GC	40GCV1	Hewlett-Packard	5890	Agilent 5975	SW846 8021/WI MOD GRO
VOA	GC	40GCV2	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
VOA	GC	40GCV3	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV4	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV5	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV8	Hewlett-Packard	5890	FID	SW846 8015 - MEE
VOA	GC	40SCREEN1	Hewlett-Packard	5890	FID	Screener
VOA	GC	40SCREEN2	Hewlett-Packard	5890	FID	Screener
VOA	SNOOP	40SCREEN3	Research Engineers	-	-	Screener
VOA	Sonicator	40SON4	LabLine	9314	-	VOA Prep
VOA	Sonicator	40SON5	LabLine	9333	-	VOA Prep
WET	Apollo	40WTA5	Tekmar/Dohrmann	Apollo 9000	-	EPA 9060
WET	Fusion	40WTA7	Teledyne	14-9600-100	-	EPA 9060, SM 5310C
WET	Analytik Jena	40WTAA	Analytik Jenna	Multi EA 4000	-	EPA 9060, SM 5310C
WET	SmartChem	40WTA9	Westco Scientific	Smartchem 200		
WET	Oxygen Meter	40WET1	YSI	5000	YSI 5010	SM 5210B--01, SM 4500-O G
WET	BOD AUTOEZ	40WETE	Thermo/Orion	10060020	-	SM 5210B
WET	Flashpoint Instrument A	40WET4	Precision	-	-	EPA 1010A
WET	Flashpoint Instrument B	40WET5	Fisher	-	-	EPA 1010A
WET	Turbidimeter	40WET6	Hach	2100P	-	EPA 180.1, SM2130B
WET	Conductivity Meter	40WET7	Accument	30	-	EPA 120.1
WET	pH Meter	40WET8	Orion	720A	-	pH Meter
WET	EH Meter	40WET9	Accument	AB15	-	EH Meter
WET	Radiometer	40WET0	Titration	TIM840		Alkalinity - 2320B
WET	pH meter	40WETB	Symphony	SB20	-	1311, 1312, ASTM D3987
WET	pH Meter	40WETC	Orion	720A	-	pH Meter
WET	pH Meter	40WETD	Corning	320	-	pH Meter
WET	MICRO DIST Rapid Distillation System	40MD1	Lachat	-	-	350.1, 9012A
WET	MICRO DIST Rapid Distillation System	40MD2	Lachat	-	-	350.1, 9012A
WET	MICRO DIST Rapid Distillation System	40MD3	Lachat	-	-	350.1, 9012A
WET	Block Digestion System	40HB01	Westco Scientific	40/20	-	365.4, 351.2
WET	Block Digestion System	40HB02	Tecator	2040	-	365.4, 351.2
WET	Hot Block Digestion System	40HB03	Environmental Express	SC100	-	6010/6020
WET	COD Reactor	40COD1	Hach	45600	-	410.4
WET	COD Reactor	40COD2	Bioscience INC	100 003	-	410.4
WET	Direct Reading Spectrophotometer	40WTA1	Hach	DR 2000	-	EPA 410.4, SM3500Cr-B, Hach 8146, Walkley Black TOC
WET	Quik Chem 8000	40WTA2	Lachat	8000	-	350.1, 365.4, 9012A, 310.2, 351.2, 353.2
WET	Ion Chromatograph	40WTA3	Dionex	DX-120	-	EPA 300.0, 9056A
WET	Ion Chromatograph	40WTA4	Dionex	DX-120	-	EPA 300.0, 9056A

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
WET	Ion Chromatograph	40WTAB	Thermo Scientific	ICS-110	-	EPA 300.0, 9056A
WET	Quik Chem 8500 Series 2	40WTAC	Lachat	8500 Series 2	-	EPA 353.2, 350.1
WET	Hellige Colony Counter	-	Hellige	-	-	
WET	Steril-Quick Autoclave	-	National	704-9000-D	-	
WET	Stereomaster Stereoscope	-	Fisher	FW 99-20-1385	-	
WET	Sonicator	40SON6	Branson	8210	-	
WET	TCLP Tumbler	40TBL1	Environmental Express	-	-	1311, 1312, ASTM D3987
WET	TCLP Tumbler	40TBL2	Environmental Express	-	-	1311, 1312, ASTM D3987
WET	Reaction Vessel Tumbler	40TBL3	Reaction Vessel	-	-	1311, 1312, ASTM D3987
WET	Centrefudge	40CENT1	International Equipment Co	H260	-	
WET	Buret	40BUR1	KIMAX	253	-	
WET	Buret	40BUR2	Pyrex	2103	-	
MET	Hot Water Bath	40WB05	Thermo Fisher Scientific	2841	-	
MET	ICP	40ICP2	Thermo	ICAP 6500	-	
MET	Chiller	40ICP2	Neslab/Thermo	Selex 900	-	6010
MET	ICPMS	40ICM2	Thermo	X Series 2	-	6020
MET	Chiller	40ICM2	Neslab/Thermo	Merlin M75	-	6020
MET	UPS	40ICM2	Toshiba	1600EP		
MET	Vacuum Pump	40ICM2	Edwards	EM28		
MET	ICPMS	40ICM3	Thermo	X Series 2	-	6020
MET	Chiller	40ICM3	Neslab/Thermo	2500	-	6020
MET	UPS	40ICM3	Toshiba	1600EP		
MET	Vacuum Pump	40ICM3	Edwards	EM28		
MET	Quick Trace Mercury Analyzer	40HG2	Cetac	M-7500		7470/7471
MET	DMA-80 Direct Mercury Analyzer	40HG4	Milestone	DMA-80	-	7473
MET	Hot Block Metals Digestion System	40HB04	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	40HB06	Environmental Express	SC154	-	6010/6020
MET	Hot Block Metals Digestion System	40HB07	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	40HB08	Environmental Express	SC196	-	6010/6020
MET	Low-level Mercury Analyzer	40LHG1	Tekran	2500	2500	1631E
MET	Low Level Mercury Analyzer	40LHG4	Analytik Jena	Mercur	-	1631E
MET	Low Level Mercury Analyzer	40LHG5	Cetac	M-8000	-	1631E

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



ATTACHMENT V- LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
MANUALS					
Quality Manual	14.1	Quality Manual	1-Oct-04	7-Oct-11	Quality
CHP/Safety	8.0	Chemical Hygiene Plan/Safety Manual	18-Jul-11	1-Jun-12	Safety
SAMPLE MAN					
S-ALL-C-002	03	Bottle Order Database	4-Oct-06	5-Oct-12	Sample Management
S-ALL-C-005	03	Operation of PacePort Customer Feedback Form	27-Nov-07	16-Nov-12	Sample Management
S-GB-C-001	04	Procedure to Preserve Samples for Volatile Organic Analysis of Solid Matrices by Method 5035	12-Apr-05	5-Oct-12	Sample Management
S-GB-C-002	04	Extruding Sample from 25 g EnCores for Volatile and Semivolatile Analyses	12-Apr-05	5-Oct-12	Sample Management
S-GB-C-005	04	Maintenance of Ice Chests and Shipping Containers	1-Jun-05	7-Dec-11	Sample Management
S-GB-C-007	03	Laboratory Tracking of Samples	6-Jun-05	27-Sep-11	Sample Management
S-GB-C-008	03	Measurement of Percent Moisture in Soils and Solids	19-Dec-06	5-Oct-12	Sample Management
S-GB-C-009	02	Subcontracting Samples		8-Oct-12	Sample Management
S-GB-C-010	04	Sample Management	12-Jul-04	10-Dec-12	Sample Management
S-GB-C-011	01	Bottle Preparation	4-Oct-06	5-Oct-12	Sample Management
S-GB-C-012	01	Review of Analytical Requests	23-Dec-08	26-Sep-12	Sample Management
METALS					
S-GB-M-001	02	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry by 1631	1-Dec-06	6-Jul-11	Metals
S-GB-M-004	04	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy (7470A/7471A_B)	28-Aug-06	19-Dec-12	Metals
S-GB-M-005	05	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010C	23-Sep-08	19-Dec-12	Metals
S-GB-M-006	04	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020A	25-Sep-08	5-Dec-11	Metals
S-GB-M-007	02	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020	13-May-10	5-Dec-11	Metals
S-GB-M-008	02	Cleaning Metals Glassware	5-May-05	21-Sep-12	Metals
S-GB-M-009	02	Hardness by Calculation	27-Oct-05	28-Sep-11	Metals
S-GB-M-011	02	The Determination of Mercury in Biological Samples by Cold Vapor Atomic Absorption Spectroscopy (245.6)	28-Dec-06	5-Dec-11	Metals
S-GB-M-013	01	Soil Fraction Preparation for Lead Analysis	21-Mar-06	6-Jul-11	Metals
S-GB-M-014	03	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010B	28-Feb-07	19-Dec-12	Metals
S-GB-M-015	02	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry (1631E)- Analytik Jena	16-Aug-11	19-Dec-12	Metals
S-GB-M-016	00	Determination of Mercury in Solids by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry	21-Mar-12	21-Mar-12	Metals

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-M-017	00	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy - CETAC M-7500 (7470A/7471A_B)	3-Aug-11	8-Aug-11	Metals
MICRO					
S-GB-MB-001	00	Fecal Coliform Determination Using the Membrane Filter Technique	13-Sep-05	5-Oct-12	Wetchem
S-GB-MB-002	00	Heterotropic Plate Count	17-Nov-05	5-Oct-12	Wetchem
S-GB-MB-003	00	Colisure Presence/Absence Test for Detection and Identification of Coliform Bacteria and *Escherichia coli* in Drinking Waters	28-Nov-05	5-Oct-12	Wetchem
WET CHEM					
S-GB-I-001	08	Total Sulfide, Iodometric Titration	26-Jan-05	16-Nov-12	Wetchem
S-GB-I-002	06	Flash Point (Pensky-Martens Closed Cup Method For Ignitability	27-Jan-05	3-Dec-12	Wetchem
S-GB-I-009	04	Ion Chromatography- Sulfate, Nitrate, Flouride (EPA 300.0)	7-Feb-05	28-Sep-12	Wetchem
S-GB-I-010	03	Wet Chemistry Glassware Cleaning	5-Feb-05	21-Sep-12	Wetchem
S-GB-I-013	03	Free Liquids (Paint Filter)	13-Jul-05	21-Sep-12	Wetchem
S-GB-I-015	03	Oxidation - Reduction Potential (Eh) Measurement	29-Jul-05	28-Sep-12	Wetchem
S-GB-I-016	03	Specific Gravity	25-Aug-05	28-Sep-12	Wetchem
S-GB-I-017	03	Ferrous Iron	30-Aug-05	28-Sep-12	Wetchem
S-GB-I-020	04	Color Determination in Aqueous Samples	15-Sep-05	28-Sep-12	Wetchem
S-GB-I-027	03	Dissolved Oxygen	1-Mar-06	28-Sep-12	Wetchem
S-GB-I-030	03	Turbidity (Nephelometric)	10-Apr-06	3-Dec-12	Wetchem
S-GB-I-037	04	The Determination of Total Organic Carbon Using the Walkley-Black Procedure	8-Aug-05	21-Sep-12	Wetchem
S-GB-I-044	03	Biochemical Oxygen Demand	15-Feb-07	28-Sep-12	Wetchem
S-GB-I-045	04	Chromium, Hexavalent-Colorimetric	15-Feb-07	3-Dec-12	Wetchem
S-GB-I-047	03	Total Kjeldahl Nitrogen using Block Digestion and Analyzed by Lachat 8000 Flow Injection following EPA Method 351.2	17-Feb-07	7-Oct-11	Wetchem
S-GB-I-048	02	Ammonia using Micro-Distillation and Analyzed by Lachat 8000 Flow Injection following EPA Method 350.1	17-Feb-07	26-Aug-11	Wetchem
S-GB-I-051	02	Nitrate and Nitrite Analyzed by Lachat 8000 Flow Injection	20-Feb-07	26-Aug-11	Wetchem
S-GB-I-052	04	Chemical Oxygen Demand, Colorimetric, Manual (Chemetric Vials)	21-Feb-07	28-Sep-12	Wetchem
S-GB-I-053	01	Acid Volatile Sulfide / Simultaneously Extracted Metals	4-Apr-07	16-Nov-09	Wetchem
S-GB-I-059	04	The Determination of Total Organic Carbon Using the Apollo 9000 Instrument	21-May-09	28-Sep-12	Wetchem
S-GB-I-061	02	Total Phosphorus using Block Digestion and Analyzed by SmartChem	1-Jun-09	30-Oct-12	Wetchem
S-GB-I-063	02	The Determination of Total Organic Carbon Using the Teledyne Tekmar Fusion Instrument	28-Dec-09	28-Sep-12	Wetchem
S-GB-I-064	01	Cyanide by SmartChem	18-Jan-10	28-Sep-12	Wetchem

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-I-066	01	Measurement of Specific Conductance in Water	12-Dec-06	7-Dec-12	Wetchem
S-GB-I-067	01	TCLP - Toxicity Characteristic Leaching Procedure, SPLP - Synthetic Precipitation Leaching Procedure and ASTM - ASTM D 3987-85	5-Jul-10	28-Sep-12	Wetchem
S-GB-I-068	01	Measurement of Volatile Solids and Solids in Water	24-Aug-07	3-Dec-12	Wetchem
S-GB-I-069	01	Alkalinity by Titration using the Radiometer TIM840 - SM 2320B	17-Dec-10	24-Aug-11	Wetchem
S-GB-I-070	01	Alkalinity by SmartChem (EPA 310.2)	19-Apr-11	24-Sep-12	Wetchem
S-GB-I-071	01	Measurement of pH in Water, Soil, and Waste	12-Dec-06	28-Sep-12	Wetchem
S-GB-I-072	01	Acidity by Titration using the Radiometer TIM840 - SM 2310B_305.1	1-May-12	16-Nov-12	Wetchem
S-GB-I-073	00	The Determination of Total Organic Carbon Using the EA4000 Instrument (EPA 9060A)	20-Sep-12	28-Sep-12	Wetchem
GAS SECTION					
S-GB-O-005	04	Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO	29-Apr-05	16-Nov-12	Gas
S-GB-O-006	04	Modified Method for Determination of Gasoline Range Organics	27-Apr-05	19-Nov-12	Gas
S-GB-O-008	05	Total Petroleum Hydrocarbons - Gasoline by Gas Chromatography Using Flame-ionization Detectors	15-Jun-05	10-Dec-12	Gas
S-GB-O-009	04	Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors	27-Apr-05	3-Dec-12	Gas
S-GB-O-010	04	Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics	29-Apr-05	30-Oct-12	Gas
S-GB-O-017	04	Analysis of Dissolved Methane, Ethane, and Ethene in Ground Water by Static Headspace and Gas Chromatography	27-Apr-05	3-Dec-12	Gas
SVOA SECTION					
S-ALL-O-038	01	Processing Tentatively Identified Compounds (TICs) for GC/MS	9-Jun-11	3-Dec-12	QA
S-GB-O-015	04	Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics	11-May-05	28-Sep-11	SVOA
S-GB-O-018	03	Determination of DRO Sample Weight and Methylene Chloride Addition	13-Apr-05	21-Sep-12	SVOA
S-GB-O-019	04	WI Modified Method for Determination of Diesel Range Organics	13-Apr-05	13-Jul-11	SVOA
S-GB-O-023	05	Total Petroleum Hydrocarbons - Diesel	25-May-05	7-Jul-11	SVOA
S-GB-O-025	03	Alcohols & Glycols by Direct Injection GC/FID	14-Dec-05	1-May-12	SVOA
S-GB-O-026	06	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by 8082	23-May-06	1-May-12	SVOA
S-GB-O-027	05	Analysis of Organochlorine Pesticides by Gas Chromatography by 8081	23-May-06	13-Jul-11	SVOA
S-GB-O-028	04	Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes	24-May-06	21-Sep-12	SVOA

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-O-031	03	Extraction of Biological Samples for Organochlorine Pesticides/PCBs	20-Jan-05	21-Sep-12	SVOA
S-GB-O-032	03	Gel Permeation Chromatography	26-Jan-05	21-Sep-12	SVOA
S-GB-O-033	02	Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis	8-Feb-05	21-Sep-12	SVOA
S-GB-O-034	03	Sulfuric Acid Cleanup	3-Feb-05	7-Jul-11	SVOA
S-GB-O-036	03	Florisil Cleanup for PCB and Toxaphene Samples	26-Jan-05	7-Jul-11	SVOA
S-GB-O-037	03	Florisil Cartridge Cleanup for Organochlorine Pesticide Samples	26-Jan-05	28-Sep-11	SVOA
S-GB-O-038	04	Silica Gel Cleanup for Organic Analysis	3-Feb-05	21-Sep-12	SVOA
S-GB-O-039	03	Copper Cleanup for the Removal of Sulfur from PCB and Toxaphene Samples	23-Aug-05	7-Jul-11	SVOA
S-GB-O-040	04	Extraction of Wipes and Oil for PCB Analysis	11-Jul-08	1-May-12	SVOA
S-GB-O-041	04	Extraction of PCBs Using the Automated Soxhlet 3541	23-Aug-07	1-May-12	SVOA
S-GB-O-043	03	Extraction of Toxaphene Using the Automated Soxhlet 3541	3-May-07	21-Sep-12	SVOA
S-GB-O-044	02	Determination of Low Level PAHs by GC/MS-SIM in Solid and Biological Matrices:8270SIMPAH	5-Feb-05	13-Jul-11	SVOA
S-GB-O-045	05	Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons, Base/Neutral/Acids, and Total Petroleum Hydrocarbons in Solid Matrices by 3546	21-Sep-07	6-Feb-13	SVOA
S-GB-O-047	03	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography following 8082A	22-Sep-08	21-Sep-12	SVOA
S-GB-O-048	02	Analysis of Fox River Polychlorinated Biphenyls (PCBs) by Gas Chromatography	4-Jun-09	13-Jul-11	SVOA
S-GB-O-049	04	Determination of Semi-Volatile Organics by GC/MS (8270)	28-Nov-06	26-Sep-12	SVOA
S-GB-O-050	02	Determination of Semi-Volatile Organics by gc/ms (Selective Ion Monitoring)	28-Nov-06	3-May-12	SVOA
S-GB-O-052	01	Extraction of PCBs In Tissue Using the Automated Soxhlet	11-Nov-09	1-May-12	SVOA
S-GB-O-053	02	Separatory Funnel Extraction by 3510C	16-Nov-06	3-May-12	SVOA
S-GB-O-054	01	Ultrasonic Extraction by 3550B	16-Nov-06	1-May-12	SVOA
S-GB-O-055	01	WI Modified Method for Determination of Extended Range Diesel Range Organics	12-Jul-11	1-May-12	SVOA
S-GB-O-057	01	Analysis of Toxaphene by Gas Chromotography Using St.John's River Waste Management Department (SJRWMD) Protocol	10-Jul-10	26-Sep-12	SVOA
S-GB-O-058	01	Analysis of Toxaphene by Gas Chromotography Using Hercules 97 and Total Area Under the Curve (TAUC)	7-Jul-10	26-Sep-12	SVOA
S-GB-O-060	00	Extraction of TPH-Diesel Using the Automated Soxhlet_3541	7-Jul-11	7-Jul-11	SVOA
S-GB-O-062	00	SVOA Sample Screening	6-Feb-13	6-Feb-13	SVOA
VOA SECTION					
S-GB-O-001	06	Sample Screening Volatile Organics Prior to Preparation	24-Aug-05	14-Nov-11	VOA

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-O-012	04	Cleaning of Syringes Used in the Analysis of Volatile Organics	12-Apr-05	17-Oct-12	VOA
S-GB-O-056	05	Determination of Volatile Organics by GC/MS 8260	14-Jul-06	30-Dec-11	
IT					
S-GB-IT-001	00	System Security and Integrity	26-Jan-05	28-Sep-11	IT
S-GB-IT-002	00	Server Back-up	4-Jun-09	30-Oct-12	IT
QA SYSTEM					
S-ALL-Q-003	07	Document Numbering	12-Apr-04	29-Nov-12	QA
S-ALL-Q-007	03	EPIC Pro: Acode Validation	15-Feb-05	24-Aug-11	QA
S-ALL-Q-008	02	EPIC Pro: Acode Addition/Modification	7-Jun-10	13-Jul-11	QA
S-ALL-Q-009	05	Laboratory Documentation	22-Dec-04	29-Nov-12	QA
S-ALL-Q-014	02	Quality system Review	5-May-04	12-Sep-12	QA
S-ALL-Q-015	00	Review of Laboratory Management System	12-Sep-12	12-Sep-12	QA
S-ALL-Q-020	05	Orientation and Training Procedures	30-Jun-05	5-Oct-12	QA
S-ALL-Q-022	03	3P Program Continuous Process Improvement	19-May-05	12-Sep-12	QA
S-ALL-Q-028	02	Use and Operation of Lab Track System	22-May-07	5-Oct-12	QA
S-ALL-Q-029	03	MintMiner Data File Review	20-Nov-07	19-Dec-12	QA
S-ALL-Q-030	04	Operation of Data Checker for Epic Pro	5-Aug-10	21-Mar-12	QA
S-ALL-Q-034	01	Anonymous Hotline Procedure	5-Aug-10	24-Aug-11	QA
S-ALL-Q-035	01	Data Recall	31-Jan-11	21-Sep-12	QA
S-ALL-Q-037	01	Data Review Process	9-Feb-11	11-Apr-11	QA
S-GB-Q-001	03	Employee Master Signature Log	10-Jan-06	14-Nov-11	QA
S-GB-Q-002	03	Training Record Files Maintained by the QAO	10-Jan-06	14-Nov-11	QA
S-GB-Q-003	02	Data Reduction, Validation, and Reporting	5-Jan-09	7-Oct-11	QA
S-GB-Q-004	03	Laboratory Notebooks and Logbooks	17-Jan-06	14-Nov-11	QA
S-GB-Q-005	03	Precision and Accuracy Measurement and Evaluation	23-Mar-06	14-Nov-11	QA
S-GB-Q-006	03	Data Archiving	27-Apr-06	14-Nov-11	QA
S-GB-Q-007	04	Method of Syringe Technique	9-May-06	14-Nov-11	QA
S-GB-Q-008	04	Preventative, routine, and non-routine maintenance	9-May-06	30-Dec-11	QA
S-GB-Q-009	04	Common Laboratory Calculations and Statistical Evaluation of Data	11-May-06	29-Nov-12	QA
S-GB-Q-010	04	Estimation of Measurement Uncertainty	15-Feb-07	25-Jul-12	QA
S-GB-Q-011	01	Method Validation and Modification Studies	7-Jun-10	25-Jul-12	QA
S-GB-Q-012	02	Purchasing of Laboratory Supplies	3-Apr-06	30-Oct-12	QA
S-GB-Q-013	02	Receipt and Storage of Laboratory Supplies	21-Mar-06	21-Sep-12	QA
S-GB-Q-016	01	Management of Change	11-May-11	25-Jul-12	QA
S-GB-Q-017	01	Preparation of SOPs	5-May-04	21-Sep-12	QA
S-GB-Q-018	01	Evaluation and Qualification of Vendors	10-Apr-08	25-Jul-12	QA
S-GB-Q-019	01	Software Validation	9-Jun-09	25-Jul-12	QA
S-GB-Q-020	01	Determination of LOD and LOQ	21-Feb-05	25-Jul-12	QA
S-GB-Q-021	01	Proficiency Testing Program	15-Feb-07	21-Sep-12	QA
S-GB-Q-022	01	Internal and External Audits	30-Dec-04	26-Sep-12	QA
S-GB-Q-023	01	MCL Violation Reporting	11-May-09	26-Sep-12	QA
S-GB-Q-024	01	Manual Integration	15-Mar-05	29-Nov-12	QA
S-GB-Q-025	01	Sample Homogenization and Sub-Sampling	16-Nov-06	29-Nov-12	QA

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-Q-026	01	Standard and Reagent Management and Traceability	9-Feb-11	29-Nov-12	QA
S-GB-Q-027	00	Corrective Action / Preventative Action Process	17-Aug-05	21-Mar-12	QA
S-GB-Q-028	01	Monitoring Storage Units	20-Apr-05	10-Oct-12	QA
S-GB-Q-029	00	Document Management	30-Dec-04	30-Dec-11	QA
S-GB-Q-030	00	Support Equipment	17-Jan-05	30-Dec-11	QA
S-GB-Q-031	00	Control Charting and Trend Analysis	9-Feb-11	21-Mar-12	QA
Safety					
S-ALL-S-001	04	Hazard Assessment	1-Jan-05	5-Oct-12	Safety
S-GB-S-001	05	Regulated Soil Handling	5-Jan-06	12-Sep-12	Safety
S-GB-S-002	03	Control of Hazardous Energy Program - Lockout/Tagout	10-Jan-06	21-Sep-12	Safety
S-GB-S-004	03	Rescue Alert System Operation	16-Nov-05	21-Sep-12	Safety
S-GB-S-007	00	Label Destruction	3-Sep-10	14-Nov-11	Safety
S-GB-S-008	00	Air Quality Monitoring and Fume Hood Monitoring	4-Dec-12	10-Dec-12	Safety
Waste					
S-GB-W-001	01	Waste Handling and Management	29-Jul-05	10-Dec-12	Waste
S-GB-W-002	00	Waste Management Training Requirements	11-Feb-09	21-Sep-12	Safety
Equipment					
S-GB-E-001	02	Use and Maintenance of the NANOpure Infinity Water Purification System	28-Mar-06	27-Sep-11	Equipment
S-GB-E-002	02	Operation of Waste Disposal Equipment	21-Sep-07	27-Sep-11	Equipment
Training					
S-ALL-T-002	04	Learn Center	25-Apr-11	16-Nov-12	Training
Laboratory					
S-GB-L-001	06	Biological Tissue, Plant, and Synthetic Material Preparation	20-Jan-05	9-Sep-11	Laboratory
S-GB-L-002	03	Small Rodent Handling and Homogenization	20-Jan-05	28-Sep-12	Laboratory
S-GB-L-003	04	The Determination of Lipids in Tissues, Fats, and Plants	20-Jan-05	9-Sep-11	Laboratory
S-GB-L-004	03	Determination of Percent Solids in Tissue Samples	3-Feb-05	9-Sep-11	Laboratory
S-GB-L-005	01	Reagent Water Quality	10-Jan-08	28-Sep-11	Laboratory
S-GB-L-007	00	Cleaning of Equipment Used in the Process of Homogenizing Biological Tissue, Plant, and Synthetic Materials.	19-Jul-11	9-Sep-11	Laboratory

ATTACHMENT VI- LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE) SCOPE AND APPLICATION CERTIFICATES ARE MAINTAINED AND FILED IN THE LOCAL QUALITY DEPARTMENT

Green Bay Laboratory

Accrediting Authority	Program Category	Accrediting Agency	Certification #	Expiration Date
Florida (NELAP)	Biological Tissue	Dept of Health, Bureau of Laboratories	E87948	6/30/2013
Florida (NELAP)	Hazardous Waste - Solid	Dept of Health, Bureau of Laboratories	E87948	6/30/2013
Florida (NELAP)	Waste Water	Dept of Health, Bureau of Laboratories	E87948	6/30/2013
Georgia	Waste Water - NELAP stipulation	Environmental Protection Divison	E87948	6/30/2013
Georgia	Waste Water - NELAP stipulation	Environmental Protection Divison	E87948	6/30/2013
Illinois (NELAP)	Hazardous Waste - Solid	Illinois EPA	200050	8/3/2013
Illinois (NELAP)	Waste Water	Illinois EPA	200050	8/3/2013
Kentucky	UST	Environmental and Public Protection Cabinet	82	8/26/2013
Louisiana (NELAP)	Hazardous Waste - Solid	Department of Environmental Quality	4168	6/30/2013
Louisiana (NELAP)	Waste Water	Department of Environmental Quality	4168	6/30/2013
Louisiana (NELAP)	Biological Tissue	Department of Environmental Quality	4168	6/30/2013
Minnesota	Hazardous Waste	Dept of Health	055-999-334	12/31/2013
Minnesota	Waste Water	Dept of Health	055-999-344	12/31/2013
Minnesota	UST	Department of Health	055-999-344	12/31/2013
New York (NELAP)	Solid - Hazardous Waste	Dept of Health	11887	4/1/2013
New York (NELAP)	Solid - Hazardous Waste	Dept of Health	11888	4/1/2013
New York (NELAP)	Waste Water	Dept of Health	11888	4/1/2013
New York (NELAP)	Waste Water	Dept of Health	11887	4/1/2013
North Dakota	Hazardous Waste	Dept of Health Chemistry Division	R-150	6/30/2013
North Dakota	Waste Water	Dept of Health Chemistry Division	R-150	6/30/2013
South Carolina	Hazardous Waste	Dept of Health & Environmental Control	83006001	8/31/2013
South Carolina	Waste Water	Dept of Health & Environmental Control	83006001	8/31/2013
US Dept of Agriculture	Foreign Soil Permit	Dept of Agriculture	S-76505	4/1/2014
Wisconsin	Drinking Water	Dept of Natural Resources	405132750	8/31/2013
Wisconsin	Hazardous Waste	Dept of Natural Resources	405132750	8/31/2013
Wisconsin	Waste Water	Dept of Natural Resources	405132750	8/31/2013
Wisconsin	Drinking Water	Dept of Agriculture, Trade & Consumer Protection	105-444	12/31/2013

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

CHAIN-OF-CUSTODY / Analytical Request Document

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

Pace Analytical™ www.pacelabs.com

Page: of

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

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

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

REGULATORY AGENCY
☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER
☐ UST ☐ RCRA ☐ OTHER

Site Location
STATE:

Section D Required Client Information

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

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

ITEM #
MATRIX CODE (see valid codes to left)
SAMPLE TYPE (G=GRAB C=COMP)
COLLECTED: COMPOSITE START DATE / TIME; COMPOSITE END/GRAB DATE / TIME
SAMPLE TEMP AT COLLECTION
OF CONTAINERS
Preservatives: Unpreserved, H_2SO_4, HNO_3, HCl, NaOH, $Na_2S_2O_3$, Methanol, Other
Analysis Test (Y/N)
Requested Analysis Filtered (Y/N)
Residual Chlorine (Y/N)
Pace Project No./ Lab I.D.

1 2 3 4 5 6 7 8 9 10 11 12

ADDITIONAL COMMENTS
RELINQUISHED BY / AFFILIATION — DATE — TIME
ACCEPTED BY / AFFILIATION — DATE — TIME
SAMPLE CONDITIONS: Temp in °C; Received on Ice (Y/N); Custody Sealed Cooler (Y/N); Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE
PRINT Name of SAMPLER:
SIGNATURE of SAMPLER:
DATE Signed (MM/DD/YY):

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

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

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acidity	SM2310B	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Actinides	HASL-300	Water		pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid		None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Alkylated PAHs		Water		$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid		$\leq 10^oC$	1 Year/40 Days
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid		None	180 days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	$\leq 6^oC$; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	$\leq 6^oC$	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	$\leq 6^oC$	28 days
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	$\leq 6^oC$	14 Days
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	$\leq 6^oC$; $Na_2S_2O_3$	24 Hours

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Base/Neutrals and Acids	8270	Solid	8oz Glass	≤ 6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Biomarkers		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6°C	48 hours
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	14 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	48 Hours
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil		
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 6°C	6 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 6°C	6 Hours
Coliform, Total and Escherichla (E. coli)	SM9223B	Water	100mL Plastic	≤ 10°C	48 Hours after collection; results from samples analyzed 30-48 Hours after collection must be qualified as analyzed >30 hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	24 Hours
Condensable Particulate	EPA 202	Air	Solutions	None	180 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Emissions					
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present-applies to SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6°C	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	30/45 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Ferrous Iron	SN3500Fe-D	Water	Glass	None	Immediate
Flashpoint/Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics-Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics-Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6°C	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	≤ 6°C; packed jars with no headspace	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	≤ 6°C in MeOH	21 Days
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤ 6°C	14/7 Days if extracts stored ≤ 6°C or 14/14 Days if extracts stored at ≤ -10°C
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Heterotrophic Plate Count (MPC)	SM9215B	Water	100mL Plastic	≤ 6°C	24 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/SM3500Cr-C,D	Water	Plastic/Glass	≤ 6°C	24 Hours
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6°C	24 Hours after extraction
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Lipids	Pace Lipids	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid			
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	≤ 6°C	28 days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	≤ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
Methanol, Ethanol	8015 modified	Water	40mL vials	≤ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	≤ 6°C	14 Days
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	48 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass		Pest: 7/40 Days; PCB: 1 Year/1 Year
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	≤ -10°C	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; ≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6°C	1 Year/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/1 Year
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Oil Range Organics- ORO					
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	14 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; ≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid			
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days
Saturated Hydrocarbons		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days	≤ 6°C; pH<2 1:1 HCl (optional)

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				unpreserved	
Saturated Hydrocarbons		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/ 9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤ 6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black	Solid	Glass	≤ 6°C	14 Days
Total Organic Halogen (TOX)	SM5320/9020/9021	Water	Glass; no headspace	≤ 6°C	14 Days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HCl	180 days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C;	7/28 Days

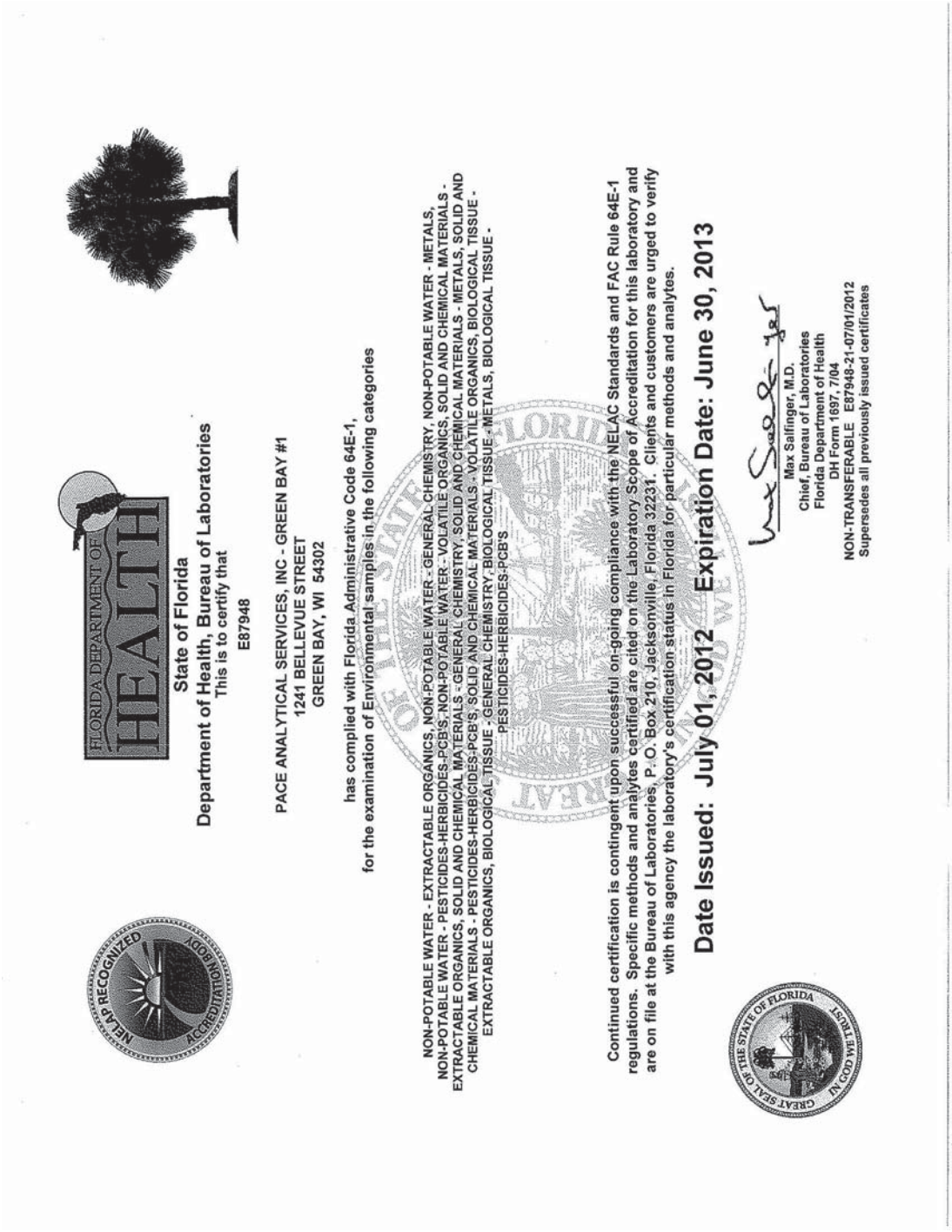
Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Hydrocarbons (aliphatic and aromatic)				packed jars with no headspace	
Volatiles	TO-14	Air	Summa Canister	None	30 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	48 Hours
Volatiles	TO-15	Air	Summa Canister	None	30 Days
Volatiles	8260	Solid	5035 vial kit	See note 1	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6°C; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days

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

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

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

ATTACHMENT IX- NELAC CERTIFICATE



FLORIDA DEPARTMENT OF HEALTH

State of Florida
Department of Health, Bureau of Laboratories
This is to certify that
E87948

PACE ANALYTICAL SERVICES, INC - GREEN BAY #1
1241 BELLEVUE STREET
GREEN BAY, WI 54302

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS, BIOLOGICAL TISSUE - EXTRACTABLE ORGANICS, BIOLOGICAL TISSUE - GENERAL CHEMISTRY, BIOLOGICAL TISSUE - METALS, BIOLOGICAL TISSUE - PESTICIDES-HERBICIDES-PCB'S

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P.O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2012 Expiration Date: June 30, 2013

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E87948-21-07/01/2012
Supersedes all previously issued certificates

NELAP RECOGNIZED ACCREDITATION BODY

GREAT SEAL OF THE STATE OF FLORIDA IN GOD WE TRUST

Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	3/19/2012
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2,4-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3,5-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2012** **Expiration Date: 6/30/2013**

Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Page 2 of 24

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Hexanone	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	4/1/2005
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 625	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acetone	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetophenone	EPA 625	Extractable Organics	NELAP	4/1/2005
Acetophenone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acidity, as CaCO3	EPA 305.1	General Chemistry	NELAP	4/1/2005
Acidity, as CaCO3	SM 2310 B (4a)	General Chemistry	NELAP	5/22/2007
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	5/15/2009
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 624	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

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Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	4/1/2005
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6010	Metals	NELAP	4/1/2005
Aluminum	EPA 6020	Metals	NELAP	4/1/2005
Ammonia as N	EPA 350.1	General Chemistry	NELAP	4/1/2005
Aniline	EPA 625	Extractable Organics	NELAP	4/1/2005
Aniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Antimony	EPA 6010	Metals	NELAP	4/1/2005
Antimony	EPA 6020	Metals	NELAP	4/1/2005
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6010	Metals	NELAP	4/1/2005
Arsenic	EPA 6020	Metals	NELAP	4/1/2005
Barium	EPA 6010	Metals	NELAP	4/1/2005
Barium	EPA 6020	Metals	NELAP	4/1/2005
Benzene	EPA 602	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Benzidine	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005

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Rick Scott
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FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzoic acid	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Beryllium	EPA 6010	Metals	NELAP	4/1/2005
Beryllium	EPA 6020	Metals	NELAP	4/1/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	4/1/2005
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/1/2005
Boron	EPA 6010	Metals	NELAP	4/1/2005
Boron	EPA 6020	Metals	NELAP	4/1/2005
Bromide	EPA 300.0	General Chemistry	NELAP	4/1/2005
Bromide	EPA 9056	General Chemistry	NELAP	4/1/2005
Bromobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 624	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Cadmium	EPA 6010	Metals	NELAP	4/1/2005
Cadmium	EPA 6020	Metals	NELAP	4/1/2005
Calcium	EPA 6010	Metals	NELAP	4/1/2005
Calcium	EPA 6020	Metals	NELAP	4/1/2005

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Rick Scott
Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbazole	EPA 8270	Extractable Organics	NELAP	4/1/2005
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	4/1/2005
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	4/1/2005
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chloride	EPA 300.0	General Chemistry	NELAP	4/1/2005
Chloride	EPA 9056	General Chemistry	NELAP	4/1/2005
Chlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 624	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chromium	EPA 6010	Metals	NELAP	4/1/2005
Chromium	EPA 6020	Metals	NELAP	4/1/2005
Chromium VI	SM 3500-Cr B (20th/21st Ed.)/UV-VIS	General Chemistry	NELAP	5/30/2007
Chrysene	EPA 625	Extractable Organics	NELAP	4/1/2005
Chrysene	EPA 8270	Extractable Organics	NELAP	4/1/2005
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Cobalt	EPA 6010	Metals	NELAP	4/1/2005
Cobalt	EPA 6020	Metals	NELAP	4/1/2005
Conductivity	EPA 120.1	General Chemistry	NELAP	4/1/2005
Copper	EPA 6010	Metals	NELAP	4/1/2005
Copper	EPA 6020	Metals	NELAP	4/1/2005
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibromomethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/1/2005
Diethyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Ethanol	EPA 8015	Volatile Organics	NELAP	5/15/2009
Ethanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 602	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 625	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluoride	EPA 300.0	General Chemistry	NELAP	4/1/2005
Fluoride	EPA 9056	General Chemistry	NELAP	4/1/2005
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	5/15/2009
Gasoline range organics (GRO)	WI-GRO	Volatile Organics	NELAP	5/15/2009
Hardness	SM 2340 B	General Chemistry	NELAP	4/1/2005
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/1/2005
Ignitability	EPA 1010	General Chemistry	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Iron	EPA 6010	Metals	NELAP	4/1/2005
Iron	EPA 6020	Metals	NELAP	4/1/2005
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isophorone	EPA 625	Extractable Organics	NELAP	4/1/2005
Isophorone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Isopropyl alcohol (2-Propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	4/1/2005
Lead	EPA 6010	Metals	NELAP	4/1/2005
Lead	EPA 6020	Metals	NELAP	4/1/2005
Lithium	EPA 6020	Metals	NELAP	5/15/2009
m/p-Xylenes	EPA 8021	Volatile Organics	NELAP	5/15/2009
m/p-Xylenes	EPA 8260	Volatile Organics	NELAP	5/15/2009
Magnesium	EPA 6010	Metals	NELAP	4/1/2005
Magnesium	EPA 6020	Metals	NELAP	4/1/2005
Manganese	EPA 6010	Metals	NELAP	4/1/2005
Manganese	EPA 6020	Metals	NELAP	4/1/2005

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Mercury	EPA 1631	Metals	NELAP	5/30/2007
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7470	Metals	NELAP	4/1/2005
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methanol	EPA 8015	Volatile Organics	NELAP	5/15/2009
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	5/15/2009
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6010	Metals	NELAP	4/1/2005
Molybdenum	EPA 6020	Metals	NELAP	4/1/2005
Naphthalene	EPA 625	Extractable Organics	NELAP	4/1/2005
Naphthalene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Nickel	EPA 6010	Metals	NELAP	4/1/2005
Nickel	EPA 6020	Metals	NELAP	4/1/2005
Nitrate	EPA 9056	General Chemistry	NELAP	4/1/2005
Nitrate as N	EPA 300.0	General Chemistry	NELAP	4/1/2005
Nitrite	EPA 9056	General Chemistry	NELAP	4/1/2005
Nitrite as N	EPA 300.0	General Chemistry	NELAP	4/1/2005
Nitrobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Propanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	4/1/2005
o-Xylene	EPA 8021	Volatile Organics	NELAP	5/15/2009
o-Xylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
pH	EPA 9040	General Chemistry	NELAP	4/1/2005
pH	SM 4500-H+-B	General Chemistry	NELAP	5/22/2007
Phenanthrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 625	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	4/1/2005
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Potassium	EPA 6010	Metals	NELAP	4/1/2005
Potassium	EPA 6020	Metals	NELAP	4/1/2005
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 625	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	5/30/2007
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	5/30/2007
Residue-total	SM 2540 B	General Chemistry	NELAP	5/30/2007
Residue-volatile	EPA 160.4	General Chemistry	NELAP	4/1/2005
Residue-volatile	SM 2540 E	General Chemistry	NELAP	5/30/2007
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Selenium	EPA 6010	Metals	NELAP	4/1/2005
Selenium	EPA 6020	Metals	NELAP	4/1/2005
Silicon	EPA 6020	Metals	NELAP	3/19/2012
Silver	EPA 6010	Metals	NELAP	4/1/2005
Silver	EPA 6020	Metals	NELAP	4/1/2005
Sodium	EPA 6010	Metals	NELAP	4/1/2005

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Sodium	EPA 6020	Metals	NELAP	4/1/2005
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Styrene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Sulfate	EPA 300.0	General Chemistry	NELAP	4/1/2005
Sulfate	EPA 9056	General Chemistry	NELAP	4/1/2005
Sulfide	SM 4500-S F (19th/20th/21st Ed.)/TITR	General Chemistry	NELAP	1/30/2008
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Thallium	EPA 6010	Metals	NELAP	4/1/2005
Thallium	EPA 6020	Metals	NELAP	4/1/2005
Tin	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toluene	EPA 602	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 624	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Total cyanide	EPA 335.4	General Chemistry	NELAP	4/1/2005
Total cyanide	EPA 9012	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	4/1/2005
Total organic carbon	EPA 9060	General Chemistry	NELAP	6/24/2011
Total organic carbon	SM 5310 C	General Chemistry	NELAP	6/24/2011
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	5/15/2009
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	5/15/2009
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Turbidity	SM 2130 B	General Chemistry	NELAP	4/1/2005
Uranium	EPA 6020	Metals	NELAP	3/19/2012
Vanadium	EPA 6010	Metals	NELAP	4/1/2005
Vanadium	EPA 6020	Metals	NELAP	4/1/2005
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 602	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 624	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Zinc	EPA 6010	Metals	NELAP	4/1/2005
Zinc	EPA 6020	Metals	NELAP	4/1/2005
Zirconium	EPA 6020	Metals	NELAP	3/19/2012

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Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	3/19/2012
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2,4-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3,5-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Hexanone	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	4/1/2005
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acetone	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetophenone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6010	Metals	NELAP	4/1/2005
Aluminum	EPA 6020	Metals	NELAP	4/1/2005
Ammonia as N	EPA 350.1	General Chemistry	NELAP	4/1/2005
Aniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Antimony	EPA 6010	Metals	NELAP	4/1/2005
Antimony	EPA 6020	Metals	NELAP	4/1/2005
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6010	Metals	NELAP	4/1/2005
Arsenic	EPA 6020	Metals	NELAP	4/1/2005
Barium	EPA 6010	Metals	NELAP	4/1/2005
Barium	EPA 6020	Metals	NELAP	4/1/2005
Benzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Benzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzoic acid	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Beryllium	EPA 6010	Metals	NELAP	4/1/2005
Beryllium	EPA 6020	Metals	NELAP	4/1/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Laboratory Scope of Accreditation

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State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/1/2005
Boron	EPA 6010	Metals	NELAP	4/1/2005
Boron	EPA 6020	Metals	NELAP	4/1/2005
Bromobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Cadmium	EPA 6010	Metals	NELAP	4/1/2005
Cadmium	EPA 6020	Metals	NELAP	4/1/2005
Calcium	EPA 6010	Metals	NELAP	4/1/2005
Calcium	EPA 6020	Metals	NELAP	4/1/2005
Carbazole	EPA 8270	Extractable Organics	NELAP	4/1/2005
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chromium	EPA 6010	Metals	NELAP	4/1/2005
Chromium	EPA 6020	Metals	NELAP	4/1/2005
Chrysene	EPA 8270	Extractable Organics	NELAP	4/1/2005
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Cobalt	EPA 6010	Metals	NELAP	4/1/2005
Cobalt	EPA 6020	Metals	NELAP	4/1/2005
Copper	EPA 6010	Metals	NELAP	4/1/2005
Copper	EPA 6020	Metals	NELAP	4/1/2005
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	5/15/2009

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/1/2005
Diethyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Ethanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 8270	Extractable Organics	NELAP	4/1/2005
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	5/15/2009
Gasoline range organics (GRO)	WI-GRO	Extractable Organics	NELAP	5/15/2009
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/1/2005
Ignitability	EPA 1010	General Chemistry	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005

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State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Iron	EPA 6010	Metals	NELAP	4/1/2005
Iron	EPA 6020	Metals	NELAP	4/1/2005
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isophorone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Isopropyl alcohol (2-Propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	4/1/2005
Lead	EPA 6010	Metals	NELAP	4/1/2005
Lead	EPA 6020	Metals	NELAP	4/1/2005
Lithium	EPA 6020	Metals	NELAP	5/15/2009
Magnesium	EPA 6010	Metals	NELAP	4/1/2005
Magnesium	EPA 6020	Metals	NELAP	4/1/2005
Manganese	EPA 6010	Metals	NELAP	4/1/2005
Manganese	EPA 6020	Metals	NELAP	4/1/2005
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7471	Metals	NELAP	4/1/2005
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6010	Metals	NELAP	4/1/2005
Molybdenum	EPA 6020	Metals	NELAP	4/1/2005
Naphthalene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Nickel	EPA 6010	Metals	NELAP	4/1/2005
Nickel	EPA 6020	Metals	NELAP	4/1/2005
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005

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State Surgeon General

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-21, expiration date June 30, 2013. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Propanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	4/1/2005
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	4/1/2005
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Percent (%) moisture	ASTM D2974-87	General Chemistry	NELAP	5/15/2009
pH	EPA 9040	General Chemistry	NELAP	4/1/2005
pH	EPA 9045	General Chemistry	NELAP	4/1/2005
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phosphorus	EPA 6020	Metals	NELAP	3/19/2012
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	4/1/2005
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Potassium	EPA 6010	Metals	NELAP	4/1/2005
Potassium	EPA 6020	Metals	NELAP	4/1/2005
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 8270	Extractable Organics	NELAP	4/1/2005
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Selenium	EPA 6010	Metals	NELAP	4/1/2005
Selenium	EPA 6020	Metals	NELAP	4/1/2005
Silver	EPA 6010	Metals	NELAP	4/1/2005
Silver	EPA 6020	Metals	NELAP	4/1/2005
Sodium	EPA 6010	Metals	NELAP	4/1/2005
Sodium	EPA 6020	Metals	NELAP	4/1/2005
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Styrene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	4/1/2005
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Thallium	EPA 6010	Metals	NELAP	4/1/2005

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Thallium	EPA 6020	Metals	NELAP	4/1/2005
Tin	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toluene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Total cyanide	EPA 9012	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/1/2005
Total nitrogen	TKN + Total nitrate-nitrite	General Chemistry	NELAP	4/1/2005
Total organic carbon	EPA 9060	General Chemistry	NELAP	5/30/2007
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	4/1/2005
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Uranium	EPA 6020	Metals	NELAP	3/19/2012
Vanadium	EPA 6010	Metals	NELAP	4/1/2005
Vanadium	EPA 6020	Metals	NELAP	4/1/2005
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Zinc	EPA 6010	Metals	NELAP	4/1/2005
Zinc	EPA 6020	Metals	NELAP	4/1/2005

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Governor

FLORIDA DEPARTMENT OF HEALTH

John H. Armstrong, MD
State Surgeon General

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	5/30/2007
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	5/30/2007
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Acenaphthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6020	Metals	NELAP	5/30/2007

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Rick Scott
Governor

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John H. Armstrong, MD
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E87948 **EPA Lab Code:** WI01103 (920) 469-2436

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Antimony	EPA 6020	Metals	NELAP	5/30/2007
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6020	Metals	NELAP	5/30/2007
Barium	EPA 6020	Metals	NELAP	5/30/2007
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Beryllium	EPA 6020	Metals	NELAP	5/30/2007
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	5/30/2007
Boron	EPA 6020	Metals	NELAP	5/30/2007
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Cadmium	EPA 6020	Metals	NELAP	5/30/2007
Calcium	EPA 6020	Metals	NELAP	5/30/2007
Carbazole	EPA 8270	Extractable Organics	NELAP	5/30/2007
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chromium	EPA 6020	Metals	NELAP	5/30/2007
Chrysene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Cobalt	EPA 6020	Metals	NELAP	5/30/2007
Copper	EPA 6020	Metals	NELAP	5/30/2007
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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State Surgeon General

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Fluorene	EPA 8270	Extractable Organics	NELAP	5/30/2007
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	5/30/2007
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Iron	EPA 6020	Metals	NELAP	5/30/2007
Isophorone	EPA 8270	Extractable Organics	NELAP	5/30/2007
Lead	EPA 6020	Metals	NELAP	5/30/2007
Lithium	EPA 6020	Metals	NELAP	5/15/2009
Magnesium	EPA 6020	Metals	NELAP	5/30/2007
Manganese	EPA 6020	Metals	NELAP	5/30/2007
Mercury	EPA 245.6	General Chemistry	NELAP	5/30/2007
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7471	Metals	NELAP	5/30/2007
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6020	Metals	NELAP	5/30/2007
Naphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Nickel	EPA 6020	Metals	NELAP	5/30/2007
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007

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1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	5/30/2007
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	5/30/2007
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Phenanthrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Phenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Potassium	EPA 6020	Metals	NELAP	5/30/2007
Pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Selenium	EPA 6020	Metals	NELAP	5/30/2007
Silver	EPA 6020	Metals	NELAP	5/30/2007
Sodium	EPA 6020	Metals	NELAP	5/30/2007
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Thallium	EPA 6020	Metals	NELAP	5/30/2007
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Vanadium	EPA 6020	Metals	NELAP	5/30/2007
Zinc	EPA 6020	Metals	NELAP	5/30/2007

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Pace Analytical Services, Inc.
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920 469-2436
Fax: 920 469-8827

STANDARD OPERATING PROCEDURE

DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) SPECTROSCOPY BY 6010C

REFERENCE METHODS: SW-846 METHOD 6010C WITH 3010A AND 3050B

LOCAL SOP NUMBER:	S-GB-M-005-REV.05
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-M-005-REV.04

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/19/12

Kate Grams, Laboratory Quality Manager — Date: 12/7/12

Chad Rusch, Department Manager — Date: 12/07/2012

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 This Standard Operating Procedure (SOP) documents the procedures used by PASI – Wisconsin to determine the concentration of specific metals in environmental water, wipe, paint chips, sludges, soils, and solid samples. The laboratory utilizes the ICP (Inductively Coupled Plasma – Optical Emission Spectrometer) and bases these documented procedures on those listed in EPA SW-846 Method 6010C. Sample preparation procedures are based on SW-846 Methods 3010A and 3050B.

2. SUMMARY OF METHOD

2.1 Samples are digested, excluding filtered groundwater, by heating with appropriate acids and oxidizing agents to solubilize the target elements. Portions of the digestates (or filtered, acidified groundwater samples) are pumped into a nebulizer to produce an aerosol. The aerosol is aspirated into the torch of an Inductively Coupled argon Plasma Optical Emission Spectrometer (ICP-OES) where it is evaporated and decomposed into atoms and ions. The plasma energy causes the target atoms to become excited and, during relaxation, emit characteristic light in the visible and/or ultraviolet emissions. Each element in the sample emits photons at a discrete wavelength(s), which are specific to that element. The light emissions are separated into wavelength and order by passing through a prism and onto an Echelle grating. The signal is then read and quantified by a Charge Injection Device (CID). The intensities of the wavelengths are proportional to the quantity of the target elements that is determined through a comparison to a known concentration (a calibration curve). The signals received from the CID are digitized and relayed to the instrument computer as an analytical signal.

2.2 Background correction may be required to compensate for spectral interferences. Background is measured adjacent to analyte lines at a wavelength selected to be free of spectral interference and which reflects the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a correction would actually degrade the analytical result.

3. SCOPE AND APPLICATION

3.1 This procedure may be used to determine concentrations of trace metals in water, wipe, paint chips, sludges, soils, and solid samples. A list of applicable compounds and Practical Reporting Limits (PRL) for aqueous, solid and TCLP/SPLP/ASTM samples are shown in Table 3.1

Table 3.1 – Elements and associated PRLs for water and leachates

	Element	Wavelength (nm)	PRL Aqueous (ug/L)	PRL Solid* (mg/kg)	PRL TCLP/SPLP/ASTM (mg/L)
Ag	Silver	328.0	10.0	1.0	0.250
Al	Aluminum	396.1	500.0	50.0	2.50
As	Arsenic	189.0	20.0	2.0	0.250
B	Boron	208.9	100.0	10.0	0.500
Ba	Barium	455.4	5.0	0.50	2.50
Be	Beryllium	313.0	4.0	0.40	0.250
Ca	Calcium	317.9	200.0	20	5.000
Cd	Cadmium	228.8	5.0	0.50	0.005
Co	Cobalt	228.6	5.0	0.50	0.250
Cr	Chromium	267.7	5.0	0.50	0.250
Cu	Copper	324.7	10.0	1.0	0.250
Fe	Iron	259.9	100.0	10.0	2.50
K	Potassium	766.4	200.0	20.0	5.000
Mg	Magnesium	279.0	200.0	20.0	5.000
Mn	Manganese	257.6	5.0	0.50	0.250
Mo	Molybdenum	202.0	20.0	2.0	0.250
Na	Sodium	589.5	1000.0	100.0	5.000
Ni	Nickel	231.6	10.0	1.0	0.250
Pb	Lead	220.3	7.5	1.0	0.0075
Sb	Antimony	206.8	20.0	2.0	0.250
Se	Selenium	196.0	20.0	2.0	0.250
Sn	Tin	189.9	50.0	5.0	0.250
Sr	Stronium	407.7	5.0	0.50	0.250
Ti	Titanium	334.9	5.0	0.50	0.250
Tl	Thallium	190.8	40.0	4.0	0.250
V	Vandaium	292.4	5.0	0.50	0.250
Zn	Zinc	206.2	40.0	4.0	0.250

* Based on standard weights/volumes (0.5g/50mL). Final RLs for individual samples may vary depending in actual weights/volumes and percent solids used. Soil includes soils, sludges, paint chips, wipes, and solids. (Unit for wipes are in Total ug.)

Soils are reported on a dry weight basis.

3.2 This procedure is restricted to use by, or under the supervision of, analysts experienced in the digestion of samples for metals analysis and analysis of digestates by ICP. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.3 This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

3.4 Current Method Detection Limits can be found in current LIMS or can be provided by the Quality Department.

4. INTERFERENCES

4.1 Spectral Interferences – Overlap of emission lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light can contribute to spectral interferences. These interferences can typically be minimized by careful selection of quantitation wavelengths, interelement corrections, and background correction.

4.2 Physical Interferences –Changes in sample viscosity, surface tension, or other effects associated with sample transport and nebulization can produce significant inaccuracies, especially in samples containing high concentrations of dissolved solids and acids. Dissolved solids may build up on the nebulizer tip, altering the sample flow rate and causing instrument drift. These effects can be minimized by sample dilution or use of a specially designed high-solids nebulizer. Use of internal standards helps in recognizing sample introduction issues.

4.3 Chemical Interferences – Molecular compound formation, ionization effects, and solute vaporization effects are typically not significant with ICP determinations. If observed, they can be minimized by careful selection of plasma and spectrometer operating parameters.

4.4 Memory Interferences – Sample deposition on the nebulizer tubing, spray chamber, and plasma torch can cause apparent sample carryover. Memory interferences can be minimized by flushing the system with rinse blanks between samples. If memory interference is suspected for a sample, the sample must be re-analyzed after a sufficient rinse period.

4.5 High Salt Concentrations – High salt concentrations in sample digestates can cause signal suppression and confuse interference tests. Use of internal standards helps in recognizing signal suppression

5. SAFETY

5.1 Standards and Reagents

5.2 The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound shall be treated as a potential health hazard. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Special care shall be taken when handling the high-concentration acids and oxidizing reagents used for sample digestion. All digestions must be conducted in a properly functioning fume hood. The use of personal protective equipment (gloves, lab coats and safety glasses) is required.

5.3 Samples

5.3.1 Although sample check-in should be notified of any hazardous samples, samples shall always be considered as “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.

5.4 Equipment

5.4.1 ICP Radio Frequency (RF) Generator – The RF generator used to induce the atomic plasma produces a high energy radio emission. The electrical requirements for this equipment are substantial. The RF generator must only be serviced by those trained specifically for service and repair of the instrument.

5.4.2 ICP RF Coil – The wound coil around the top of the torch produces a high energy, oscillating radio frequency field. The field is substantial and may interfere with surrounding electronics including implanted medical devices. Those individuals with such devices (i.e. pacemakers) must stay clear of this instrument while in operation.

6. DEFINITIONS

6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

6.2 Instrumental Detection Limit (IDL) – The concentration equivalent to a signal, due to the analyte, which is equal to the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day at the same wavelength.

6.3 Instrument Check Standard – A multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. This is the same as the Continuing Calibration Verification Standard (CCV) referred to in the calibration section of this SOP.

6.4 Spectral Interference Check Solution (SIC) – A solution of selected analytes of higher concentrations used to evaluate the procedural routine for correcting known inter-element spectral interferences. This is also known as the Inter-element Correction Solution (ICSA).

6.5 Linear Detection Range – Defined as the upper limit of quantitation for an analyte. The LDR is determined as the upper range limit of an observed signal that is no more than 10% below the level extrapolated from the lower standards.

6.6 Interference Check Sample - (ICSAB) – A solution containing both interfering and analyte elements of known concentration that can be used to verify background and inter-element correction factors.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Collection, Preservation, Storage and Hold Times

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil/ Solid/ Wipe/Paint Chip/Sludge	Pre-cleaned plastic or glass containers. Refer to Pace S-ALL-Q-021, *Sub-Sampling* SOP, for obtaining representative aliquots.	No Preservation	Refrigerated at ≤°C	Up to 6 months
Total Metals –Aqueous	Pre-cleaned plastic containers with HNO_3	Nitric Acid (HNO_3) to pH<2, preserved at time of collection	Room Temperature	Up to 6 months
Dissolved Metals – Aqueous(1)	Pre-cleaned plastic containers – Filter sample at time of collection through a 0.45µm membrane filter.	Nitric Acid (HNO_3) to pH<2, filtered then preserved at time of collection.	Room Temperature	Up to 6 months
TCLP/SPLP/ASTM	Pre-cleaned plastic containers.	Filtered then preserved at time of digestion. -Refrigerate at 4±2 °C until preservation. -Nitric Acid (HNO_3) to pH<2	Refrigerate at 4±2 °C	From Field Collection to Leach Extraction: 180 Days. From Digestion to Determinative Analysis: 180 Days. (Total Elapsed Time: 360 Days).

(1) If filtration cannot be performed in the field, the sample must be taken in a pre-cleaned, unpreserved plastic container and transported to the lab as soon as possible. On arrival at the lab, the sample must be filtered and then preserved to a pH <2. The sample must sit for **24 hours** prior to preparation. This is to ensure that the sample does not have a buffering effect that raises the pH to above <2. The data is qualified if sample preservation fails to bring pH down to ≤2. (Note: TCLP Extracts are not required to sit for 24 hours prior to digestion after the addition of nitric acid.)

7.2 Preservative Check

The pH of samples must be verified to be pH<2 and documented in the Sample Receiving or Metals pH logbook prior to aliquoting for analysis. If a sample has a pH>2, additional preservative must be added to return the sample to <2. Acid volume shall not to exceed 2% of the container capacity. Once adjusted, the sample must be allowed to sit for 24 hours prior to preparation. If the sample is not able to maintain a preservation of a pH of<2, then it must be qualified as such.

8. EQUIPMENT AND SUPPLIES

8.1 Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
iCAP System	Thermo Scientific	ICAP 6500	Serial Number: 20073913
Autosampler	Cetac	ASX-520	Serial Number: 080735A 520
Data System	iTEVA	2.0.039	
Reciculator	Neslab	ThermoFlex900	Serial Number: 107271037
Analytical Balance			

8.2 General Supplies

Item	Vendor (or equivalent)	Model / ID (or equivalent)	Catalog # (or equivalent)	Description
Argon – high purity grade	PraxAir			Bulk
Autosampler Vials	Fisher Scientific	15 mL Polypropylene Test Tubes	14-375-150	
Autosampler Pump Tubing	Analytical West		PT-2180PS	red/red 1.14mm ID – Carrier
			PT-2200SAS	1.30mm ID – Waste
			PT-2080PS-F	orange/orange 0.38mm ID – Internal Standard
Digestion Vials	Environmental Express		SC499	Pre-cleaned polypropylene 70-mL screw cap vials.
Watch Glass	Environmental Express			Polypropylene, to fit 70-mL digestion vial
Heating Block	Environmental Express			Polypropylene, to fit 70-mL digestion vial
Mechanical Pipettes and Tips	Eppendorf			Or equivalent
Filter paper	Whatman		541 Filter Paper	
pH Strips				
Y Connectors	Hewlett-Packard		G1820-65106	
Duo Torch / Duo Ceramic Cone	Thermo Fisher Scientific		8423-120-51241 / 84223-120-51261	
Spray Chamber/Spray Chamber adapter	Thermo Fisher Scientific		8423-120-51411 / 8423-120-51251	
SC-FAST Probe	ESI		SC-5037-3995-150	7” Teflon/Carbon Probe 1.0mmID
SC-FAST Waste Line	ESI		SC-0323-0002-215	Waste Line
SC-FAST Valve	ESI	F7	SC-0599-1024	F7 Valve Head
	ESI		ES-2501-PPF2	
	ESI		ES-2501-PPMZ	
Peri-Pump	ESI			Fluoropolymer union, barbed
Nebulizer Line	ESI		SC-0317-0250-30	
Nuts and Ferrules	ESI		SC-0599-0116-K	For high flow 1/16” tubing
Nuts and Ferrules	ESI		SC-0599-0108-W	For high flow 1/8” tubing
Vacuum Line	ESI		SC-0321-32-215	For SC-FAST high flow valve
Sample Loop	ESI		SC-0318-15	1.5mL sample loop
Nebulizer	ESI		ES-2040	Micro Flow PFA-St nebulizer

8.3 Glassware

Glassware	Vendor / Item # / Description
Volumetric Flasks	Class A
Graduated Cylinders	Class A

9. REAGENTS AND STANDARDS

9.1 Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Nano-pure Water	ASTM Type II, or equivalent (18mOhm or higher resistivity)	Nano-pure Water
Nitric Acid (HNO_3)	Trace metals grade, Fisher Insta-analyzed, or equivalent	J.T Baker Cat #9598-34 or equivalent
Hydrochloric Acid (HCl)	Trace metals grade, Fisher Insta-analyzed, or equivalent	Fisher Cat # A508 or equivalent
Hydrogen Peroxide (H_2O_2)	30% certified reagent grade; trace metals grade if available. Stabilized solutions come with Sodium added.	Fisher Cat # H325-4 or equivalent
Acidified Reagent Water	5mL Nitric Acid and 5mL Hydrochloric Acid per 100mL Nano-pure water	

9.2 Standards Definitions, Storage, and Sources

9.2.1 Definitions

Standard	Description	Comments
Initial Calibration Standard(s)	Standards prepared from single and/or multi-element standard(s) at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range for single point calibration.	Calibration standards can be made and used for 6 months unless a standard used to make them expires earlier.
Initial Calibration Verification Standard	Standard prepared near the midpoint of the calibration range and is used to verify the accuracy of the calibration.	Must be prepared from a source independent of CCV and Calibration Solutions.
Continuing Calibration Verification Standard	Standards prepared from single and/or multi-element standards at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range. This standard verifies the accuracy of the calibration curve.	May be prepared from the same source as the calibrations standards
Reporting Limit Verification Standard (RLVS)	Must be performed after calibration and prior to samples and at the end of the analytical sequence. The recovery must be between 70 and 130% to report sample data.	NELAC requirement, a client specific requirement for certain QAPPs
Inter-element correction solution (ICSA)	A solution prepared with high concentrations of interfering elements (Al, Ca, Fe, Mg...). This is run daily at a minimum to verify the inter-element correction factors.	Al, Ca, Fe, Mg
Inter-element correction solution (ICSAB)	A solution prepared with high concentrations of interfering elements and low level concentrations of the other target elements. This is analyzed daily and immediately following the ICSA solution.	Not a 6010C requirement but may be required on a client specific basis.
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard solution contains a non-target element (yttrium (Y)) at a concentration yielding an appropriate final concentration in the sample.	Y at 5ppm with Li. Added in-line to all samples and standards.
Single Element Standards	Stock standards purchased from venders containing one element. Used for checking IECs and may be used for checking linear ranges.	Must be 99.99% pure.
Spiking Standard	This solution contains all target analytes and should not be prepared from the same standards as the calibration standards.	

9.2.2 Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	▪ Concentrated reference solution purchased directly from approved vendor	▪ Manufacturer's recommended expiration date for unopened standards.	▪ Manufacturer's recommended storage conditions ▪ When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	▪ Reference solutions prepared by dilutions of the stock solution	▪ Should be prepared as needed or at least every 6 months. The intermediate or working standard can not exceed the expiration date recommended by the manufacturer for each component used.	▪ Store at room temperature

9.3 Standard Sources

9.3.1 Standards are prepared from commercially available multi-compound stock solutions. The sources of the stock solutions, formulas for preparing dilutions and working standards, and concentrations for all compounds are presented in tables within section 9.3. All intermediate and working standards are prepared using acidified Nano-pure water and used immediately or stored for no longer than 6 months.

9.3.2 Analytical Working Standard Preparations - Depending on the volume of each solution needed, the standards are brought to volume in class A volumetric flasks with Nano-pure water unless otherwise noted.

Table 9.3.1 – Calibration Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	2 ml	10 mL HNO3 / 10 mL HCl	200 ml	1.0 ppm
	Ba	100 ppm				1.0 ppm
	Be	100 ppm				1.0 ppm
	Cd	100 ppm				1.0 ppm
	Co	100 ppm				1.0 ppm
	Cr	100 ppm				1.0 ppm
	Cu	100 ppm				1.0 ppm
	Mn	100 ppm				1.0 ppm
	Ni	100 ppm				1.0 ppm
	Pb	100 ppm				1.0 ppm
	Se	100 ppm				1.0 ppm
	Sr	100 ppm				1.0 ppm
	Tl	100 ppm				1.0 ppm
	V	100 ppm				1.0 ppm
	Zn	100 ppm				1.0 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	4 ml			2.0 ppm
	Mo	100 ppm				2.0 ppm
	Sb	100 ppm				2.0 ppm
	Sn	100 ppm				2.0 ppm
	Ti	100 ppm				2.0 ppm
	Ag	50 ppm				1.0 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	10 ml			50 ppm
	Ca	1000 ppm				50 ppm
	Fe	1000 ppm				50 ppm
	K	1000 ppm				50 ppm
	Mg	1000 ppm				50 ppm
	Na	1000 ppm				50 ppm

Table 9.3.2 – CCV Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	5.0 ml	50 mL HNO3/ 50 mL HCl	1000 ml	0.5 ppm
	Ba	100 ppm				0.5 ppm
	Be	100 ppm				0.5 ppm
	Cd	100 ppm				0.5 ppm
	Co	100 ppm				0.5 ppm
	Cr	100 ppm				0.5 ppm
	Cu	100 ppm				0.5 ppm
	Mn	100 ppm				0.5 ppm
	Ni	100 ppm				0.5 ppm
	Pb	100 ppm				0.5 ppm
	Se	100 ppm				0.5 ppm
	Sr	100 ppm				0.5 ppm
	Tl	100 ppm				0.5 ppm
	V	100 ppm				0.5 ppm
	Zn	100 ppm				0.5 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	10.0 ml			1.0 ppm
	Mo	100 ppm				1.0 ppm
	Sb	100 ppm				1.0 ppm
	Sn	100 ppm				1.0 ppm
	Ti	100 ppm				1.0 ppm
	Ag	50 ppm				0.5 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	25.0 ml			25 ppm
	Ca	1000 ppm				25 ppm
	Fe	1000 ppm				25 ppm
	K	1000 ppm				25 ppm
	Mg	1000 ppm				25 ppm
	Na	1000 ppm				25 ppm

Table 9.3.3 – ICV (Second Source) Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
IV #1 (PA-STD-1B)	As	200 ppm	0.5 ml	10 mL HNO3/ 10 mL HCl	200 ml	0.5 ppm
	Ba	200 ppm				0.5 ppm
	Be	200 ppm				0.5 ppm
	Cd	200 ppm				0.5 ppm
	Co	200 ppm				0.5 ppm
	Cr	200 ppm				0.5 ppm
	Cu	200 ppm				0.5 ppm
	Mn	200 ppm				0.5 ppm
	Ni	200 ppm				0.5 ppm
	Pb	200 ppm				0.5 ppm
	Se	200 ppm				0.5 ppm
	Sr	200 ppm				0.5 ppm
	Tl	200 ppm				0.5 ppm
	V	200 ppm				0.5 ppm
	Zn	200 ppm				0.5 ppm
IV #2 (PA-STD-2B)	B	200 ppm	1.0 ml			1.0 ppm
	Mo	200 ppm				1.0 ppm
	Sb	200 ppm				1.0 ppm
	Sn	200 ppm				1.0 ppm
	Ti	200 ppm				1.0 ppm
	Ag	100 ppm				0.5 ppm
IV #3 (PA-STD-3B)	Al	2000 ppm	2.5 ml			25 ppm
	Ca	2000 ppm				25 ppm
	Fe	2000 ppm				25 ppm
	K	2000 ppm				25 ppm
	Mg	2000 ppm				25 ppm
	Na	2000 ppm				25 ppm

Table 9.3.4 – Spiking Standard (for LCS and MS/MSD) Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)	Spike Concentration *
IV #1 (PA-STD-1B)	As	200 ppm	25 ml	12 mL HNO3	200 ml	50 ppm	500 ppb
	Ba	200 ppm				50 ppm	500 ppb
	Be	200 ppm				50 ppm	500 ppb
	Cd	200 ppm				50 ppm	500 ppb
	Co	200 ppm				50 ppm	500 ppb
	Cr	200 ppm				50 ppm	500 ppb
	Cu	200 ppm				50 ppm	500 ppb
	Mn	200 ppm				50 ppm	500 ppb
	Ni	200 ppm				50 ppm	500 ppb
	Pb	200 ppm				50 ppm	500 ppb
	Se	200 ppm				50 ppm	500 ppb
	Sr	200 ppm				50 ppm	500 ppb
	Tl	200 ppm				50 ppm	500 ppb
	V	200 ppm				50 ppm	500 ppb
	Zn	200 ppm				50 ppm	500 ppb
IV #2 (PA-STD-2B)	B	200 ppm	25 ml			50 ppm	500 ppb
	Mo	200 ppm				50 ppm	500 ppb
	Sb	200 ppm				50 ppm	500 ppb
	Sn	200 ppm				50 ppm	500 ppb
	Ti	200 ppm				50 ppm	500 ppb
	Ag	100 ppm				25 ppm	250 ppb
IV #3 (PA-STD-3B)	Al	2000 ppm	25 ml			500 ppm	5000 ppb
	Ca	2000 ppm				500 ppm	5000 ppb
	Fe	2000 ppm				500 ppm	5000 ppb
	K	2000 ppm				500 ppm	5000 ppb
	Mg	2000 ppm				500 ppm	5000 ppb
	Na	2000 ppm				500 ppm	5000 ppb

*** Use 1.0 ml of spiking std to 50 ml**

Table 9.3.5 – Low Level Check Standard (CRDL) Intermediate

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)*	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppb)
4400-100118CS01	Ag	10 ppm	10 ml	5 mL HNO3 / 5 mL HCl	100 ml	1,000 ppb
	Al	500 ppm				50,000 ppb
	As	20 ppm				2,000 ppb
	B	100 ppm				10,000 ppb
	Ba	5 ppm				500 ppb
	Be	4 ppm				400 ppb
	Ca	200 ppm				20,000 ppb
	Cd	5 ppm				500 ppb
	Co	5 ppm				500 ppb
	Cr	5 ppm				500 ppb
	Cu	10 ppm				1,000 ppb
	Fe	100 ppm				10,000 ppb
	K	1000 ppm				1000,000 ppb
	Mg	200 ppm				20,000 ppb
	Mn	5 ppm				500 ppb
	Mo	20 ppm				2,000 ppb
	Na	1000 ppm				100,000 ppb
	Ni	10 ppm				1,000 ppb
	Pb	7.5 ppm				750 ppb
	Sb	20 ppm				2,000 ppb
	Se	20 ppm				2,000 ppb
	Sn	50 ppm				5,000 ppb
	Sr	5 ppm				500 ppb
	Ti	5 ppm				500 ppb
	Tl	40 ppm				4,000 ppb
	V	5 ppm				500 ppb
	Zn	40 ppm				4,000 ppb

Table 9.3.5b – Low Level Check Standard (CRDL) Working Solution

Low Level Check Standard Intermediate	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)*	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppb)
CRDL Intermediate (In Table 9.3.5)	Ag	1,000 ppb	2 ml	10 mL HNO3 / 10 mL HCl	200 ml	10 ppb
	Al	50,000 ppb				500 ppb
	As	2,000 ppb				20 ppb
	B	10,000 ppb				100 ppb
	Ba	500 ppb				5 ppb
	Be	400 ppb				4 ppb
	Ca	20,000 ppb				200 ppb
	Cd	500 ppb				5 ppb
	Co	500 ppb				5 ppb
	Cr	500 ppb				5 ppb
	Cu	1,000 ppb				10 ppb
	Fe	10,000 ppb				100 ppb
	K	100,000 ppb				1000 ppb
	Mg	20,000 ppb				200 ppb
	Mn	500 ppb				5 ppb
	Mo	2,000 ppb				20 ppb
	Na	100,000 ppb				1000 ppb
	Ni	1,000 ppb				10 ppb
	Pb	750 ppb				7.5 ppb
	Sb	2,000 ppb				20 ppb
	Se	2,000 ppb				20 ppb
	Sn	5,000 ppb				50 ppb
	Sr	500 ppb				5 ppb
	Ti	500 ppb				5 ppb
	Tl	4,000 ppb				40 ppb
	V	500 ppb				5 ppb
	Zn	4,000 ppb				40 ppb

Table 9.3.6– Inter-element Correction Solution (ICSA) Working Dilutions and Concentrations

Pace Standard #	Vendor	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (mg/L)	Stock Concentration (µg/mL)
Single Element Al	Ultra Scientific	ICP-113	10	200	Al	500	10000
Single Element Ca	Ultra Scientific	ICP-120	10	200	Ca	500	10000
Single Element Mg	Ultra Scientific	ICP-112	10	200	Mg	500	10000
Single Element Fe	Ultra Scientific	ICP-126	4	200	Fe	200	10000

Table 9.3.6b– Inter-element Correction Solution (ICSAB) Working Dilutions and Conc. – (As needed).

Pace Standard #	Vendor	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (µg/mL)	Stock Concentration (µg/mL)
Single Element Al	Ultra Scientific	ICP-113	9.5	200	Al	475	10000
Single Element Ca	Ultra Scientific	ICP-120	9.5	200	Ca	475	10000
Single Element Mg	Ultra Scientific	ICP-112	9.5	200	Mg	475	10000
Single Element Fe	Ultra Scientific	ICP-126	3.5	200	Fe	175	10000
IV #1 (PA-STD-1B)	Inorganic Ventures	PA-STD-1B	0.5	200	As	0.5	200 ppm
					Ba	0.5	200 ppm
					Be	0.5	200 ppm
					Cd	0.5	200 ppm
					Co	0.5	200 ppm
					Cr	0.5	200 ppm
					Cu	0.5	200 ppm
					Mn	0.5	200 ppm
					Ni	0.5	200 ppm
					Pb	0.5	200 ppm
					Se	0.5	200 ppm
					Sr	0.5	200 ppm
					Tl	0.5	200 ppm
					V	0.5	200 ppm
					Zn	0.5	200 ppm
IV #2 (PA-STD-2B)	Inorganic Ventures	PA-STD-2B	1.0	200	B	1.0	200 ppm
					Mo	1.0	200 ppm
					Sb	1.0	200 ppm
					Sn	1.0	200 ppm
					Ti	1.0	200 ppm
					Ag	0.5	100 ppm
IV #3 (PA-STD-3B)	Inorganic Ventures	PA-STD-3B	2.5	200	Al	25	2000 ppm
					Ca	25	2000 ppm
					Fe	25	2000 ppm
					K	25	2000 ppm
					Mg	25	2000 ppm
					Na	25	2000 ppm
Single Element Ni	Ultra Scientific	ICP-028	0.1	200	Ni	1.0	1000 ppm

Table 9.3.7– Internal Standard Solution Working Dilutions and Concentrations

Pace Standard #	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (mg/L)	Stock Concentration (mg/L)
IS	ICP69N-1 (Accustandard)	5	1000	Yttrium	5 mg/L	1000
	ICP-103-5 (Ultra Scientific)	100	1000	Lithium	1000 mg/L	10000

10. CALIBRATION

10.1 Profile

The iCAP 6500 ICP will automatically check a carbon reference line against ambient air conditions each time the plasma is ignited to maintain wavelength accuracy.

10.2 Daily Initial Calibration

Allow the instrument 30 minutes to warm up after igniting the torch. The ICP must be calibrated each time it is set up for analysis or every 24 hours according to the manufacturer's instructions. Calibration requires analysis of a Calibration Blank and at least one level of calibration solution. Calibration standards can be made and used for 6 months unless a standard used to make them expires earlier. It would then expire on that date. Instrument standardization date and time must be recorded in the raw data. See Table 9.3.1 for details on the preparation of the Initial Calibration Standard.

10.3 Calibration Verification

10.3.1 Initial Calibration Verification (ICV) / Second Source

To assess the accuracy relative to the purity of the calibration standards, a standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as **Secondary Source Verification** or, alternatively as **Initial Calibration Verification**. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. See the most current version of SOP S-GB-Q-026 *Standard and Reagent Management and Traceability* for more information of standards. This standard must be analyzed immediately after the calibration and before any batch QC or client samples. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{ICV} - \text{TrueValue}_{ICV}] / \text{TrueValue}_{ICV} * 100$$

See Table 9.3.3 for details on the preparation of this standard. See Table 10.1 for control criteria

10.3.2 Reporting Limit Verification Standard (CRDL)

With every Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) must also be analyzed and meet established acceptance criteria. The CRDL is analyzed prior to any samples being analyzed and at the end of the analytical sequence. Additional CRDLs may be analyzed throughout the analytical sequence at the analyst's discretion. The limits for the CRDL are +/- 30% of the true concentration. See Table 9.3.5 for details on the preparation of this standard.

10.3.3 ICSA (Interference Check Standard) Verification

With every Initial Calibration, the inter-element check solution standard must be run to verify the inter-element correction factors. At a minimum, the ICSA standard must be analyzed at the beginning of the run, following the initial calibration and evaluated against the criteria in Table 10.1. If client specific requirements dictate that the ICSA must be analyzed at both the beginning and end of the analysis, the ICSA may be analyzed immediately after the completion of those specific samples and is not required to be placed at the very end of the sequence. If client specific requirements dictate that the ICSAB be run, the ICSAB must be analyzed immediately following the ICSA and be held to the criteria in Table 10.1. See Table 9.3.6 and 9.3.6b for details on preparation of these standards.

10.3.4 Continuing Calibration Verification (CCV)

As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as **Continuing Calibration Verification**. The validity of the initial calibration is checked after every 10 samples and at the end of an analytical sequence by analyzing a midpoint calibration standard (CCV).

The values obtained from the analysis of the CCV are compared to the true values and a percent difference calculated according to the formula in Section 10.3.1. The percent difference must meet the method specified criteria in Table 10.1 for the analysis to proceed. See Table 9.3.2 on the preparation of this standard.

Table 10.1 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression for elements requiring multiple calibration levels	Correlation coefficient ≥ 0.998	If not met, remake standards and recalibrate. One point calibration is standard for the Wisconsin laboratory.
Initial Calibration Verification Standard (Second Source)	Immediately after each initial calibration	≤±10% of true value The % RSD between replicate integrations of an ICV std must be <5% (min of 2 integrations).	If 4 or more replicates are used for analysis the RSD must be ≤ 3%
Calibration Blank	Immediately after each initial calibration (ICB), every 10 samples (CCB), and at the end of the analytical sequence	≤ PRL	If criteria are not met, follow the corrective actions in 10.4.2. If the concentration in the blank is outside the control limit and <1/10 the concentration in the sample, the sample result may be reported.
CRDL Standard	Immediately after verification and at the end of the analytical sequence.	Each element tested must be at least between 70-130% of the PRL value.	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.4.
ICSA	Once per ICAL, immediately after verification	Results for non-interference elements = ND ± 2x RL Recovery of interfering elements must be 80-120% of true value	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.4.5
ICSAB	Once per ICAL, immediately after ICSA if requested by the client.	Results for non-interference elements = True Value ± 20% (or 2xRL, whichever is greater)	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.4.5
Continuing Calibration Verification	After every 10 samples and at the end of analytical sequence	≤±10% of true value The % RSD between replicate integrations of a CCV std must be <5% (min of 2 integrations).	If criteria not met, follow corrective actions in 10.4.6

10.4 Calibration Corrective Actions

10.4.1 Calibration Linearity Problems

10.4.1.1 Reanalyze the original calibration standards to determine instrument consistency.
10.4.1.2 Prepare and analyze a new calibration standard to determine preparation consistency / standard integrity.
10.4.1.3 Perform instrument maintenance, document in instrument log book
10.4.1.4 Recalibrate instrument according to procedures in Section 10.

10.4.2 Calibration Blank Problems

10.4.2.1 Reanalyze the original CB standard to determine instrument consistency.
10.4.2.2 Prepare and analyze a new CB standard to determine preparation consistency / standard integrity. Note: If you run two CB, only one may be used for reporting purposes.
10.4.2.3 Perform instrument maintenance, document in instrument log book
10.4.2.4 Recalibrate instrument according to procedures in Section 10.
10.4.2.5 Reanalyze any samples not bracketed by passing CBs.
10.4.2.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing CB that exceeded criteria may be reported if 10X concentration in the blank is less than the reported result.
Samples analyzed with a bracketing CB that exceeded criteria may be reported if the sample result is a non-detect.

10.4.3 Initial Calibration Verification (Secondary Source) Problems

10.4.3.1 Reanalyze the original ICV standard to determine instrument consistency.
10.4.3.2 Prepare and analyze a new ICV standard to determine preparation consistency / standard integrity.
10.4.3.3 Perform instrument maintenance, document in instrument log book
10.4.3.4 Recalibrate instrument according to procedures in Section 10.
10.4.3.5 Reanalyze any samples not bracketed by passing ICVs.
10.4.3.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing ICV that exceeded criteria due to an increase in response may be reported if the target analytes were not detected in the samples.

10.4.4 CRDL Standard Problems

10.4.4.1 Check CAL0 – Elevated counts resulting from contamination may cause the CRDL to fail low.
10.4.4.2 Reanalyze the original CRDL standard to determine instrument consistency.
10.4.4.3 Prepare and analyze a new CRDL standard to determine preparation consistency / standard integrity.
10.4.4.4 Perform instrument maintenance, document in instrument log book
10.4.4.5 Recalibrate instrument according to procedures in Section 10.

10.4.4.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with CRDL that is outside criteria may be footnoted or the CRDL for the reported sample adjusted.

10.4.5 ICSA/ICSAB Standard Problems

10.4.5.1 Reanalyze the original **ICSA/ICSAB** standard to determine instrument consistency.
10.4.5.2 Prepare and analyze a new **ICSA/ICSAB** standard to determine preparation consistency / standard integrity.
10.4.5.3 Adjust IEC and rerun **ICSA/ICSAB.** To update IECs, follow the instruction below.
10.4.5.4 The Inter-element Correction Factors (IEC's) are used to correct for false signals (positive and negative) produced when peaks overlap in the spectra.
10.4.5.5 To set up Initial IEC, run single element standards with only background correction points in place. The standards are run at the linear range.

The following calculation is done:

$$\frac{\text{Element result (ug/L)}}{\text{Interfering element result (ug/L)}} = \text{IEC}$$

10.4.5.6 This ratio is entered in the IEC table in the method file. Type the ratio under the k1 header in the appropriate row. Enter a negative before the ratio if it is a negative interference.
10.4.5.6.1 Perform instrument maintenance, document in instrument log book
10.4.5.6.2 Recalibrate instrument according to procedures in Section 10.

10.4.6 Continuing Verification Problems

10.4.6.1 Reanalyze the original CCV standard to determine instrument consistency.
10.4.6.2 Prepare and analyze a new CCV standard to determine preparation consistency / standard integrity.
10.4.6.3 Perform instrument maintenance, document in instrument log book
10.4.6.4 Recalibrate instrument according to procedures in Section 10.
10.4.6.5 Reanalyze any samples not bracketed by passing CCVs.
10.4.6.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing CCV that exceeded criteria due to an increase in response may be reported if the target analytes were not detected in the samples.

11. PROCEDURES

11.1 Aqueous Sample Preparation for Total or Dissolved Metals by 3010A – Hot Block Digestion

11.1.1 Groundwater samples for dissolved metals may be analyzed without digestion. All other water samples must be digested.

11.1.2 Aqueous samples for total or dissolved metals (if visual precipitation) may be prepared according to EPA 3010A. Samples for dissolved metals must be filtered prior to preservation. Once preserved, samples must be allowed to sit for 24 hours prior to preparation. TCLP/SPLP/ASTM extracts do not require to sit for 24 hours prior to digestion.

11.1.3 Verify that the hood is turned on. Turn on the Hot Block and set the temperature so that samples on the block maintain a temperature of 95 +/- 5ºC. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the sample temperature in the sample preparation logbook. The well position should be rotated on a per batch basis to ensure that the block temperature is verified across the block.

11.1.4 Place a 50mL well-mixed aliquot of each sample in digestion vials. Place 50 mL of Nano-pure water into digestion vials for each MB and LCS (one per 20 samples). Prepare the MS and MSD by placing 50mL aliquots of selected sample from the batch into digestion vials. One MS and one duplicate or MSD must be prepared for each analytical batch of 20 samples or less. Additionally, one MS must be prepared for each TCLP/SPLP/ASTM sample matrix digested in a digestion batch.

11.1.5 Spike the LCS, MS, and MSD with 1.0mL of the spiking solution.

11.1.6 Carefully add the digestion reagents to each sample, MB, LCS, MS, and MSD vial.

Digestion Program for all Aqueous Samples, EPA Method 3010A

Steps	Reagents[1]	Actions
1	5.0mL concentrated HNO_3, 2.5 mL HCl	Heat at 95 +/- 5ºC °C for 4 hours
2		Remove digestates from Hot Block and allow to cool in the hood.
3		Adjust the final volume of each vial to 50 mL by diluting with 18 MOhm Nano-Pure water. Place on screw cap and shake thoroughly.
4		Centrifuge, let settle, or filter sample only if there is concern that insoluble materials may clog the nebulizer. Filtering is liable to cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and pre-rinsed with dilute HNO_3. If filtration is required, filter all QC samples as well.

1. Acid ratios must be maintained to match method. Those specified are based on a 50mL sample volume

Note: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the samples and re-prep.

11.1.7 Record the lot numbers of all reagents and materials in the digestion logbook. This includes the lot numbers of the digestion tubes, filters or filter paper and any additional materials in contact with the sample matrices.

11.2 Soil/ Solid/Sludge/Paint Chip/Wipe Sample Preparation by 3050B

11.2.1 Soil, sludges, and solids are prepared according to Method 3050B. Wipes and Paint Chips are also prepared using 3050B.

11.2.2 Verify that the hood is turned on. Turn on the Hot Block and set the temperature to 95±5ºC. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the sample well ID and temperature in the sample logbook. This well measurement should be rotated on a per batch basis to ensure that the block temperature is consistent across the block.

11.2.3 To prepare Paint Chips for digestion, transfer paint chips into labeled drying tins. Dry samples at 103-105 degrees Celsius for 12-24 hours. Remove samples from oven and desiccate for 1 hour.

11.2.4 If possible, mix each sample thoroughly to homogenize. If homogenization is not feasible, remove the top layer of the sample from the container and discard. Remove a sample aliquot in the range of 0.5 – 0.599 g for analysis (0.5g dry sample). Weigh the aliquot to the nearest 0.01g and transfer to a digestion vial. Refer to Pace S-ALL-Q-021, *Sub-Sampling* SOP, for more details. For paint chips, Weigh out 0.5 gram of sample into a digestion tube. If the sample available is less than 0.5 gram, use the entire sample. For Wipes, digest the WHOLE wipe in the digestion vial. For sludges with % solids >10% and <50%, weigh out 2.5-5 grams.

11.2.5 Prepare the MB and LCS by adding 2.0 mL of Nano-pure water into digestion vials containing 0.500-0.504g of Teflon chips. The weight is documented as 0.500g so as to not bias the results. Prepare the duplicate, MS and MSD by placing aliquots of a selected sample from the batch into digestion vials as described for environmental samples. Spike the LCS, MS, and MSD with 1.0 mL spiking solution.

11.2.6 Carefully add the digestion reagents to each sample, MB, LCS, MS, and MSD vial and heat them with the Hot Block at 95±5ºC according to the following digestion program.

11.2.7 MS/MSDs are not performed on paint chips, because the sample concentration is frequently excessively high and dwarfs any spike. As a result an LCSD is performed for paint chip batches.

Digestion Program for Soil/ Solid samples by method 3050B

Steps	Reagents[1]	Actions
1	Nano-Pure Water	Add Appoximately 2.0 mLs of water to all vessels in the prep batch.
2	10.0 mL 1:1 HNO3	Cover with watch glass and heat without boiling for 2 hours. Remove from heat and cool.
3	2.5 mL 30% H_2O_2	Cover with watch glass and heat on Hot Block for 15 minutes or until effervescence subsides. Warming will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Remove and allow to cool.
4	2.5mL concentrated HCl	Reflux on Hot Block until reaction is complete (no brown fumes) without boiling. Allow samples to cool.
5		Dilute sample to 50-mL final volume with 18 MOhm Nano-Pure water. Place screw cap on vials, shake, allow sample to settle, centrifuge at 2000-25000 rpm for 10 minutes, OR and filter if necessary.

1. Acid ratios must be maintained to match method. Those specified are based on a 0.5g sample amount
2. In effect, this produces the 1:1 nitric acid (required by method 3050) in the actual digestion cup.

11.2.8 Record the lot numbers of all reagents and materials in the digestion logbook. This includes the lot numbers of the digestion tubes, filters or filter paper and any additional materials in contact with the sample matrices.

11.3 ICP System Preparation

11.3.1 Preventative Maintenance

Inspect the sample introduction system including the nebulizer, torch, injector tube and up-take tubing for salt deposits, dirt and debris that could restrict solution flow and affect instrument performance. Document all maintenance in ICP maintenance logbook.

See Attachment I for troubleshooting guide.

Operating Parameters

Set up and configure the ICP according to manufacturer's operating instructions using operating parameters shown in the following table.

Table 11.3.1 - Operating Parameter for ICP Systems

Operating Parameter	ICAP 6500 20073913
Argon Flow	80 L/min
Nebulizer Pressure	0.55 L/min
RF Output	1175 W
Pump Speed	15 rpm
Aux Gas Flow	0.5 L/min
Coolant Gas Flow	15 l/min

11.3.2 Calibrate ICP according to Section 10.

11.4 Sample Analysis

11.4.1 Batch Sequence

If using an autosampler, enter an autosampler sequence into the ICP data system per manufacturer's instuctions. Calibration blanks, standards, initial checks, and interlement correction standards shall be run before analysis of environmental samples. All samples, including calibration, checks, and other standards, can also be run manually.

Table 11.4.1 – Example analytical sequence after calibration of a blank and a standard.

Autosampler Position	Sample Description	Autosampler Position	Sample Description
1	ICV	27	Sample 15
2	ICB	28	Sample 16
3	CRDL	29	CCV
4	ICSA *	30	CCB
5	CCV	31	Sample 17
6	CCB	32	Sample 18
7	MB1	33	Sample 19
8	LCS1	34	Sample 20
9	Sample 1	35	MB2
10	Sample 2	36	LCS2
11	Sample 3	37	Sample 21
12	Sample 3 Matrix Spike	38	Sample 22
13	Sample 3 Matrix Spike Duplicate	39	Sample 23
14	Sample 4	40	Sample 24
15	Sample 5	41	CCV
16	Sample 6	42	CCB
17	CCV	43	Sample 25
18	CCB	44	Sample 26
19	Sample 7	45	Sample 27
20	Sample 8	46	Sample 28
21	Sample 9	47	Sample 28 Matrix Spike
22	Sample 10	48	Sample 28 Matrix Spike Duplicate
23	Sample 11	49	Sample 29
24	Sample 12	50	CRDL
25	Sample 13	51	CCV
26	Sample 14	52	CCB

* ICSAB will follow the ICSA if needed/requested by client.

Load Autosampler

11.4.2.1 Load the autosampler tray according to the vial position on the sequence table.

11.5 Sample Analysis

11.5.1 Analyze all standards, quality control samples, and environmental samples.

11.6 Data Reduction

11.6.1 **Quantitative Analysis –** The instrument produces results in ug/L. If the initial sample aliquot and final digestate volumes are the same, the ICP data system will calculate the concentration of each element directly. If the initial and final volumes are different, the actual values are posted into the LIMS system and calculations are performed in the LIMS.

11.6.1.1 Water Samples – Since initial sample aliquot and final digestate volumes are the same, the iCAP analyzer data system will calculate the concentration directly. No further calculations are necessary unless the sample was diluted.

11.6.1.2 Soil/Solid/Paint Chip/Sludge Samples -

$$\text{Final Result (mg/kg dry weight corrected)} = \frac{\text{Raw data result (ug/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{Sample weight (g)} * \text{Dry weight for soil/solid (decimal form)}}$$

11.6.1.3 Wipes

$$\text{Final Result (Total ug)} = \frac{\text{Raw data result (ug/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{1 wipe}}$$

11.6.2 Samples with analyte concentrations that exceed 90% of the upper linear range must be diluted and re-analyzed.

11.6.3 Any sample in which any element value is below the instrument detection limit or PRL must be reported as less than the PRL (or ND) for that specific element. Do not report data below the element PRL concentration unless it is qualified as an "estimated" result with a "J" or "B" data qualifier.

11.6.4 Metals Analytical Review and Reporting System (ADAMS) or LimsLink is used to transfer data from the instrument to the LIMS system.

12. QUALITY CONTROL

12.1 Refer to the most current version of the Pace Quality Manual Appendix I Quality Control Calculations and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

12.2 Instrument Quality Control

12.3 Refer to Table 10.1 for initial and continuing calibration criteria and corrective actions. This includes ICV, CCV, CB, RLVS, and ICSA standard checks.

12.4 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Nano-Pure water	One per 20 samples	1) Target analytes must be less than the RL.	1) Re-analyze blank to confirm failure. 2) Qualify results and / or re-extract associated samples. **Exceptions:** 1) If sample ND, report sample without qualification 2) If sample result >10x MB detects no qualification is needed. 3) If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Nano-Pure Water and Spiking solution (see table 9.3.4 for elements)	One per 20 samples	Laboratory Calculated Limits or default limits of 80-120% Should not exceed 80-120%	1) Reanalyze the LCS to verify failure 2) If LCS passes, review samples for potential problems 3) If problem persists, check spike solution 4) Re-digest and re-analyze samples where possible **Exceptions:** 1) If LCS rec > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Environmental Sample and Spiking solution (see table 9.3.4 for elements)	One per 20 samples. For leached samples one per sample matrix (soil, and wood chips would each require a separate matrix spike).	Laboratory Calculated Limits or default limits of 75-125% Should not exceed 75-125%	1) If LCS and MBs are acceptable, the MS may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate OR (alternative) Sample Dup	A duplicate sample OR MS/MSD pair must be analyzed per batch of 20 samples.	Laboratory Derived Limits for RPD or default limits of 20%. Recovery criteria should not exceed 75-125%.	1) Results not meeting criteria must be footnoted on client report.

12.5 Dilution Test – A dilution test is recommended but not required for method 6010C. A dilution test is run only at client request.

12.5.1 Perform a 5X dilution on one sample per preparation batch. Diluted and undiluted sample results should agree within 10% of each other if the sample contains analyte concentrations at least 50 times the PRL. If agreement between diluted and undiluted samples meeting the concentration requirements is >10%, then matrix effect should be suspected and samples footnoted appropriately.

12.6 Post-digestion Spike Test or Analyte Addition – Post digestion spikes are recommended but not required for method 6010C. A post spike is run only at client request.

12.6.1 Spike one digestate from a preparation batch to achieve the same analyte addition level as found in the LCS. The acceptable recovery range is 80-120%. If outside this range, matrix effects should be suspected and samples annotated.

12.7 **Internal Standards –** Yttrium is used as an internal standard. Yttrium is not calibrated for. Precision of the Yttrium reps are evaluated using < or = 5% RSD. Accuracy of the Yttrium average count levels are evaluated using 50-150% recovery as compared to the calibration blank. If either of these criteria is not meet, the sample or quality control standard must be rerun, possibly at a dilution.

12.8 **Multiple Instrument Integration RSD** – The RSD is required to be evaluated between multiple instrument integrations. The RSD must be less than or equal to 20% if the analyte concentration is greater than the reporting limit. If the RSD is greater than 20%, the laboratory must reanalyze the sample.

13. METHOD PERFORMANCE

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted annually per S-GB-Q-020, *Determination of LOD and LOQ* per instrument.

13.2 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures*.

13.3 **Linear Dynamic Range Study**

13.3.1 It is a Pace best practice to perform this linear dynamic range study determination once, keep the data, and then annually or semi-annually verify with a single high point. If additional range is to be considered, the laboratory must perform the study as indicated below.

13.3.2 A linear dynamic range study must be conducted at least annually. The study is conducted for each element by analyzing increasing concentrations (at least 3 levels) until the results generated exceed ±10% difference from the true value. The highest concentration within the 10% criteria is the maximum of the linear range for that element.

13.4 **Instrument Detection Limits (IDLs)** in ug/L are estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined at least quarterly.

13.5 **Lower limit of quantitation check sample (LLQC)**

The LLQC samples should be analyzed after establishing the PRLs, and on an as needed basis to demonstrate the desired the detection capability. The LLQC sample is carried through the entire preparation and analytical procedure. Acceptance criteria are ±30% of the true value.

13.6 Method Modifications

13.6.1 Method modifications for EPA method 6010C are as follows:

13.6.2 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.

13.6.3 All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.

13.6.4 Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories

13.6.5 When insufficient volume for the method specified matrix spike/matrix spike duplicate (MS/MSD) is not provided by the client, a laboratory control spike duplicate will be analyzed to demonstrate precision criteria. Laboratory batches will be qualified with the appropriate "M5" data qualifier. When performing this analysis on paint chip samples, a MS/MSD will not be completed on the samples due to high levels of elements present in the native sample.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management*.

14.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. REFERENCES

15.1 USEPA, SW-846, Method 6010C Revision 3, "Inductively Coupled Plasma Atomic Emission Spectrometry", February 2007.

15.2 USEPA, SW-846, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectrometry", December 1996.

15.3 USEPA, SW-846, Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils", December 1996.

16. TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS, APPENDICES, ETC.

16.1 **Attachment I** - ICP Troubleshooting Guide

16.2 **Attachment II** – ICP Flowchart

17. REVISIONS

Document Number	Reason for Change	Date
S-ALL-GB-M-005-REV.00	First Issue	22Sept2008
S-GB-M-005-REV.01	Update Signature Page	26May2009
S-GB-M-005-Rev.02	Updated Signature Page Table 3.1: Updated Pb PRL to 7.5ug/L Sections 9.2.1 and 10.2: Added that calibration solutions can be used until manufacturer's expiration date Section 11: Changed HNO3 to 5mL from 3mL.	28Apr2010
S-GB-M-005-Rev.03	Body of Document: Included Leach Information Corrected Formatting throughout document. Corrected Header information to 6010C from 6010B. Table 3.1: Updated Reporting Limits to match current. Table 3.1, Section 11.6.1.3: Changed unit for Wipe to Total ug. Section 7.1: Added TCLP/SPLP/ASTM Preservation Requirement and Holding Times. Section 7.2: Preservation Check: Changed maximum volume of acid to add as 2% from 5%. Table 9.3.6: Updated Vendor and Catalog number. Section 11.6.4: Updated program for data reduction to ADAMS from MARRS.	28Jun2011
S-GB-M-005-Rev.04	Table 3.1: Updated TCLP/SPLP/ASTM PRLs. Section 9 Tables: Updated formatting to merged cells when the same standard, acids, final volume, etc. are used for one complete standard. Section 9: Updated expiration dates of standards to 6 month or manufacturer expiration date, whichever is sooner. Added Table 9.3.5 CRDL Intermediate Standard. Section 13.4: Changed requirement for IDL Studies to quarterly from yearly. Section 13.6.4 and 13.6.5: Added method modifications. Throughout document: Updated instances of Reagent Water to Nano-Pure Water, updated RLVS to CRDL standard.	30Sep2011
S-GB-M-005-Rev.05	Section 4.2: Updated language on physical interferences. Section 4.5: Updated high salt interferences language. Table 9.3.2: Updated volume of standards used. Table .9.3.5: Updated K concentration in stock and working std. Table 9.3.6: Updated Ag concentration in stock and working std. Section 11.2.4: Updated digestion procedures.	07Dec2012

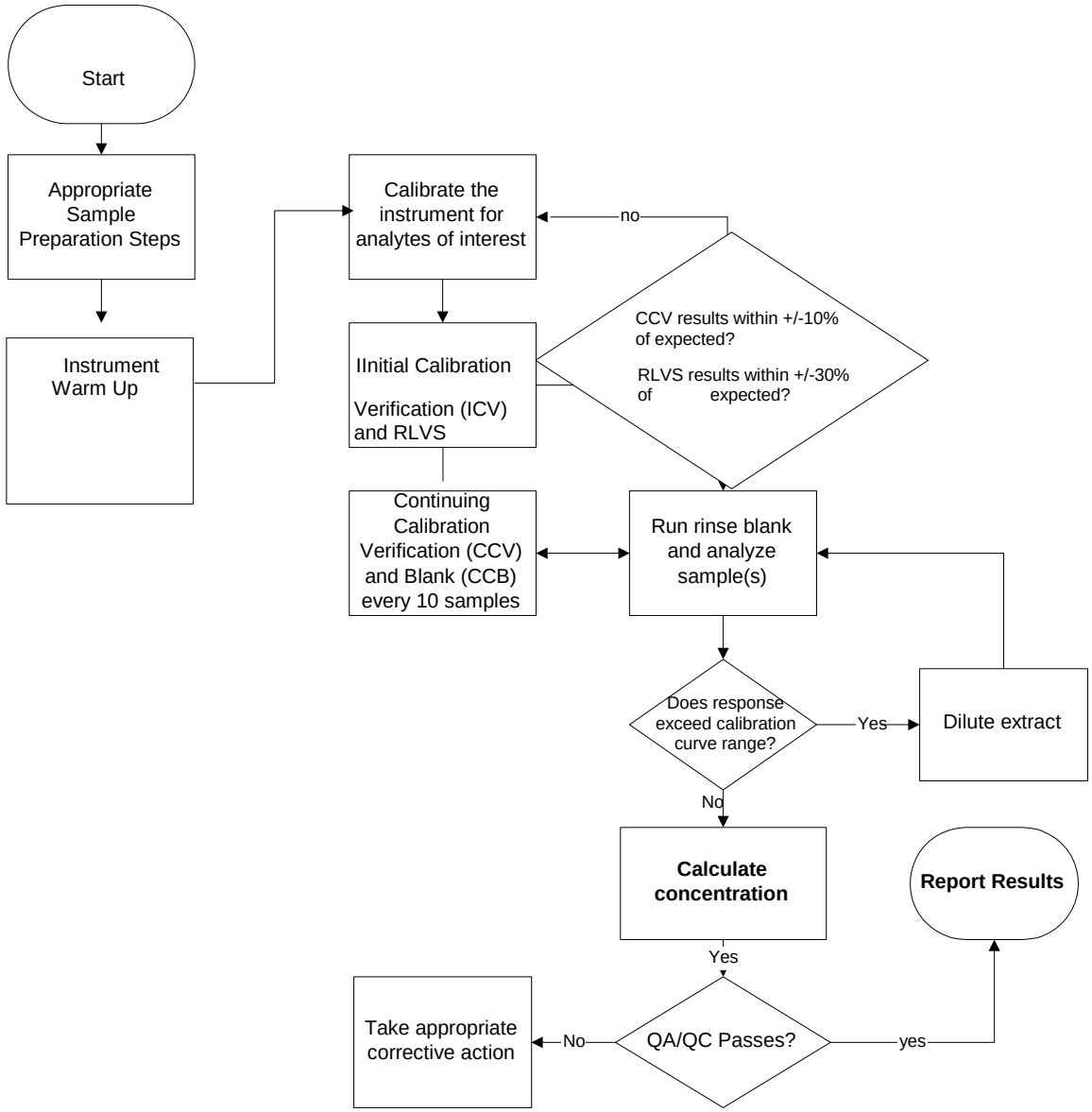
Attachment I

GENERAL ICP TROUBLESHOOTING GUIDE

Problem	Solution
Poor linearity in a dilution series of standards.	This may be as easy as not having spiked your standards correctly. Before you remake your standards, first examine the sample introduction area, making sure you're getting a consistent flow of sample through your pump tubing and that your connectors are not too tight. Then check your nebulizer and injector for visible build up. If build up is present, clean in a dilute nitric acid (HNO_3) solution for about 10-15 minutes. Also examine your torch, making sure it is also reasonably clean. Lastly, check the argon tank to determine that it is not running on empty. If everything is satisfactory, remake standards. If problems persist, try a second source of standards to determine if your initial set of standards has gone bad.
Bubbles are collecting in the back area of the nebulizer (glass type).	The reason why bubbles form in this back area is because the pump tubing is not inserted deeply enough into the cavity of the nebulizer. Cut the tip of the pump tubing to be inserted into the nebulizer at a roughly 45º angle, preferably with a razor blade to obtain a nice, clean cut. Dip the cut end into a wetting agent such as dilute Triton X solution, and start to insert into the nebulizer. Using moderate pressure, begin to work the pump tubing into the nebulizer while twisting the tubing. The twisting action causes the tubing to gradually decrease in diameter, making it easier to insert deeply into the cavity.
Hard instrument failure (software related). These are generally problems associated with the software communication with the instrument.	Exit out of the program. Shut down the PC that is linked to the instrument itself and restart. During the PC reboot process, locate the reset button(s) on the front and/or back of the instrument and depress them. Enter back into the program and continue your analysis. This generally will fix this type of problem, but if you encounter additional problems of this type, then a service call may be necessary for more advanced troubleshooting advice.
Peristaltic pump occasionally stutters or stops. This problem may be hard to notice since this is a gradual wearing of the belt that attaches to the pump motor and turns the rollers themselves.	Replace the belt if you have an available spare. If you do not have a replacement, a service call needs to be made. A service person will generally come out and install the belt but on occasion, based on your mechanical abilities, they may ship the part to you and have you perform the installation.

Attachment II

ICP Flowchart



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STANDARD OPERATING PROCEDURE

MEASUREMENT OF pH IN WATER, SOIL, AND WASTE

REFERENCE METHODS: SM 4500-H^+ AND SW-846 METHODS 9040B AND 9045C

LOCAL SOP NUMBER:	S-GB-I-071-REV.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S -GB-I-071-REV.00
SOP TEMPLATE NUMBER:	SOT-ALL-I-015-Rev.00

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 09/28/12

Kate Grams, Laboratory Quality Manager — Date: 9/26/12

Chad Rusch, Department Manager — Date: 09/27/2012

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

The purpose of this SOP is to provide a laboratory specific procedure to measure pH in water, soil, and waste samples while meeting the requirements of SM Method 4500-H^+ and SW846 Methods 9040B and 9045C.

2. Summary of Method

2.1. Water samples and wastes containing at least 20% water are measured electrometrically using a combination pH electrode with temperature compensation. The method for analyzing this type of sample is either SM Method 4500-H^+ or SW846 9040B and must be reported referencing one of these methods.

2.2. Soils and solids containing less than 20% water are mixed with reagent water and the pH of the resulting aqueous suspension is measured in the same manner as a water sample. The method for analyzing this type of sample is SW846 9045C and must be reported referencing this method.

3. Scope and Application

3.1. This method is applicable to water samples (including drinking water, surface water, groundwater, saline water, and domestic and industrial wastewaters), soils, sludges, and solid wastes.

3.2. Since pH measurement requires some water content, concentrated acids and bases and concentrated acids and bases mixed with inert substances cannot be measured.

3.3. This SOP was prepared for method-compliant use by all Pace Analytical environmental laboratories.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced with electrode measurements. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

4.2. Sodium error at pH levels greater than 10 can be reduced or eliminated by using a “low sodium error” electrode.

4.3. Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or washing with detergent, then rinsing with distilled water. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.

4.4. Sample temperature can cause variations in electrometric measurement of pH in two ways, by causing a change in electrode output and by actually altering the sample pH. Since both variation sources cannot be fully controlled, measurements should be made using a temperature compensating electrode and the analysts should report the sample temperature at the time of analysis along with the pH result. **Coatings of oily material or particulate matter can impair electrode response. The electrode should be thoroughly rinsed and cleaned between samples. Wiping of the electrode using an optical safe wipe may be required to remove oil or clay like soils.**

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

6. Definitions

Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Plastic container (125 mL).	None	If samples cannot be analyzed immediately, they must be stored at 4±2 °C	Samples should be analyzed as soon as possible following collection, preferably at the collection site.(1,2)
Soil	Plastic container (4-oz)	None	If samples cannot be analyzed immediately, they must be stored at 4±2 °C	Samples should be analyzed as soon as possible following collection, preferably at the collection site.(2)

(1) For aqueous samples analyzed and reported according to SM 4500-H^+, PASI observes a 15 minute hold time. Laboratories must qualify results as exceeding the recommended hold time for this ***field parameter*** when analysis occurs beyond this time frame.

(2) For samples analyzed and reported according to either SW-846 9040B or 9045C, PASI observes a 24 hour hold time. Laboratories must qualify results as exceeding the recommend hold time when analysis occurs beyond this time frame.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
pH Meter	Orion	Orion 720A	
Glass Combination Electrode with Automatic Temperature Compensation	Orion	9157BN	Orion Triode pH Electrode
Analytical Balance	Mettler	PJ 480	Capable of weighing to 0.01 g

8.2. General Supplies

Item	Vendor	Model / ID	Catalog #	Description
Disposable Beakers, 50-mL	Fisher		01-29107	
Magnetic Stirrer	Fisher	Corning		
Spin Bars	Fisher			
Wash Bottle	Fisher			
Graduated cylinders	Fisher			50-mL or 100-mL
Laboratory Wipe Tissues	Fisher		06-666A	Kimwipes

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	De-ionized water	ASTM Type II water
Electrode Filling Solution	Used for filling electrode	Orion p/n 90004

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, and second source verification.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Primary Calibration Buffer, pH 2.00 ± 0.02	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB96-1, or equivalent
Primary Calibration Buffer, pH 7.00 ± 0.01	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB107-4, or equivalent
Primary Calibration Buffer, pH 10.00 ± 0.02	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB115-4, or equivalent
Primary Calibration Buffer, pH 12.45 ± 0.01	This standard solution is used for daily calibration of the measurement system. Manufactured by Oakton – p/n 00654-12	MG Scientific p/n T-00654-12, or equivalent
Secondary Source Calibration Buffer, pH 4.00 ± 0.01	This standard solution is used to verify calibration before sample analysis.	Fisher p/n SB101-4, or equivalent
Secondary Source Calibration Buffer, pH 7.00 ± 0.01	This standard solution is used to verify calibration before sample analysis. Manufactured by Oakton – p/n 00654-04.	Fisher p/n13300153, or equivalent

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Primary Calibration Buffer, pH 2.00 ± 0.02	Fisher p/n SB96-1, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 7.00 ± 0.01	Fisher p/n SB107-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 10.00 ± 0.02	Fisher p/n SB115-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 12.45 ± 0.01	MG Scientific p/n T-00654-12, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Secondary Source Calibration Buffer, pH 4.00 ± 0.01	Fisher p/n SB101-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Secondary Source Calibration Buffer, pH 7.00 ± 0.01	Fisher p/n13300153, or equivalent	Manufacturer's recommended expiration date	Store at room temperature

10. Calibration

10.1. Calibration - The measurement system must be calibrated daily with four calibration buffer solutions. These solutions must bracket the range of pH values measured. Additional calibration points are recommended provided the pH meter can accept additional calibration data points.

10.1.1. Press ***power*** to turn the meter on. Press **2nd/channel** until the correct input channel is selected.

10.1.2. Press the **mode** key until the pH mode indicator is displayed.

10.1.3. Press **calibrate.** CALIBRATE is displayed. The time and date of the last calibration is displayed.

10.1.4. After a few seconds ENTER NO. BUFFERS is displayed. Use the numeric keys to enter the number of buffers to be used, then press **yes**. Enter four for the number of buffers to be used.

10.1.5. Place a stir bar in the beaker, put the beaker on the stirrer, and adjust the stirrer to a moderate speed. When the BUFFER 1 prompt appears place electrode(s) in first buffer containing stir bar on.

10.1.6. When the electrode signal has stabilized, the prompt READY CAL AS will appear. Use the numeric keys to enter the value of the buffer. Press **yes** to enter the value.

10.1.7. Rinse the electrode with DI water and gently wipe with a tissue. Repeat steps 5 and 6 for each buffer.

10.1.8. The average electrode slope is displayed at the SLOPE prompt.

10.1.9. The meter automatically proceeds to MEASURE mode.

10.1.10. Record the readings for each buffer used during calibration along with the slope in the pH logbook. (See Attachment I.)

10.2. Initial Calibration Verification – The calibration must be verified with two secondary source calibration buffers prior to conducting measurements on samples. Perform initial calibration verification using the two secondary source calibrations buffers (4.00 and 7.00 pH Units). The control criteria for this verification is listed in Table 12.1.

10.3. Continuing Calibration Verification – The calibration must be verified with a single point verification buffer (pH 7.00) every 10 samples. The control criterion for this verification is listed in Table 12.1.

11. Procedure

11.1. Water Samples

11.1.1. Allow samples to warm to room temperature. Unless a temperature compensated probe is used, samples must be within ±2°C of the standard buffer solutions used for calibration.

11.1.2. Place the sample in a 50-mL disposable beaker, using sufficient volume to cover the sensing elements of the electrode and provide adequate clearance for the magnetic stirring bar. Place a magnetic stirring bar in the beaker and place the beaker on the stirrer. Turn on the stirrer to a moderate speed. The sample should be noticeably mixing but should not develop a vortex.

11.1.3. Rinse the electrode with DI water and gently wipe with a tissue. Place the pH electrode into the sample beaker, taking care to provide adequate clearance for the stirring bar.

11.1.4. Wait for the meter reading to stabilize and note the pH.

11.2. Soil and Solid Samples

11.2.1. Place 20g of sample into a 50-mL disposable beaker. Add 20mLs of DI water and a stirring bar to the beaker and place it on the stirrer. Turn the stirrer to a moderate speed and continuously stir the suspension for 5 minutes.

11.2.2. If the matrix is hydroscopic and has absorbed all the water leaving no remaining free liquid for analysis, repeat the preparation using 20g of sample and 40mLs of DI water.

11.2.3. Turn off the stirrer and allow the sample to sit, permitting the suspended particulates an opportunity to settle to the bottom of the container. Samples may be allowed to sit for up to one hour prior to analysis. Excessive solids in suspension may speed the clogging of the frit between the electrodes.

11.2.4. Place the pH electrode and ATC electrodes into the sample beaker, taking care to immerse the combination electrode just below the suspension. Wait for the meter reading to stabilize and record the pH result and time analyzed. Record the results to 0.1 pH units.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Secondary Source Calibration Buffers	4.00 and 7.00 pH Units Secondary Calibration Buffer Solutions	Daily after calibration	± 0.1 pH Units	Re-calibrate
CCV Buffer	7.00 pH Units Secondary Calibration Buffer Solution	One after every 10 samples and at the end of the batch of samples.	± 0.1 pH Units	Terminate analysis, correct the problem, re-calibrate the system, and re-analyze the check solutions and all samples that are not bracketed by acceptable QC.
Duplicate Sample	Sample	One per batch of 20 or fewer samples for soil matrices. None required for aqueous samples.	5.0% RPD	Terminate analysis, correct the problem, re-calibrate the system, and re-analyze the check solutions and all samples that are not bracketed by acceptable QC.
Calibration of pH Probe				Calibrate the temperature function of the pH probe on an annual basis as per the current revision of PASI SOP S-GB-Q-030, *Support Equipment.* Record the calibration in the thermometer calibration logbook and label the probe with the date of calibration.

13. Method Performance

13.1. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement). All results must be in control to qualify the analyst for reporting sample results (within 0.1 pH units of the true value). Results of DOC studies for each analyst shall be retained in the lab quality assurance office. DOCs must be repeated annually. For this test four analysis of the second source pH 7 buffer will serve as the DOCs.

13.2. Method Modifications: If the matrix is hydroscopic and has absorbed all the water leaving no remaining free liquid for analysis, repeat the preparation using 20g of sample and 40mLs of DI water. This is a method modification of 9045C. Method 9045C requires a 1:1 ratio, but since this would not be possible with a sample of this nature the noted procedure will be followed and the sample qualified as such.

14. Pollution Prevention and Waste Management

14.1. Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

14.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. References

15.1. Method 4500-H^+ B., Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

15.2. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 9040B "pH Electrometric Measurement".

15.3. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 9045C "Soil and Waste pH".

15.4. Pace Analytical Quality Manual; latest revision.

15.5. NELAC Standard; Quality Systems section; latest revision.

16. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

Attachment I - pH Logbook Template.

17. Revisions

Document Number	Reason for Change	Date
SOT-ALL-I-015-rev.00	Restructured document format to new corporate template. Included requirement for duplicate analysis for all aqueous matrices	12Dec2006
S-ALL-GB-I-015-REV.00	Corporate Template updated for use in the Green Bay Laboratory	27Aug2008
S-ALL-GB-I-015_REV.01	Update Signature Page Update Benchsheet	26May2009
S-GB-S-071-Rev.00	Updated SOP numbering following current revision of S-ALL-Q-003 Updated Waste Management and Handling SOP throughout document. Updated pH Logbook to new revision.	27Sep2011
S-GB-I-071-Rev.01	Section 13.2 – Added Method Modification information	26Sept2012

ATTACHMENT I
pH Logbook

Pace Analytical

pH (SW 846 9040B, SW 846 9045C, SM 4500-H+B)

Logbook #

Meter: Orion Model 720A (40WET8)	Meter Serial Number: 008577	
Meter Calibration Date:	Analyst:	
Initial Calibration pH Buffer Solutions		
Value	**pH Readings**	**Reagent Code**
pH 2.00		
pH 7.00		
pH 10.0		
pH 12.45		
Slope:		
Second Source pH Buffer Solutions		
pH 4.00		
pH 7.00		

Sample No.	Date	Time	Matrix	pH.	Adjusted pH	Chlorine	Worksheet #	Analyst

Calibrate pH meter daily with pH buffers 2.0, 7.0, 10.0 & 12.45. Verify the calibration with the second source pH buffers 4.00 and 7.00 (Control Limits +/-0.10 SU). Analyzed second source pH buffer 7.00 after every 10 readings (Control Limits +/- 0.10 SU). Analyze duplicate sample every 20 samples (Control Limits +/- 0.10 SU). For SW846 9040B analyze consecutive measurements of the sample to verify they read within +/- 0.10 SU)

F-GB-I-009-Rev.06 (28Sep2011)

Review/Date_____

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STANDARD OPERATING PROCEDURE

Measurement of Volatile Solids and Solids in Waters

Reference Methods: Standard Methods 2540-B, -C, -D, -E, -G, ASTM D2974 and EPA 160.4

SOP NUMBER:	S-GB-I-068-Rev.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S- GB-I-068-Rev.00

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/03/12

Kate Grams, Laboratory Quality Manager — Date: 11/29/12

Chad Rusch, Department Manager — Date: 12/03/12

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose /Identification of Method

1.1 This Standard Operating Procedure (SOP) describes operations used to measure the parameters in the following table:

Table 1.

Test		Matrix	Method	Reports as
TS	Total Solids	Water	SM 2540B	mg/L
TS	Total Solids	Solid	SM 2540G	%
TSS	Total Suspended Solids	Water	SM 2540D	mg/L
TDS	Total Dissolved Solids	Water	SM 2540C	mg/L
TVS	Total Volatile Solids	Water	EPA 160.4	mg/L
TVS	Total Volatile Solids	Solid	EPA 160.4	%
TVSS	Total Volatile Suspended Solids	Water	EPA 160.4	mg/L
OM	Organic Matter	Solid	ASTM D2974-87(C)	%
FOC	Fractional Organic Carbon	Solid	ASTM D2974-87	%

2. Summary of Method

2.1 Total Solids (TS) – A well-mixed sample is evaporated to dryness and the residue solids are measured gravimetrically.

2.2 Total Suspended Solids (TSS) – A well-mixed sample is filtered. The residue collected by the filter is dried and measured gravimetrically.

2.3 Total Dissolved Solids (TDS) – A well-mixed sample is filtered. The filtrate passing through the filter is evaporated to dryness and the residue solids are measured gravimetrically.

2.4 Total Volatile Solids (TVS, TVSS) – Residue obtained from the determination of TS, or TSS is ignited at 550ºC in a muffle furnace. The loss of weight on ignition is reported as % Volatile Solids for solid samples and mg/L Volatile Solids for liquid samples.

2.5 Fractional Organic Carbon (FOC) - Residue obtained from the determination of TS is ignited at 440ºC in a muffle furnace. The weight loss after ignition is multiplied by a 0.58 factor and reported as % Fractional Organic Carbon.

2.6 Organic Matter (OM) - Residue obtained from the determination of TS is ignited at 440ºC in a muffle furnace. The weight loss after ignition is reported as % Organic Matter.

3. Scope and Application

3.1 This method is applicable to the determination of Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS), and Total Volatile Solids (TVS) in water samples.

3.2 This method is applicable to the determination of Total Solids (TS) and its Volatile (TVS) fractions in soil and semisolid samples. The terms total solids apply to matter left after drying in an oven at 103-105º C and igniting in a muffle furnace at a 550ºC temperature and a defined time interval.

3.3 This method is applicable to the determination of Fractional Organic Carbon (FOC) and Organic Matter (OM) in soil and semisolid samples. The term fractional organic carbon (FOC) applies to the weight loss after ignition, multiplied by a 0.58 factor and reported as % Fractional Organic Carbon.

3.4 This procedure is restricted to use by, or under the supervision of, analysts experienced with the use of laboratory information systems, balances, desiccators, and ovens. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1 This SOP applies to surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, soils, and sludges.

5. Limits of Detection and Quantitation

5.1 The reporting limit (LOQ) for all analytes is listed in Table 1 for the listed methods. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

Table 2.
REPORTING LIMITS

COMPOUND NAME	Aqueous	Solids
	Units mg/L	Units %w/w
	LOQ	LOQ
TS	20	0.1
TSS	10	
TDS	20	
TVS, TVSS	20	
FOC		0.058
OM		0.1

6. Interferences

6.1 Non-representative materials, e.g., leaves and sticks should be removed from the sample prior to measurement unless it is determined that their inclusion is desired.

6.2 Measurements are subject to negative bias for samples containing significant quantities of ammonium carbonate, volatile organics, or other volatile materials that could be lost during drying.

6.3 The residue of samples for TS and TDS that are highly mineralized, especially containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require longer drying, desiccation, and rapid weighing.

6.4 Samples for TS and TDS containing high concentrations of bicarbonate will require careful, and possibly prolonged, drying to ensure that all bicarbonate is converted to carbonate.

6.5 The volumes of aliquots for TS and TDS should be selected to limit the total residue to 200 mg to prevent the residue from crusting over and trapping water during drying.

6.6 Samples for TSS with high TDS, such as saline waters, brines, and some wastes, may be subject to positive bias. Care must be taken to properly rinse the filter to minimize the bias.

7. Sample Collection, Preservation Shipment and Storage

Table 3
SAMPLE COLLECTION, PRESERVATION, HOLD TIME SHIPMENT AND STORAGE

Matrix	Analytical Method	Container(s)	Preservation / Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	SM 2540B SM 2540C SM 2540D EPA 160.4	Clean Plastic or glass containers	None / 7 Days	On ice ≤6° Celsius	Walk-in cooler ≤6° C
Soils	SM 2540G ASTM D2974-87	Clean Plastic or glass containers	None / 7 Days	On ice ≤6° Celsius	Walk-in cooler ≤6° C

8. Definitions

8.1 Batch – A grouping of 20, or less, environmental samples of similar matrix, which are prepared and/or analyzed together with the same method.

8.2 Blank Sample – A sample analyzed to determine potential contamination attributed to sample handling and analysis.

8.3 Duplicate Sample – A second aliquot of the same environmental sample analyzed in the same manner as the original sample in order to evaluate precision.

8.4 Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required.

8.5 Laboratory Control Sample (LCS) – A blank matrix sample spiked with a known concentration of analytes of interest. Laboratory accuracy is evaluated using the LCS.

8.6 Laboratory Information Management System (LIMS) – This is a system for transferring, processing, storing, and reporting analytical results.

8.7 Replicate Samples – Samples collected at the same time, from the same place, for the same analysis, as the original sample in order to determine precision between samples.

8.8 Pace Reporting Limit (PRL) – The lowest level that will be reported by this method. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.

8.9 Solid – matrix such as soils, sediments, sludge, organic liquids, oils or aqueous products and by-products of industrial processes, and aqueous samples with more than 10% settleable solids.

8.10 Total Solids Content (TSC) – Total Solids Content is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature of 103-105ºC.

8.11 Volatile Solids – Residue obtained from the determination of TS is ignited at 550ºC in a muffle furnace. The loss of weight on ignition is reported as % Volatile Solids.

8.12 Refer to Section 10 of the Pace Quality Manual for additional terms used in this SOP.

9. Equipment and Supplies

9.1 Analytical balance – Electronic, capable of weighing to 0.1 mg.

9.2 Vacuum Filtration System – Includes filter holder, membrane filter funnel, vacuum flask and vacuum pump.

9.3 Glass Fiber Filters (for TDS) – GE Water & Process Technologies PreSep Prefiler, Glass, 1.0 Micron, 47mm, or equivalent.

9.4 Glass Fiber Filters (for TSS) – Pre-weighed from Environmental Express, ProWeigh 47mm Glass Fiber Filters, 1.5 µm, with matched aluminum evaporating dishes, or equivalent.

9.5 Graduated Cylinders – 25, 50, 100, 250, 500 and 1000mL.

9.6 Wide bore serological pipettes and pipette helper.

9.7 Drying Ovens – Capable of maintaining 103-105ºC and 178-182ºC.

9.8 Desiccator

9.9 Indicating Desiccant – Drierite Anhydrous Calcium Sulfate, Stock # 2305, or equivalent.

9.10 Laboratory Scoopulas

9.11 Muffle furnace (for volatile solids) – Capable of maintaining temperature of 440ºC and 550ºC.

9.12 Evaporating Beaker (TS/TDS/TVS/OM/FOC) – 50 mL

10. Reagents and Standards

10.1 ERA DataPacK Total Volatile Solids Standard, Cat # 913 for TVS, OM, and FOC analysis, or equivalent.

10.2 ERA QC Plus Solids, Cat #4033 for TS, TSS, and TDS analyses, or equivalent.

10.3 NSI Solutions, Cat # QCI-084 for TS, TSS, and TDS analysis, or equivalent.

10.4 Nanopure Reagent Water – ASTM Type II, or equivalent.

11. Calibration and Standardization

11.1 Balance Calibration – The only required calibration is the analytical balance.

11.2 The balance must be calibrated at least annually by an outside agency and checked **daily** across the range of use, using Class 1 or 2 weights as per the most current version of SOP S-GB-Q-030 *Support Equipment.*

11.3 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

11.3.1 1g for TSS and TVSS with a tolerance of +/-0.0100g

11.3.2 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

12. Procedure

12.1 Create the analytical batch in Horizon.

12.2 Open the Electronic Logbook and create a new worksheet for the batch to be analyzed.

12.3 Pre-Weighed Dried Beaker. Ignite the evaporating beaker at 550ºC for 1 hour in a muffle furnace. Cool in desiccator and store there until ready to use. Record their weight prior to use.

12.4 Check that the oven temperature is within required specifications before placing samples into oven as per the most current version of SOPS-GB-Q-030 *Support Equipment*. Document temperature in the Electronic Logbook.

12.5 **Weighing Dry/Muffle Weights**

12.5.1 Oven temperatures are the same as those described for the test being conducted.

12.5.2 Place the samples back into the oven for 15 minutes and repeat desiccating, cooling, and weighing steps until weight change is $\leq$0.0005g.

12.5.3 If the first and second weights agree within 0.0005 g, or if the weight loss between the first and second weighing is less than 4%, the analysis is complete. If not, place them back into the oven for and additional 15 minutes. Cool in the desiccator, and weigh a third time.

12.5.4 If the third weigh is within 0.0005 g of either the first or second weight or if the weight loss between the second and the third weighing is less than 4%, the analysis is complete.

12.5.5 If the weight loss between the second and third weighing is more than 0.0005g and 4%, heat, cool, and weigh the sample a fourth time and use the fourth weight to calculate the result.

12.6 **Total Solids Determination (TS)**

12.6.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be $\leq$0.0005g before use.

12.6.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in Electronic Logbook.

12.6.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of NSI Solutions (or equivalent) and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in Electronic Logbook.

12.6.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.6.5 Sample

12.5.4.1 For Solid Samples – Weight out 1 to 1.5g of sample in a tared and dried evaporating beaker. Record the weight in Electronic Logbook.

12.5.4.2 For Aqueous Samples - Shake the sample thoroughly and measure an aliquot (between 1mL and 50mL, typically 50mL) into a pre-dried evaporating beaker using a graduated cylinder. Record the volume in Electronic Logbook.

12.6.6 Place the samples to dry in the oven overnight at 103-105°C. (Overnight is a period of time $\geq$ 8 hours.)

12.6.7 After drying overnight, remove from oven and place into the desiccator to cool. After the samples have cooled, obtain the weight of the samples as per Section 13.5, and record in Electronic Logbook.

12.6.8 If Total Volatile Solids (TVS) are also to be performed proceed to Section 12.9. Note: TVS utilizes a different LCS than the TS.

12.7 **Total Suspended Solids Determination (TSS)**

12.7.1 Record Filter IDs and weights in Electronic Logbook for samples and batch QC.

12.7.2 Method blank (MB) – Using a graduated cylinder measure out 1000mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.7.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of Alpha-Trol (or equivalent); record the volume in Electronic Logbook.

12.7.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.7.5 For wastewater, the Pace Analytical Services, Inc. - Green Bay Laboratory uses 500mL in order to meet Wisconsin Reporting Limit requirement of 2.0 mg/L.

12.7.6 For South Carolina work a sample volume is chosen to yield between 2.5 and 200 mg dried residue. If volume filtered fails to meet minimum yield, increase sample volume up to 1 L.

12.7.7 Shake the sample thoroughly and measure the chosen aliquot using a graduated cylinder.

12.7.8 Prepare the filter system with a fresh pre-weighed filter. Handle the filter with forceps only. Wet the filter with a small volume of Nanopure water to seat it. Discard the rinse water.

12.7.9 Filter the sample and wash the filter 3 times with approximately 10mL of Nanopure water. Maintain the filter vacuum for about 1 minute after filtration is complete.

12.7.10 Remove the filter with forceps and place it back into the evaporating dish.

12.7.11 Place the samples (filters and residue) to dry in the oven a minimum of 3 hours at 103-105°C.

12.7.12 After drying, remove from oven and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.7.13 If Total Volatile Suspended Solids (TVSS) are also to be performed proceed to Section 11.10. TVSS utilizes a different LCS than TSS.

12.8 **Total Dissolved Solids (TDS)**

12.8.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.8.2 Method blank (MB) – Using a graduated cylinder measure out 50mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.8.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of APG Solids APG plus Standard (or equivalent), record the volume in Electronic Logbook.

12.8.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.8.5 Shake the sample thoroughly and measure a 50mL aliquot using a graduated cylinder.

12.8.6 Prepare the filter system with a fresh filter. Handle the filter with forceps only. Wet the filter with a small volume of Nanopure water to seat it. Discard the rinse water.

12.8.7 Filter the sample and transfer the filtrate to a pre-weighed dried evaporating beaker.

12.8.1 Place the samples to dry in an oven that has it temperature under a program. The oven starts out at <100 degrees C and increases in temperature according to the program until it reaches 178 - 182 degrees C. It is then held there for a minimum of 1 hour.

12.8.2 After drying overnight, remove from oven and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.9 **Total Volatile Solids (TVS)**

12.9.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.9.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.9.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.9.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.9.5 Sample

12.5.4.3 For Solids Samples – Weight out 1 to 1.5g of sample in a pre-dried, tared evaporating beaker. Record the weight in Electronic Logbook.

12.5.4.4 For Aqueous Samples - Shake the sample thoroughly and measure an aliquot (between 1mL and 50mL, typically 50mL) into a pre-dried evaporating beaker using a graduated cylinder. Record the volume in Electronic Logbook.

12.9.6 Follow TS Section to obtain a dried residue.

12.9.7 Place the evaporating beaker or filter and evaporating dish into the muffle furnace at **550°C** for 1 hour.

12.9.8 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.10 Total Volatile Suspended Solids (TVSS)

12.10.1 Method blank (MB) – Using a graduated cylinder, measure out 1000mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.10.2 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.10.3 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.10.4 Shake the sample thoroughly and measure the chosen aliquot (see sections 11.7.5 and 11.7.6) using a graduated cylinder. Record the volume in Electronic Logbook.

12.10.5 Follow TSS Section to obtain a dried residue.

12.10.6 Place the filter and evaporating dish into the muffle furnace at **550ºC** for 1 hour. Write the filter number on the evaporating dish – paper label will burn off.

12.10.7 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5 in Electronic Logbook.

12.11 Fractional Organic Carbon (FOC) and Organic Matter (OM)

12.11.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.11.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pour into a tared evaporating beaker, record the weight in Electronic Logbook.

12.11.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker, and record the weight in Electronic Logbook.

12.11.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.11.5 Samples – Into a tared evaporating beaker weigh out about 1-1.5g of sample. Record the weight in Electronic Logbook.

12.11.6 Follow TS Section to obtain a dried residue.

12.11.7 Place the evaporating beakers into the muffle furnace at **440ºC** for 1 hour.

12.11.8 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5 in Electronic Logbook.

12.12 **Entering Results into Horizon**

12.12.1 Post samples and batch QC using Electronic Logbook.

12.12.2 Evaluate batch QC in Horizon.

13. Quality Control

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 Method Blank (MB) – A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples.

13.2.1 TS, TDS, TVS, FOC, and OM all use 50mL of deionized water for the method blank.

13.2.2 TSS and TVSS both use 1000mL of deionized water for the method blank.

13.2.3 For tests that are reported in %, the MB is evaluated in mg/Kg. The mg/kg result must be less than 20 mg/kg. Data can be reported if the sample mg/Kg result is 10x greater than the method blank. The value reported in Epic Pro will be adjusted to 0%.

13.2.4 For tests that are reported in mg/L the absolute value must be < PRL.

13.2.5 When measurements are above the PRL, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples analyzed since the last compliant calibration blank

13.2.6 If the analyte is detected in the method blank between the MDL and the PRL, then samples need to be qualified with a "B" when MDL reporting is required and sample results are greater than the MDL and less than 10 times the absolute value detected in the blank. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample-by-sample basis. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

13.3 Laboratory Control Sample (LCS) – The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples.

13.3.1 ERA DataPacK Total Volatile Solids Standard, Cat. # 913 is used for TVS, FOC, and OM analysis. Equivalent can be used.

13.3.2 APG Solids APG Plus standard, Cat #4033 is used for TDS analysis. Equivalent can be used.

13.3.3 NSI Solutions, Cat # QCI-084 for TS, TSS, and TDS analysis, or equivalent. Equivalent can be used.

13.3.4 A Laboratory Control Spike Duplicate (LCSD) must be analyzed if the client requests one.

13.3.5 The LCS recovered concentration must be within calculated in-house limits or default limits of $\pm$ 20%.

13.3.6 For tests that are reported in %, the LCS is evaluated in mg/L.

13.3.7 When measurements are outside the control limits, check for errors in calculations, standards preparation, and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.3.8 If no errors are found, sufficient sample is available, it is within hold, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still outside the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.3.9 If sufficient sample volume is not available or the samples are now outside of hold, report the sample data with a qualifier flag (L0) on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.3.10 When an LCSD is performed, the precision between the LCS and LCSD must be ≤10% RPD.

13.3.11 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.3.12 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.4 Duplicate – Analyze one sample in duplicate per 10 samples or less. The sample used for the duplicate is selected at random, unless specified by the client. The relative percent different (RPD) between duplicates must be ≤10%.

13.4.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.4.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an "R1" data qualifier

13.5 Constant Weight – Place the samples back into the 103-105ºC oven, and/or 440/550ºC muffle furnace for 15 minutes and repeat cooling, weighing, and desiccating steps until weight change is ≤0.0005g or the weight loss between the two weights is less than 4%.

13.6 Balance – The balance must be verified daily across the range of use, before use, as per the most current version of SOP S-GB-Q-030 *Support Equipment*.

13.6.1 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

13.6.2 1g for TSS and TVSS with a tolerance of +/-0.0100g

13.6.3 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

13.7 Oven – The oven temperature must be within required specifications before placing samples into oven and the temp is recorded in the Electronic Logbook with the correction factor, as per the most current version of SOP S-GB-Q-030 *Support Equipment.*

13.8 Hold – When preparation of a sample exceeds 7 days past the time of collection, notify the project manager before proceeding. If a sample is run past 7 days after collection, flag the result with an "appropriate data qualifier and fill out a LabTrack report.

13.9 See attachments Table A and Table B for a summary of QC

14. Data Analysis and Calculations

14.1 Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS) in mg/L $= \frac{(A - B) \times 1000 \times 1000}{V}$

Where:	V = sample volume (mL)
Total Solids	A = final weight (dry weight of residue and dish, g) B = dish weight (g)
Total Suspended Solids	A = final weight (dry weight of residue and filter, g) B = filter weight (g)
Total Dissolved Solids	A = final weight (dry weight of residue and dish, g) B = dish weight (g)

14.2 Volatile Solids (TVS and TVSS mg/L) $= \frac{(A - D) \times 1000 \times 1000}{V}$

14.3 Volatile Solids (TVS %) $= \frac{(A - D) \times 100}{(A - B)}$

14.4 % FOC $= ((1-((D-B) / (A – B))) \times 100) \times (0.58)$

14.5 % OM = ((1-((D-B) / (A-B))) x 100)

Where:

A = dry weight (weight of dried residue and dish, g)
B = dish weight (g)
C = weight of wet sample and dish (g)
D = muffled weight (weight of residue and dish after ignition, g)
V = volume (mL)

14.6 Precision - The relative percent difference (RPD) is calculated as:

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

Where: **S** = sample value
D = duplicate value

14.7 Constant Weight is calculated as:

$$\textbf{Constant Weight (\%)} = \frac{|FW - CW| \ * 100}{(FW + CW)/2}$$

Where: FW = First Weighing, mg

CW = Confirmation Weighing, mg

If FW – CW is 0.0005 g or less, then there is not a need to calculate as a percentage

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See Section 13 and Table 3: Quality Control, and Table 4: Data Assessment for a listing of Quality Control Criteria.

Table 5.
QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SM 2540G – ASTM D2974-87** **Frequency**	**Acceptance Criteria**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	For solid samples the MB must be < 20 mg/Kg and is reported as 0%. For aqueous samples the MB absolute value must be <RL. Detections > MDL but < RL must be evaluated for data qualification.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. None performed on TVSS.	Recovery must be within +/- 20% of the true value. RPD must be < 10%
Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.	RPD must be < 10%

Table 6.
ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**SM2540G – ASTM 2974-87 If these conditions are not achieved ⇒**
Method Blank	• 1
Duplicate	• 2
Accuracy & Precision Laboratory Control Spikes	• 3

1. In the absence of project specific requirements, sample detects less than 20 times the method blank contamination level are reported with the appropriate data qualifier. Sample detects greater than 20 times the method blank contamination are reported without qualification.
2. In the absence of project specific or method requirements, in-house generated limits will be used. If the DUP fails precision control limits flag the parent with the appropriate data qualifier.
3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate qualifier qualifiers to reflect the deficiencies.

16. Corrective Actions for Out-of-Control Data

16.1 See Section 13 and Table 6.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 If not specifically listed in Section 13 or Table 6, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers.

18. Method Performance

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 Demonstration of Capability (DOC) – Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC). DOC's must be repeated annually. Analyze 4 replicate environmental sample aliquots. Calculate the mean and standard deviations of the results. The relative percent different (RPD) between replicates must be $\leq$ 5% or the study must be repeated.

19. Method Modifications

19.1 Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1 Maintain the analytical balance, ovens, and furnaces according to the most current revision of SOP S-GB-Q-030, *Support Equipment.*

21. Troubleshooting

21.1 Refer to the support equipment operating manual.

22. Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001 *Waste Handling and Management* most current revision or replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. References

25.1 Pace Analytical Quality Assurance Manual (most current revision or r*eplacement).*

25.2 *T*he NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (most current revision or replacement).

25.3 Method 2540-G, Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992.

25.4 ASTM D2974-87, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, May 29, 1987

25.5 VA30007.00.004, Recommendations Regarding the Sampling and Analysis of Fractional Organic Carbon (FOC) in Soils, Technical Decision Compendium, Rule OAC 3745-300-07, March 2000

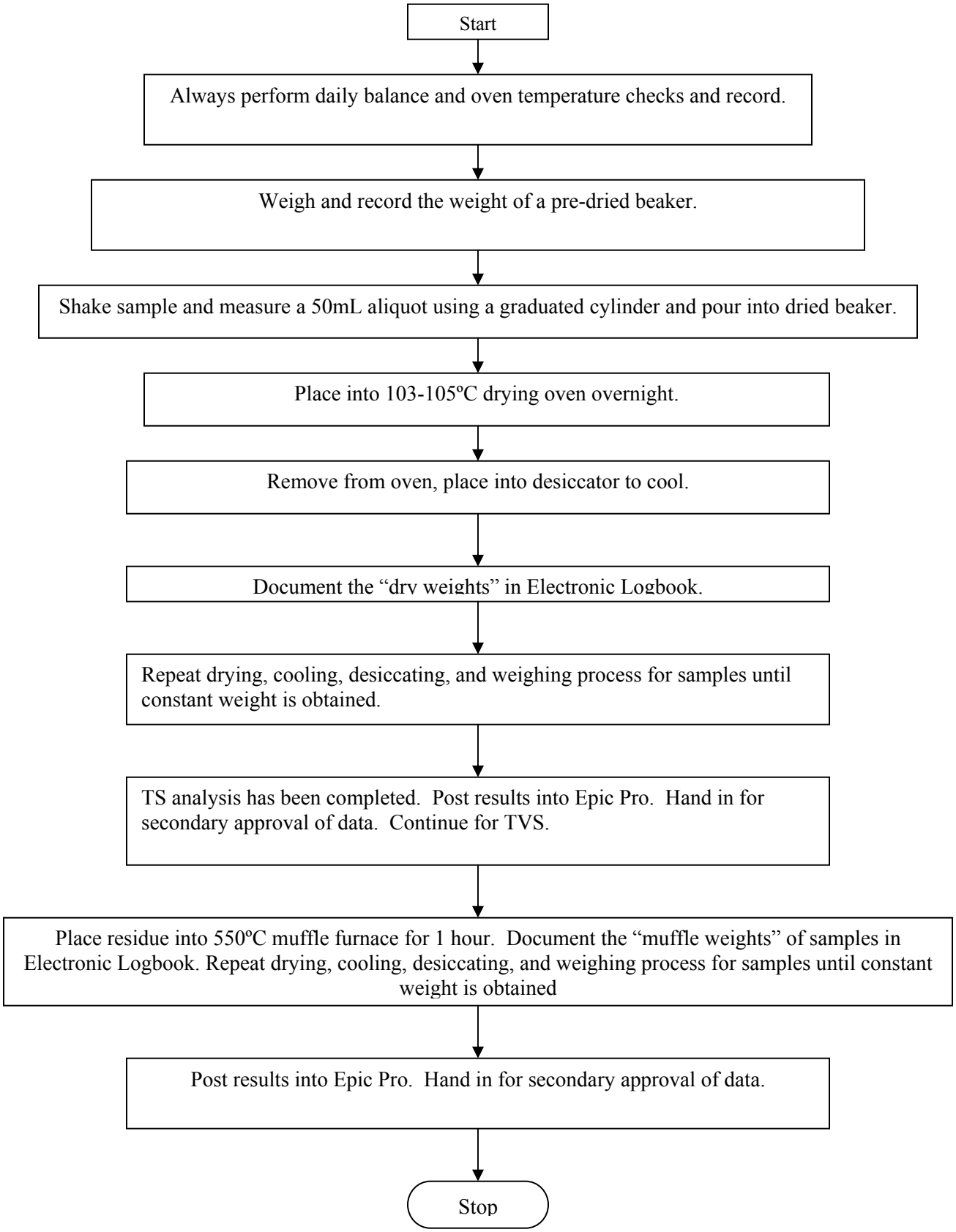
26. Tables, Diagrams, Flowcharts, Appendices, Addenda, etc.

26.1 Flow charts – Attachment I.

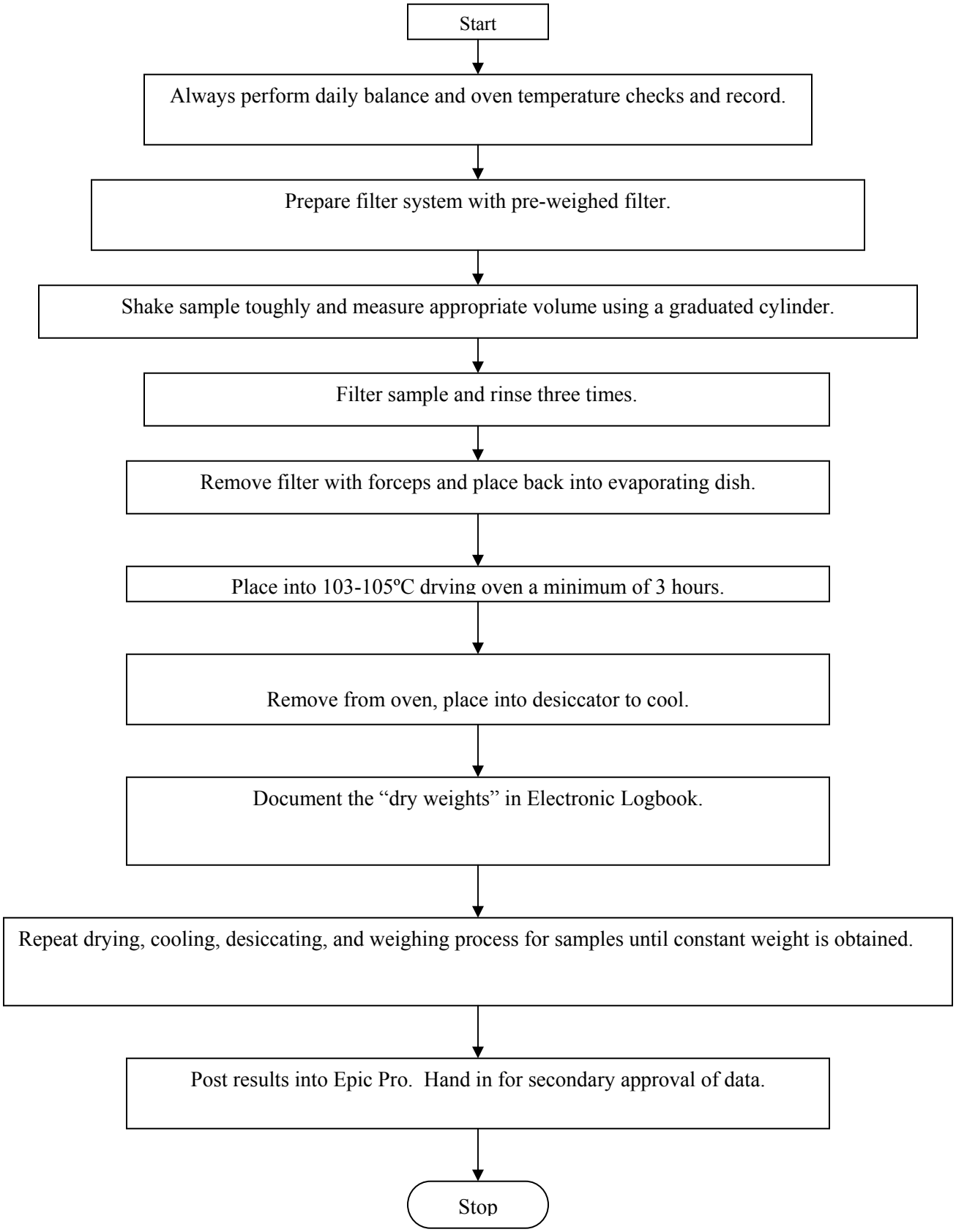
27. Revisions

Document Number	Reason for Change	Date
S-ALL-GB-I-014-Rev.1	Consolidated SOPs ALL-I-014-rev.1, ALL-GB-I-014-rev.1, and GB-I-050-Rev.0. Section 12.6.2 – added South Carolina minimum residue requirements.	24Aug2007
S-ALL-GB-I-014-Rev.2	Corrected FOC and OM calculations and definitions	05Oct2007
S-ALL-GB-I-014-Rev.3	Clarified analysis steps for each test. Changed qualifiers to match Epic Pro.	28Apr2008
S-ALL-GB-I-014-Rev.4	Included notations on how to evaluate MB and LCS for tests reporting in %. Updated Signature Page to Periodic Review.	29May2009
S-GB-I-068-Rev.00	Updated numbering format to match SOP: S-ALL-Q-003, Document Numbering Changed Qualifier from & to L0 and * to R1.	21Sept2010
S-GB-I-068-Rev.01	Updated SOP format to be consistent with SOP: S-GB-Q-017 *Preparation of SOPs* Updated SOP references throughout document. Throughout document: Changed LimsLink to Electronic Prep Log. Sections 2, 3.2, 8.1.2: Removed information relating to Fixed Solids (FS). Sections 2, 3.3, 12.11: Removed information relating to ASH. Section 12.8.1: Changed temperature program to ramp up to 100 deg C. Section 12.10.2: Added LCS requirement to TVSS methods. Tables A and B: Moved and renamed in appropriate sections of SOP.	29Nov2012

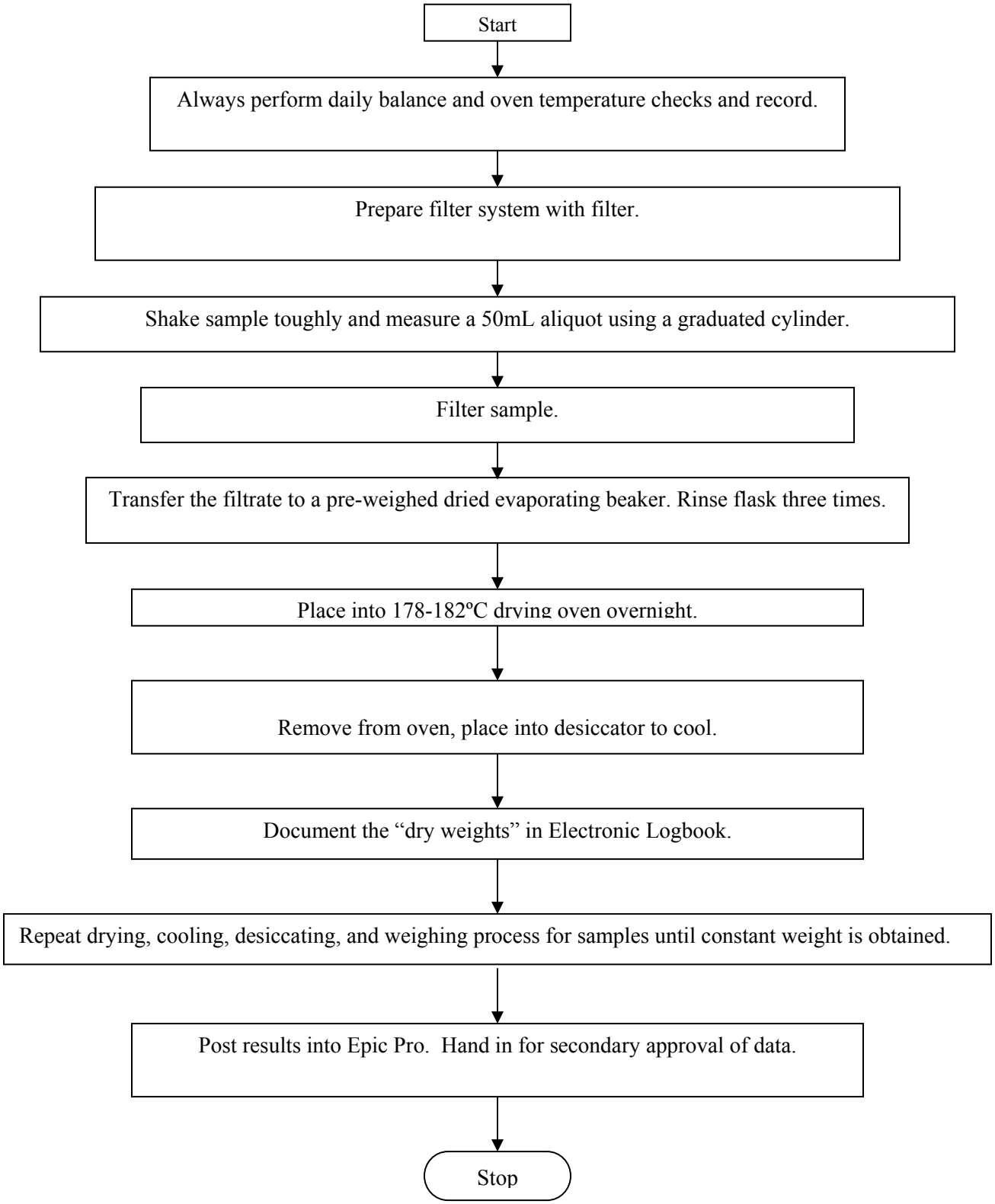
Attachment I – TS and TVS FLOWCHART



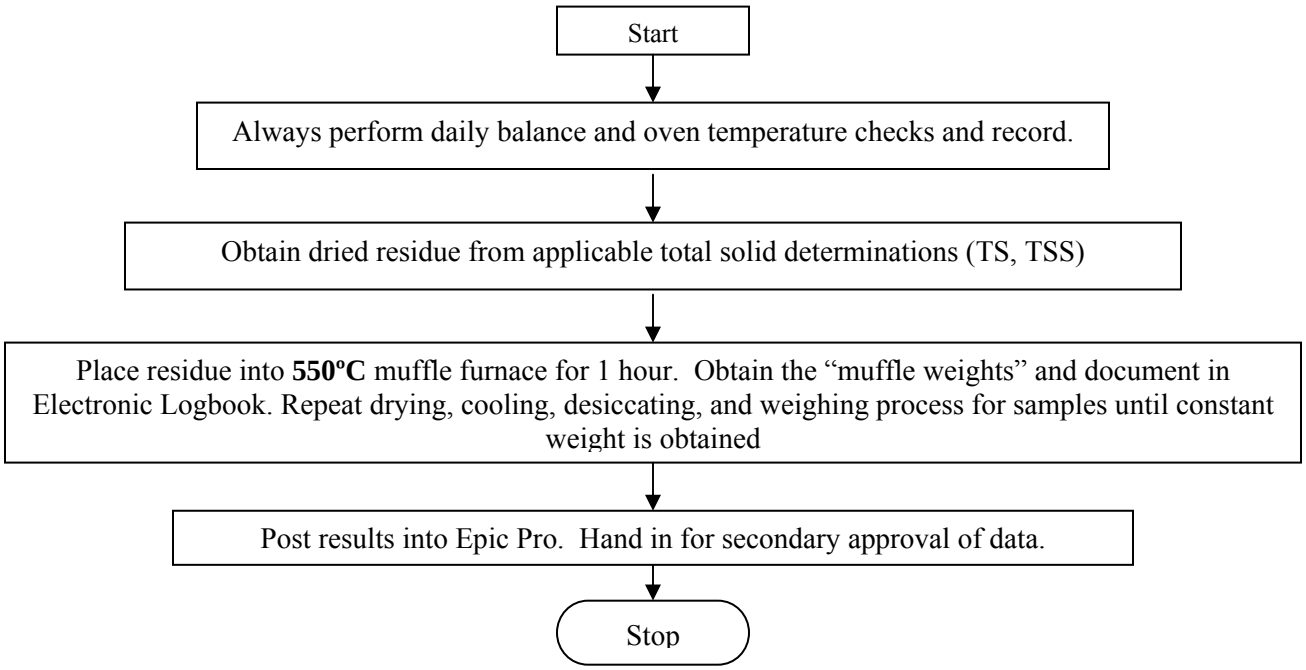
Attachment I – TSS FLOWCHART



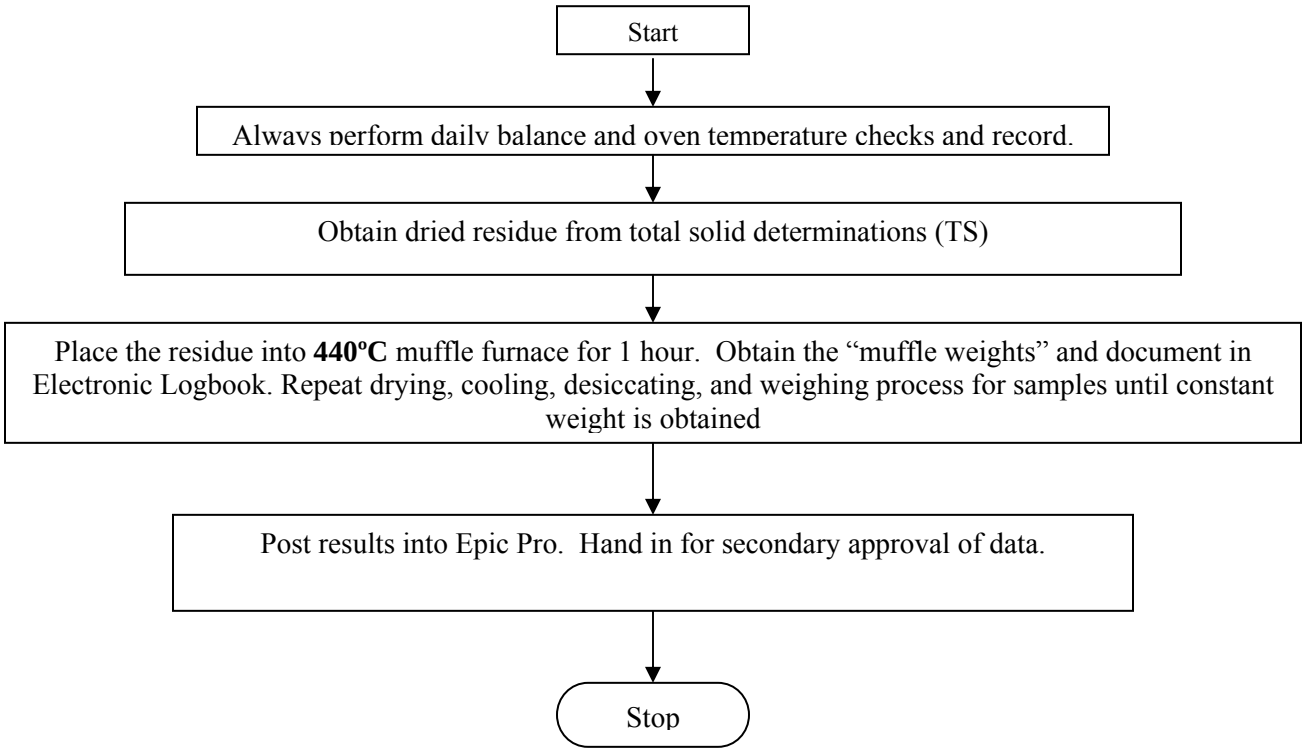
Attachment I – TDS FLOWCHART



Attachment I – TVS and TVSS FLOWCHART



Attachment I – OM and FOC FLOWCHART



APPENDIX E

GZA Field and Equipment SOPs

Equipment SOP 1
ppbRAE 3000/MiniRae 3000 Portable VOC Monitor

1.0 Scope and Application

1.1 The ppbRae 3000/MiniRAE 3000 Portable VOC Monitor is a compact monitor designed as a broadband VOC gas monitor and data logger for work in hazardous environments. It monitors VOCs using a Photo-Ionization Detector (PID) with a 9.8 eV, 10.6 eV, or 11.7 eV gas discharge lamp. It gives real-time measurements and activates alarm signals whenever the exposure exceeds pre-set limits. Prior to factory shipment, the ppbRae 3000 is pre-set with default alarm limits and the sensor is pre-calibrated with standard calibration gas. After the monitor is fully charged, it is ready for immediate operation.

1.2 The rechargeable battery can run up to 16 hours of continuous monitoring. The instrument is designed to continuously monitor VOC vapor at ppb levels. Pre-set alarm thresholds are set for STEL, TWA, low- and high-level peak values. Audio buzzer and flashing LED display are activated when the limits are exceeded. The instrument has a 260,000-point data logging storage capacity for data download to a personal computer.

1.3 The ppbRAE 3000/MiniRAE 3000 consists of a PID with associated microcomputer and electronic circuit. The unit is housed in a rugged case with a backlit LCD and three keys to provide easy user interface. It also has a built-in flashlight for operational ease in dark locations.

2.0 Health and Safety Warnings

2.1 For safety reasons, this equipment must be operated and serviced by qualified personnel only. Read and understand the instruction manual completely before operating or servicing.

2.2 Use only RAE Systems battery packs, Part Nos. 059-3051, 059-3052, or 059-3054. This instrument has not been tested in an explosive gas/air atmosphere having an oxygen concentration greater than 21%. Substitution of components may impair intrinsic safety. Recharge batteries only in non-hazardous locations.

3.0 Cautions

3.1 To reduce the risk of electric shock, turn off the power before removing the monitor cover. Disconnect the battery before removing the sensor module for service. Never operate the monitor while the cover is removed. Remove the monitor cover and sensor module only in an area known to be non-hazardous.

3.2 The monitor equipment is classified as to intrinsic safety for use in class I, division 1, groups A, S, C, D, or non-hazardous locations only.

4.0 Personnel Qualifications

4.1 Read and understand the ppbRAE 3000/MiniRAE 3000 User's Manual.

5.0 Apparatus and Materials

5.1 The main components of the ppbRAE 3000/MiniRAE 3000 Portable VOC monitor include:

- Three keys for the user to interact with the monitor: one operation key and two programming keys for normal operation or programming of the monitor.
- LCD display with back light for direct readout and calculated measurements.
- Buzzer and red LEDs for alarm signaling whenever the exposures exceed pre-set limits.
- Wrist strap.
- Charge contact for plugging directly to the charging station.
- Gas entry and exit ports.
- Serial communication port for PC interface.
- External alarm and analog output port.
- Protective rubber cover.
- Button for flashlight to assist view in dark areas.

6.0 Measurement Range and Resolution

6.1 The MiniRAE 3000 has the following measurement range and resolution for the various lamps:

Lamp	Range	Resolution
10.6 eV	0.1 ppm to 15,000 ppm	0.1 ppm
9.8 eV	0.1 oom to 5,000 ppm	0.1 ppm
11.7 eV	0.1 ppm to 2,000 ppm	0.1 ppm

6.2 The ppbRAE 3000 has the following measurement range and resolution for the various lamps:

Lamp	Range	Resolution
10.6 eV	0.001 ppm to 10,000 ppm	0.001 ppm
9.8 eV	0.01 oom to 5,000 ppm	0.01 ppm
11.7 eV	0.01 ppm to 2,000 ppm	0.01 ppm

7.0 Summary of Method

7.1 To turn on the ppbRAE 3000/miniRAE 3000 portable VOC monitor, press [MODE] key for one second and release. The audio buzzer will beep once and the air pump will turn on. The display will show "ON! ..." and then "Ver n.nn" to indicate the unit's current firmware version number. Next displayed are the serial number, the model number, Operating Mode, current date and time, unit internal temperature, gas selected, high low, STEL, TWAlAVG alarm limits, battery voltage, and shut-off voltage. Also displayed are internal mode settings such as User mode, Alarm mode, datalog time remaining and log periods in the respective order.

7.2 For field screening soil samples, refer to Field SOP 2.

7.3 To turn off the ppbRAE 3000/MiniRAE 3000 portable VOC monitor, press and hold the [MODE] key for 5 seconds. The monitor will beep once per second during the power-down sequence with a countdown timer showing the number of remaining seconds. The message "Off! " flashes on the LCD display and the display will go blank indicating that the monitor is turned off.

7.4 When the monitor is turned off, all the current real-time data, including last measured value, are erased. However, the datalog data is preserved in non-volatile memory. Even if the battery is disconnected, the datalog data will not be lost. While the power is off, the real time clock will continue to operate until the battery is completely drained (usually in four to five days without any charging). If the battery is completely drained or is disconnected from the monitor for more that 30 minutes, the real time clock will be lost. In this case, the user needs to enter the real time clock information again, as described in Section 4, or send the PC clock during configuration through the PC communication.

8.0 Instrument Calibration/Maintenance

8.1 Fresh Air Calibration

8.1.1 The first sub-menu shows: "Zero Calibration"

8.1.2 If you have pressed [Y/+] to enter Zero calibration, then you will see this message: "please apply zero gas…"

8.1.3 Make sure that the ppbRAE 3000/MiniRAE 3000 is connected to one of the "fresh" air sources.

8.1.4 Press the [Y/+] key, the display shows "zeroing" followed by a countdown timer.

8.1.5 After about a 30-second pause, the display will show the message "zeroing is done! Reading = x.x ppm..." Press any key or wait about 20 seconds, the monitor will return back to "Fresh air Calibration?" submenu.

Note: The charcoal filter has a check box so that the user can mark off a box each time the filter has been used. The charcoal filter should be replaced after four calibrations.

8.2 Span Calibration

This procedure determines the second point of the sensor calibration curve for the sensor. A cylinder of standard reference gas (span gas) fitted with a 500 cc/min flow-limiting regulator or a flow-matching regulator is needed to perform this procedure. Choose 500 cc/min regulator needed to perform this procedure. Choose 500 cc/min regulator only because the flow rate matches the flow rate of the pump inside. Alternatively, the span gas can first be filled into a Tedlar Bag. Connect the calibration adapter to the inlet port of the ppbRAE 3000/MiniRAE 3000 monitor, and connect the tube to the regulator or Tedlar bag.

8.2.1 Make sure the monitor is connected to one of the span gas sources described above.

8.2.2 Press the [Y/+] on "Span Calibration" to start the calibration. The display shows the gas name and the span value of the corresponding gas.

8.2.3 Turn on the valve of the span gas supply. Press [Y/+] to initiate calibration.

8.2.4 Display shows "calibrating… 30" with a countdown timer showing the number of remaining seconds while the monitor performs the calibration.

8.2.5 To abort the calibration, press any key during the countdown. The display shows "Aborted!" and return to "Span Calibration" sub-menu.

8.2.6 When the countdown timer reaches 0, the display shows "Span 1 is done! Reading = x.x ppm".

Note: The reading should be close to the span gas value.

8.2.7 During calibration, the monitor waits for an increased signal before starting the countdown timer. If a minimal response is not obtained after 35 seconds, the monitor displays "No Gas!" Check that the span gas valve is on and for lamp or sensor failure before trying again.

8.2.8 Turn off the flow of gas. Disconnect the calibration adapter or Tedlar bag from the ppbRAE 3000/MiniRAE 3000 monitor.

8.3 Maintenance

The major maintenance items of the ppbRAE 3000/MiniRAE 3000 are (note: maintenance should be performed by qualified personnel only):

- Battery pack.
- Sensor module.
- PID lamp.
- Sampling pump.
- Inlet connectors and filters.

8.3.1 Battery charging and replacement

When the display shows a flashing message "Bat," the battery requires recharging. It is recommended to recharge the ppbRAE 3000/MiniRAE 3000 monitor upon returning from fieldwork. A fully charged battery runs a ppbRAE 3000/MiniRAE 3000 monitor for 16 hours continuously. The built-in charging circuit is controlled by the micro-controller to prevent over-charging. The battery may be replaced in the field (in area known to be non-hazardous), if required. Refer to the manual for instructions on Replacing the Battery Pack and Replacing the Alkaline Battery adapter.

9.0 Troubleshooting

PROBLEM	POSSIBLE REASONS AND SOLUTIONS
Cannot turn on power after charging the battery	**Reasons:** Discharged battery. Defective battery. **Solutions:** Charge or replace battery.
Lost password	**Solutions:** Call Technical Support at +1 408-752-0723 or toll-free at +1 888-723-4800
Reading abnormally high	**Reasons:** Dirty sensor module. Dirty filter. Excessive moisture and water condensation. Incorrect calibration **Solutions:** Replace filter. Blow-dry the sensor module. Calibrate the unit

PROBLEM	POSSIBLE REASONS AND SOLUTIONS
Buzzer inoperative	**Reasons:** Bad buzzer. **Solutions:** Check that buzzer is not turned off. Call authorized service center.
Inlet flow too low	**Reasons:** Pump diaphragm damaged or has debris. Flow path leaks. **Solutions:** Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Call Technical Support.
Reading abnormally low	**Reasons:** Incorrect calibration. Dirty filter. Dirty sensor module. Weak or dirty lamp. Air leakage. **Solutions:** Remove calibration adapter. Calibrate the unit. Check for air leakage. Replace filter.
"Lamp" message during operation	**Reasons:** Lamp drive circuit. Weak or defective PID lamp, defective. **Solutions:** Turn the unit off and back on. Replace UV lamp.

10.0 References

- MiniRAE 3000 User's Guide, Document No.: 059-4020-000. Revision C. August 2010.

- ppbRAE 3000 User's Guide, Document No.: 059-4021-000. Revision C. August 2010.

Equipment SOP 2
Heron Dipper-T (200 feet) Water Level Indicator

1.0 Scope and Application

The Heron Dipper-T water level indicator is used to measure the water level and depth of monitoring wells. This instrument is composed of an approximately 200-foot long, ⅜-inch, polyethylene tape connected to a 7-inch long stainless steel probe that is approximately ⅝-inch wide. The tape is marked in tenths, hundredths and 1-foot increments. The tape has stainless steel conductors embedded in the plastic. The probe is made of stainless steel.

2.0 Apparatus and Materials

- Water Level Indicator.
- Extra 9-Volt Battery.
- Deionized (DI) Water.
- Wipes.
- Logbook/Field Notes.

3.0 Summary of Method

3.1 Operating Procedures

3.1.1 Depress the small, white button to check battery and tone level. Adjust by turning the dial.

3.1.2 Lower the probe into the well. The probe will "buzz" when water is reached. Be sure to record the reading.

3.1.3 To get a well depth measurement, turn off the sound and lower into the well until the bottom is reached. Record the reading.

3.1.4 For additional detail on measuring water levels, refer to Field SOP 5.

4.0 Instrument Calibration/Maintenance

4.1 No calibration is necessary.

4.2 Care of the meter and probe.

4.2.1 After measuring the water level and/or total depth in a monitoring well, carefully wind the tape while wiping down with a wipe moistened with DI water.

4.2.2 For more thorough cleaning, unwind the tape and probe and wash with a mild detergent. The tape and probe may be cleaned and degreased with the following: soap solution, naphtha alconox 10%, Joy detergent 10%, and isopropyl or isobutyl alcohols. Rinse the tape and probe thoroughly with water after washing is complete.

4.3 Battery Replacement

4.3.1 Use an alkaline 9-volt battery.

4.3.2 The battery is housed in a battery box in the reel hub. To remove, unscrew the faceplate screws and remove the battery box cover.

4.3.3 Replace the battery then replace the battery box cover onto the reel.

5.0 Troubleshooting

SYMPTOM	CAUSE	REMEDY
No sound when the unit is tested	Dead battery.	Replace battery.
	Broken wire in tape.	Locate break, splice and seal.
	Disconnected wire inside probe.	Contact manufacturer.
Continuous sound after probe is removed from water	Probe tip is dirty.	Clean probe thoroughly, as specified in 4.2.2 above.
	Excess moisture is present on back of the electronic panel.	Wipe the back of the electronic panel with a dry, clean cloth.
	Probe/tape connection may have a break or leak.	Contact manufacturer.

6.0 References

- Operation/Maintenance Instructions and Warranty for Dipper-T, Little Dipper, Skinny Dipper, Water Tape and Potable Water Meter Water Level Indicators, published by Heron Instruments, Inc.

- Product Brochure – Heron Instruments Inc.

Field SOP 1
Surface and Subsurface Soil Sample Collection

1.0 Purpose

The methods and equipment used for soil sampling depend on the sample depth, type of sample and type of soil. The sampling equipment must be constructed of material that is compatible with the sample media and the analyte of interest. Subsurface soil may be sampled using conventional drilling and split-spoon methods, direct-push methods such as Geoprobe® techniques, or using a backhoe.

2.0 Equipment

2.1 Subsurface Soil Sampling

Subsurface soil will generally be collected from depths greater than 24 inches below natural ground surface. Collection of subsurface soil samples can be accomplished using:

- Conventional drilling and split-spoon methods;
- Direct push Geoprobe® methods;
- Hand auger methods; and
- Test pit excavation methods

2.2 Soil sampling equipment includes the following:

- Safety equipment, as specified in the HASP;
- Tape measure;
- Survey stakes or flags;
- Ziploc™ plastic bags;
- Labels;
- Chain-of-custody records;
- Coolers;
- Ice; and
- Decontamination supplies/equipment.

3.0 Procedures

3.1 Subsurface Soil Sampling (using split-spoon, direct push macro-core, or hand auger)

The procedure for split-spoon or direct push sampling using a macro-core describes the extraction of undisturbed soil cores of 18 to 60 inches in length. A series of consecutive cores may be sampled to give a complete soil column, or an auger may be used to drill down to the desired depth for sampling. The split-spoon is then driven to its sampling depth through the bottom of the augured borehole and the soil core extracted. A direct push Dual-Tube sampler, as is used in Geoprobe® boring techniques, is pneumatically driven to its sampling depth and the soil core (generally contained in an internal acetate liner of the sampler) is extracted. The split-spoon sampling device may be used to collect information such as soil density. All work should be performed in accordance with ASTM D1586-08a, Penetration Test and Split Barrel Sampling of Soils.

3.2 Pre-Job Preparation:

- Determine the extent of the sampling effort, the sampling methods to be employed and the types and amounts of equipment and supplies required.

- Ensure that appropriate sample bottleware has been procured for the required routine and quality control (QC) sample analyses and that the analytical laboratory has been contacted and is prepared to receive the samples.

- Obtain utility clearance for subsurface excavation/penetration locations, as required.

- Use stakes, flagging, or buoys to identify and mark all sampling locations.

Subsurface soil samples are collected using the following procedures:

- Prior to sampling, it is important to ensure that all sampling locations are clear of buried utility lines.

- Place the split-spoon, Dual-Tube and/or hand auger sampler in a perpendicular position on the sample material.

- The split-spoon sampler should be driven by a 140-pound weight falling freely 30 inches, and the macro-core sampler pneumatically hammered to the desired sample depth; for the split-spoon 24-inch intervals is the norm and for the macro-core sampler, 48 or 60 inches is the norm. Hand augers are limited and should be advanced to the targeted depth for sample retrieval.

- Record the number of blows to drive each 6-inch interval on the boring log to the desired sample depth if using split-spoon methods.

- Withdraw the split-spoon and/or Dual-Tube sampler from the borehole and open by unscrewing the bit and head of the sample device to access the internal soil sample. If a split sample is desired, a clean, stainless steel knife should be used to divide the sampler's contents in half, lengthwise. The contents of the hand auger should be emptied onto a clean plastic sheet for description and sampling.

- Place a portion of the soil sample in a plastic bag for field screening of volatile organic vapors in accordance with Field SOP 2, and record all pertinent data on the soil boring logs.

- Collect a representative soil sample from the targeted sample interval and fill the appropriate laboratory-provided glass containers with soil for the prescribed analysis.

- Place the soil samples on ice and deliver samples to the laboratory for analysis under chain-of-custody pursuant to Field SOP 7.

- Follow proper decontamination procedures pursuant to Field SOP 3.

3.3 Borehole Abandonment:

Boreholes exceeding 2 feet in depth should be backfilled and sealed with bentonite pellets (holeplug). For borings less than 2 feet, backfilling the boring with native soil materials (non-impacted) is acceptable.

If bentonite pellets are used, the pellets should be poured slowly into the borehole to avoid "bridging." Bentonite should be placed from the bottom of the boring to approximately 2 feet from the ground surface. Sufficient potable water should be added to hydrate the bentonite.

4.0 Soil Sample Preservation

Dry weight samples do not require preservation. Those which will be tested for VOCs require methanol preservation in accordance with USEPA Method 5035A:

- Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Always wear gloves whenever handling the sample vials.

- Using the sample collection device provided, collect approximately 10 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere.

- Using the sample collection device, add about 10 g (4 to 6 cm) of soil to the vial.

- Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store the sample on ice at 4° C.

5.0 Records and Documentation

Soil samples will be described and classified in general accordance with the USCS (equivalent to ASTM methods D2487 and 2488). Sample descriptions and classifications will be recorded on the sample collection log and/or soil boring log. A soil sample collection log will be completed for each sample submitted to the laboratory for analysis. For each sample, record sample identification (location, depth, etc.), time of collection, time of VOC vapor measurement and maximum concentration measured by the PID. Subsequent to completion of surface soil sampling, the sample hole will be backfilled with native material, or in the case of deeper subsurface soil borings (greater than 24 inches in depth), backfilling with dry or hydrated bentonite hole plug. To the extent feasible, the area will be restored to its previous condition.

6.0 Special Notes

The objective of QA/QC is to identify and implement methodologies that limit the introduction of error into sampling and analytical procedures.

Field SOP 2
Soil Screening for Organic Vapors

1.0 Purpose

This procedure is to set guidelines for performing a field screening method that measures for the presence of VOCs in soils. It is intended to be a field method used while drilling and sampling.

2.0 Equipment and Materials

- Ziploc™ bags;
- Trowels or small shovels;
- Indelible marking pen; and
- PID such as a ppbRAE 3000 or MiniRAE 3000, with appropriate UV lamp (10.0 eV, 10.6 eV, or 11.8 eV), calibration gases and other required items for PID use.
    - Check with the Project Manager before conducting field work to confirm the lamp size to be used on any given assignment.

3.0 Procedures

- Soil samples for headspace analysis may be collected using the following sampling techniques:
    - Hollow-stem auger split-spoon sampling;
    - Direct-push Geoprobe® sampling;
    - Collection of soil off the augers (auger cutting);
    - Excavated test pits; or
    - Shallow surface sampling using a trowel or hand auger.
- The PID will be calibrated daily, as needed through the day, and at the end of the day. Refer to GZA field SOP for discussion of calibration and use of a PID; also refer to the manufacturer's manual.
- Each soil sample shall be handled using a new pair of disposable gloves and/or a decontaminated trowel. A soil sample is placed in a Ziploc™ bag until it is half full (creating a vacant headspace in the bag above the sample). The Ziploc™ portion of

the bag is then sealed. The sampled interval, boring identification number and sampling time are marked on the bag.

- Vigorously shake the sample for 15 seconds and store in a near room-temperature (70° F) environment for at least 10 minutes, but not more than 2 hours. This ensures that VOCs volatize uniformly from the soil into the bag headspace. When ambient temperatures are below 32° F, headspace samples should be stored in a heated vehicle or building.

- The sample should also be shaken just prior to taking the VOC measurement to release any trapped volatiles within the soil. Because of the possibility for high PID readings, the instrument should be set to read maximum VOC levels. The VOC measurement is taken by gently inserting the sample probe through a small opening in the corner of the Ziploc™ bag in order to read the air space above the soil. Do not allow the probe to contact soil contained within the bag. The PID reaches a peak response within approximately five seconds; therefore, the headspace reading should be taken between 5 and 10 seconds after inserting the probe into the bag.

- Record the maximum concentration measured by the PID onto a boring log, test pit log, or appropriate field data collection sheet as instrument units (IUs) above background.

- All sampling equipment, including trowels or shovels, shall be decontaminated prior to next use. All used bags shall be discarded.

4.0 Records and Documentation

Readings will be recorded on an applicable log sheet for the work being performed in accordance with the work plan. For the event, record ambient temperature, method of warming samples, weather conditions, PID model and lamp energy rating, calibration standards used and times and results of calibrations. For each sample, record sample identification (location, depth, etc.), time of collection, time of VOC measurement and maximum concentration measured by PID.

5.0 Special Notes

The air within the bag is often high in moisture content, which will often affect performance of the PID. Be aware for this occurrence and check the responsiveness and calibration of your meter frequently.

6.0 References

- ppbRAE 3000 User's Guide, August 2010 Rev. C, RAE Systems.

- MiniRAE 3000 User's Guide, August 2010 Rev. C, RAE Systems.

Field SOP 3
Decontamination

1.0 Purpose

This SOP is designed to provide a procedure for preventing or minimizing the potential for cross-contamination of environmental samples. This SOP focuses on small equipment decontamination (e.g., split spoons, hand augers, water level meters, oil water interface probes, sediment dredge tools, etc.), which combines both physical and chemical removal steps and also provides guidance for cleaning pumps and large items used by contractors (drillers, excavators). Removing or neutralizing contaminants from equipment not only minimizes the likelihood of sample cross-contamination, but reduces or eliminates transfer of contaminants to clean areas and prevents the mixing of incompatible substances.

Note that Project Sampling and Analysis Plans (SAPs) and/or Quality Assurance Project Plans (QAPPs) may call for collection of Equipment Blank samples following certain decontamination events. This SOP does not attempt to identify when or how such samples might be needed.

2.0 Equipment and Materials

The following is a list of equipment and material commonly used for decontamination:

- Non-phosphate detergent (alconox);
- 10% reagent-grade nitric acid with spray bottles;
- Pesticide-grade methanol with spray bottles;
- Tap water;
- Distilled/deionized water;
- Long- and short-handled brushes and bottle brushes;
- Drop cloth/plastic sheeting;
- Paper towels;
- Plastic or galvanized tubs or buckets;
- Pressurized sprayers;
- Aluminum foil;

- Ziploc™ plastic bags;
- Appropriate PPE (i.e., safety glasses, appropriate gloves, boots, as may be called for in HASP);
- SDS for any chemicals brought to the site; and
- Manufacturer's handling procedures.

3.0 Procedures

3.1 Small Equipment Procedures

Small equipment would consist of non-disposable equipment such as flow through cells, Grundfos pump, hand tools, etc.

The small equipment decontamination procedure is summarized as follows:

1. Physical removal;
2. Non-phosphate detergent wash;
3. Distilled/deionized water rinse;
4. 10% nitric acid rinse (for metals sample collection);
5. Distilled/deionized water rinse;
6. Pesticide-grade methanol rinse (for organics sample collection);
7. Distilled/deionized water rinse;
8. Air dry; and
9. Secure clean equipment.

Note that these steps are used when constituents of concern include metals, VOCs and petroleum compounds. Other rinses (hexane/acetone) would be appropriate if constituents of concern included PCBs, SVOCs, pesticides and/or herbicides.

<u>Physical Removal</u>

Fill a wash basin or a large bucket with non-phosphate detergent and tap water. First, remove any solids and contain these solids for disposal with other field residual wastes. Then, scrub equipment with soap and water using bottle brushes

and/or bristle brushes. Wash soap off of equipment with tap water from a low-pressure sprayer. Repeat as many times as necessary. If there is no acid or solvent rinse to follow, a final rinse should be done with distilled/deionized water from a low-pressure sprayer.

Sensitive equipment, which is not waterproof, should be wiped down – first with a damp, soapy wipe, followed by several damp DI-water wipes.

Nitric Acid

Follow this step if metals are a constituent of concern (otherwise skip this step). Complete physical removal step above. Then, using a spray bottle, rinse sampling equipment with nitric acid solution. Begin spraying (inside and outside) at end of the equipment, allowing the acid to drip to the other end into a 5-gallon bucket. Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Solvent Sprayer - Methanol

Follow this step if organics (VOCs or petroleum compounds) are constituents of concern (otherwise skip this step). Complete physical removal step above. Then, using a spray bottle, rinse sampling equipment with pesticide-grade methanol. Begin spraying (inside and outside) at one end of the equipment, allowing the solvent to drip to the other end into a 5-gallon bucket. Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Clean Equipment Drop

Lay clean equipment on clean plastic sheeting in the equipment laydown area or wipe down with a clean paper towel or cloth. After air drying or wiping dry, wrap sampling equipment with aluminum foil, plastic, place in storage container, or cover with other protective material.

3.2 Pump Decontamination Procedures

Decontaminating pumps must be done prior to first-use and between wells. It is generally expected that pumps being decontaminated will NOT have visible residue from usage in a well, except perhaps mud. In the event that product residue is visible on a pump or strong product odors are present, the field staff MUST DISCUSS WITH PROJECT MANAGER whether to attempt field decontamination of a pump. The Project Manager may decide to replace the pump rather than attempt decontamination.

Many pumps can be easily disassembled for decontamination and should be disassembled and then cleaned as per Section 3.1. This procedure assumes that no visible residue is present.

a. Physical removal - non-phosphate detergent wash:

- Submerse pump (intake) into the detergent wash (bucket), operate pump until the bucket is empty;

b. Tap water rinse:

- Submerse pump (intake) into the rinse water (bucket), operate pump until the bucket is empty;

c. 10% nitric acid rinse (when metals are target analyte);

d. Distilled/deionized water rinse;

e. Solvent rinse (pesticide-grade methanol when organics are target analyte);

f. Air dry; and

g. Secure clean equipment.

3.3 Decontamination of Drilling Equipment and Tools

Decontamination of contractor equipment must be done prior to first-use and between locations. It is the responsibility of GZA field staff to inspect all tools and equipment prior to start of work.

a. Larger equipment (i.e., drill rigs, augers, rods [including Geoprobe®], casing, etc.) are to be decontaminated first by dry-brushing as needed, then by using a steam cleaner or high pressure water sprayer. The physical removal, scraping, or dry-brushing steps will generate soils and cuttings wastes (solids) that must be contained/collected for disposal with other field residual wastes.

b. The drilling contractor will be required to construct a decontamination pad where decontamination activities are to be conducted. The decontamination pad must serve also as a leak-proof holding area for all decontamination and rinse waters.

c. GZA field staff are expected to observe equipment that has been decontaminated and to approve the decontamination effort or request further cleaning.

d. When decontamination of equipment is complete, all decontamination and rinse waters are to be pumped from the decontamination pad into 55-gallon drums.

3.4 Management of Decontamination Wastes

Refer also to the GZA SOP concerning the handling of Investigation-Derived Wastes.

- Solid Waste – Place all solid waste materials generated from the decontamination area (i.e., soils removed from augers, gloves and plastic sheeting, etc.) in an approved container.

- Liquid Waste – Empty used soap water, and acid together first into 5-gallon plastic bucket to dilute the acid. ALWAYS ADD ACID TO WATER (not water to acid) to avoid reaction. Then place solvent liquid wastes into the 5-gallon plastic bucket and store with lid on. Do not pour solvent waste and acid waste together without first diluting the acid waste with the used soap water. Pouring the solvent waste and acid waste together without dilution would result in a chemical reaction resulting in chemical fumes.

- Buckets and brushes must be evaluated for possible re-use or disposal, depending upon the condition and whether they can be suitably cleaned.

4.0 Records and Documentation

Decontamination procedures should be documented in the field log book.

5.0 Special Notes

All field activities must be carried out in accordance with a site-specific Health and Safety Plan. MSDSs are required to be on-site when using acid and/or solvent solutions for decontamination.

6.0 Applicable Standards and References

United States Environmental Protection Agency – Environmental Response Team Standard Operating Procedures "Sampling Equipment Decontamination" SOP #2006 Revision 0.0, August 11, 1994.

Field SOP 4
Monitoring Well Development

1.0 Purpose

The purpose of well development is to produce groundwater that is free of sediment, drill cuttings and drilling fluids. Often, the driller that performed the installation of the monitoring well will also perform the well development.

2.0 Equipment and Materials

- Site map.
- Keys and/or other tools required to gain access into wells.
- Well construction details (boring log that describes diameter, depth, screened interval, volume of water lost into formation during drilling of well).
- Bailer/surge block or pump.
- Generator or battery (if needed).
- Polyethylene tubing (if needed).
- Air monitoring device, such as PID and/or methane monitor (if needed) with appropriate appurtenances and calibration gases required for use.
- Check with the HASP before conducting field work to determine the equipment needed for air monitoring.
- Electronic water level indicator.
- Logbook and data sheet.
- PPE: sampling gloves, protective eyewear, steel-toe boots, or as specified by the site-specific HASP.
- Decontamination supplies.

3.0 Procedures

Wells sealed with grout or slurry shall be developed after a minimum waiting period of 12 hours after installation is completed.

3.1 Decontaminate or pre-clean equipment and ensure it is in working order.

3.2 Open the well cap. Monitor headspace of well with field monitoring equipment and record results in log book, if required by HASP. Take appropriate action based on readings and HASP guidance.

3.3 Measure and record the static groundwater and total depth of the well, both to the nearest 0.1 foot. Compare the measured total depth to the total depth recorded on the boring log to determine the thickness of sediment settled at the bottom of the well (to be removed).

3.3.1 All permanent monitoring wells that cannot be purged dry shall be developed by the following procedures:

- Alternatively surge and purge (for a minimum of 30 minutes). The surge and purge cycle shall consist of several minutes of surging followed by several minutes of purging to remove the material collecting at the bottom of the well.

- The surging shall move formation water in and out of the well screen. The surging shall be accomplished by using either a bailer or surge block or by pumping the well sufficiently to cause a drawdown and then allowing the well to recover and then repeating the process.

Note: When a surge block is used, care should be taken to avoid drawing the annular space material into the filter pack or well screen.

- After the final purge and surge cycle is completed, the well shall be pumped or bailed until 10 well volumes of water are removed or until the well produces sediment-free water.

- If sediment-free water is not obtained, any remaining sediment shall be removed from the bottom the well.

- Well volumes shall be calculated using the total volume of water present within the well casing and filter pack.

3.3.2 All permanent monitoring wells that can be purged dry shall be developed in a manner that limits agitation during the purging process. Wells which

can be purged dry may not be surged and no water may be added to the well. The well shall be purged slowly to minimize sediment disturbance.

3.4 Complete field log and close and lock the well.

3.5 Decontaminate equipment upon completion of well development.

4.0 Records and Documentation

Information and readings will be recorded on an applicable log sheet for the work being performed in accordance with the work plan.

- For each well, record the well identification number, volume of water used during well construction, air monitoring results (if any); depth-to-water measurements, total well depth measurements, calculation of one well-volume, surge and purge method and time period, and details of subsequent pumping, if applicable.

5.0 Special Notes

Several other well-development methods are also available (i.e., air surging/air lift, over-pumping, etc.). Prior to using these other techniques, obtain approval of GZA Project Manager.

Field SOP 5
Water Level Measurement

1.0 Purpose

The purpose of this procedure is to set guidelines for the determination of depth to water in an open borehole, cased borehole, monitoring well, or piezometer where separate phase product is not encountered. Generally, water level measurements are used to prepare water table maps and determine groundwater flow directions, as well as other aquifer characteristics.

2.0 Equipment and Materials

- Keys and/or other tools required to gain access into wells.
- Electronic depth-to-water measurement device.
- Air monitoring device, such as PID and/or methane monitor with appropriate appurtenances and calibration gases required for use.
    - Check with the project HASP before conducting field work to determine the equipment needed for air monitoring.
- Decontamination supplies.

3.0 Procedures

- Decontaminate or pre-clean equipment and ensure it is in working order.
- If possible, and when applicable, the sequence of field measurements should proceed from the least-contaminated wells to the most-contaminated wells.
- Open the well cap. If required by site-specific conditions, monitor headspace of well with field monitoring equipment and record results in log book. Take appropriate action based on readings and HASP guidance. After removing a water/air-tight well cap (e.g., flush-mounted well), allow the water level in the well to stabilize. This may be especially important for wells screened in silt and clay (low permeability) formations.
- When opening the well cap, be aware that the well can be under pressure. Therefore, have a firm grip on the well cap during removal.
- Water levels will be measured in all wells prior to performing groundwater sampling at the site.

- Lower water measuring device into well until the audible signal indicates the water surface has been encountered. Depth to water level measurements will be taken from a marked reference point at the well-head. The measuring point at the top of the casing will have been surveyed and permanently marked, if the top of well casing is not horizontal, to ensure that measurements will be consistently taken from the same reference point.

- For electrical sounders lower the device into the well until the water surface is reached, as indicated by a tone or meter deflection. Record the distance from the water surface to the reference point.

- Depth-to-water readings will be verified by repeating the procedure until successive measurements differ by no more than 0.01 foot.

- Following completion of depth-to-water measurement, deploy the water level device to the bottom of the well and record the total well depth, if required. This measurement does not need to be to a high degree of accuracy and is typically conducted at a lower frequency than measuring water levels. It is intended to establish the approximate height of the water column and also to document over time whether there is a change in the condition of the well.

- During removal of the water level measurement device, observe for any indication of free-phase product (odors, visible globules of product). If product is encountered unexpectedly, contact the GZA Project Manager promptly.

- Complete the field log, including depth-to-water measurements, total well depth, if measured, and observed condition of the well. Close and lock the well.

- Decontaminate the measuring device upon completing the groundwater and well depth measurements at each well.

4.0 Records and Documentation

Readings will be recorded in the project field log book or on the applicable log sheet for the work being performed and in accordance with the work plan.

- For the field work event, record the personnel present at the Site, date, weather and ambient temperature, air monitoring equipment details (such as PID model and lamp energy rating) if monitoring is required by the HASP, water level measuring device model and decontamination procedure/solutions.

- For each well, record the well identification, time of measurement, air monitoring results (if applicable), depth-to-water measurements, well-head reference point used, total well depth measurement, if measured, and physical condition of the well.

5.0 Special Notes

- All water level measurements at a given site should be preferably collected within a 24-hour period. However, certain situations may produce rapidly changing groundwater levels that necessitate taking measurements in a shorter time period, if possible. Rapid groundwater level changes may occur due to atmospheric pressure changes; changes in river stage, impoundment level, or flow in unlined ditches; pumping of nearby wells; snow melt; and/or precipitation.

- Before measurements are made, water levels in piezometers and monitoring wells should be allowed to stabilize for a minimum of 24 hours after well construction and development. In low yield situations, recovery of water levels to equilibrium may take longer.

- Depth-to-water measurements should be recorded to the nearest 0.01 feet.

6.0 Applicable Standards and References

USEPA Environmental Response Team Standard Operating Procedures 2043 Manual Water Level Measurements, Revision 0.0, February 11, 2000.

Field SOP 6
Groundwater Grab Samples
Temporary Monitoring Wells

1.0 Purpose

The methods and equipment used for groundwater grab sampling depend on the groundwater depth, diameter of access to groundwater and laboratory analyses to be conducted. The sampling equipment must be constructed of material that is compatible with the analyte of interest and the intended purpose for use of the data. For groundwater grab samples, the sampling and laboratory analyses are conducted for screening purposes and not necessarily for compliance to a standard. Decisions regarding follow-up installation of monitoring wells and standard monitoring well purging and groundwater sampling may occur as a result of the screening-level data obtained from groundwater grab samples.

When groundwater grab samples are collected shortly after temporary well installation (i.e., during the same day), purging is conducted to reduce turbidity and remove the minor volume of water in the area directly impacted by the installation procedure rather than to remove stagnant water from the well and filter pack.

2.0 Equipment

- Safety equipment, as specified in the HASP;
- Water-level indicator;
- Bailer with disposable rope;
- Peristaltic pump;
- Polyethylene and Tygon® tubing;
- Calibrated container;
- Pre-preserved, laboratory-supplied sample containers;
- Ziploc™ plastic bags;
- Labels;
- Chain-of-custody records;
- Coolers;

- Ice; and

- Decontamination supplies/equipment

3.0 Sampling Procedures

3.1 Purging When the Water Level is Within the Suction Depth (typically when the water level is within 25 feet of the pump):

- Cut sufficient tubing to reach the bottom of the temporary well screen.

- Measure the water level in the temporary well.

- Slowly lower the tubing to approximately the middle of the water column and begin purging at rates less than approximately 1,000 milliliters per minute.

- Measure the water level in the temporary well while purging.

- If the water level has declined by more than a ⅓ of the initial water column, decrease the purge rate to stabilize the water level at no more than ⅓ of the initial water column.

- Sustained pumping at the low drawdown rates will usually result in a relatively clear, low turbidity sample.

- Continue purging until low turbidity water is being purged and at least 2 casing volumes of water have been removed from the temporary well.

- Transfer water from the peristaltic pump discharge to the laboratory-supplied containers for the prescribed analyses.

- If excessive drawdown continues after lowering the purge rate, purge the well to dryness and allow the water level to recover before collecting the laboratory analytical samples.

- Place the soil samples on ice and deliver samples to the laboratory for analysis under chain-of-custody pursuant to Field SOP 7.

- Follow proper decontamination procedures pursuant to Field SOP 3.

3.2 Purging When the Water Level is Greater than the Suction Depth:

- Measure the water level in the temporary well.

Field SOP 6
Groundwater Grab Samples
Open Borholes or Temporary Monitoring Wells

- Slowly lower a small-diameter bailer into the temporary well and slowly retrieve the bailer to minimize agitation of the water in the well.

- Continue purging with a bailer until at least 2 casing volumes of water have been removed from the temporary well and preferably until low turbidity water is being purged.

- Obtaining relatively sediment-free samples is not always possible when purging with a bailer.

- After purging is complete, transfer water from the bailer to the laboratory-supplied containers for the prescribed analyses.

- If excessive drawdown occurs during purging, purge the well to dryness and allow the water level to recover before collecting the laboratory analytical samples with a bailer.

- Place the soil samples on ice and deliver samples to the laboratory for analysis under chain-of-custody pursuant to Field SOP 7.

- Follow proper decontamination procedures pursuant to Field SOP 3.

Field SOP 7
Chain-of-Custody

Sample tracking, security and chain-of-custody procedures provide a legal record of sample transport, possession and handling.

1.0 Sample Identification

Use waterproof labels or a similar method to identify each sample container. Use a permanent waterproof marker. Avoid placing labels on container lids; however, if you do place a label on a lid, make sure it is attached to the container as well. Labels should include the following information:

- A unique sample number;
- Site/project name;
- Date and time sample collected;
- Sample collector's initials; and
- Preservation and analysis required.

2.0 Sample Seals

Some sampling protocols require sample seals. Affix the seal to the sample container so that it has to be broken to open the container. Write your initials, date and time on the seal.

3.0 Shipping Custody Seal

Use a laboratory-provided chain-of-custody seal on the shipping container/cooler used for shipping. Affix the seal to the shipping container/cooler so that it has to be broken to open the container. If you use tape, write your signature, date and time on the tape.

4.0 Chain-of-Custody Record

Complete the laboratory-supplied chain-of-custody record for each sampling event. Each time the samples change possession, whoever relinquishes and whoever receives the samples must write their signature, date and time on the chain-of-custody form.

Note: If you place the chain-of-custody record in a sealed shipper, you do not need to have the commercial courier (e.g., UPS) sign, date and time the record; however, the courier should have a record of when he or she picked up the samples and where they were sent.

Note: Trip Blanks should also be recorded and included on the Chain-of-Custody Record. Sample numbers on the chain of custody must match those on the sample bottles.

Field SOP 8
Handling of Investigation-Derived Waste
(Decontamination Water, Purge Water, Drill Cuttings, Disposable Supplies)

1.0 Purpose

The following guidelines have been developed for the disposal of wastes generated from field activities at properties potentially impacted by hazardous substances and/or petroleum. This procedure is intended to address the following waste streams:

- Soil generated during drilling/well installation;
- Water generated during installation and development of monitoring wells; and
- Field supply residuals (used gloves, decontamination supplies, etc.).

The goal of residual disposal is to minimize potential contamination due to exposure of residuals to the environment and also to ensure that defined roles and responsibilities for waste management are understood. This SOP is not intended to cover all situations that may arise, but provides the basic guidelines for field decisions.

2.0 Equipment and Materials

If containers are needed for containment or storage of any wastes, they must be acquired from a reputable provider of clean (unused and/or certified recycled), DOT-approved, 55-gallon capacity, open top, steel drums with removable steel top and bolt ring. The drums shall have a removable, 2-inch diameter bung port on the sealable top and be of a minimum 18-gauge (1.1 mm nominal thickness) heavy-duty steel construction.

3.0 Procedures - Waste Collection and Storage

The primary sources of liquid (typically groundwater) and solid (soil) residuals will be generated during drilling, well development and soil and/or groundwater sampling programs. All waste is to be moved to a centralized location daily upon completion of activity at each location during a work day.

3.1 Water

1. Residual wastewater will be generated from the following field activities:
    - Drilling activities;
    - Decontamination process; and/or
    - Well purge water (development).

2. Wash waters from the decontamination process will be captured and containerized in the 55-gallon drum(s). In most cases, the water will be the detergent wash and rinsate liquids.

3. Water produced from drilling, development and purging of monitoring wells will be containerized in 55-gallon drums or in 5-gallon pails and transferred to drums.

4. Containerized wastewater residuals will be appropriately labeled with the following information:

   - Generator name, address and phone number;
   - Contents: general description and source location (e.g., "purge water from MW-1 and MW-2");
   - Date generated;
   - If sampled by GZA, date of sampling and project identification (Project File Number); and
   - Marked as hazardous or non-hazardous waste.

3.2 Solid Waste

1. The residual solid materials will primarily consist of soil drill cuttings, but also waste materials generated during sampling events such as Geoprobe® liners, tubing, PPE, etc. Cuttings from hollow-stem auger drilling activities may be temporarily placed on plastic sheeting at the drill site and covered or containerized in 55-gallon drums; the preferred option is directly into drums.

2. All containerized soil waste residuals on the site will be appropriately labeled with the following information:

   - Generator name, address and phone number;
   - Contents: general description and source location (e.g., "drilling soils from borings SB-1 and SB-2");
   - Date generated;

- If sampled by GZA, date of sampling and project identification (Project File Number); and

- Marked as hazardous or non-hazardous waste

4.0 Procedures - Waste Characterization, Transport and Disposal

4.1 It will be necessary to perform waste characterization to make proper transport and disposal arrangements. The purpose of waste characterization is to determine whether waste is hazardous or non-hazardous and whether other specific criteria of individual disposal facilities can be met. Solid and liquid waste can often be sufficiently characterized with data derived from the investigation. If these data are insufficient, a sample from each waste stream is required for a complete hazardous waste assessment. Decisions about whether any testing is needed and the parameters for laboratory analyses must be made on a case-by-case basis in consultation with the Project Manager for the particular site.

4.2 Typically, all solids and liquids will require a waste profile so that the investigation-derived waste can be properly manifested and disposed of at a licensed solid-waste landfill and/or waste treatment facility.

4.3 GZA will transmit waste profile analytical data and ready-to-sign waste profiles to the Project Manager for signature. GZA will work with the project manager to obtain the necessary approvals for disposal, schedule/coordinate shipping of the waste and all associated payments and required recordkeeping documents.

5.0 Records and Documentation

At the end of any field effort that generates waste that will be left on a site, the GZA field notes should include an inventory of the materials including: type of waste, volume, detailed origin of waste, storage container(s), number of containers, where stored, how secured and whether any samples were taken that will be analyzed to characterize this waste.

GZA will retain copies of sampling notes and laboratory data.

APPENDIX F

Field Data Sheets

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

CHAIN OF CUSTODY

Lab Work Order #:

Mail Report To:
Company:
Address:
E-mail Address:
Invoice To:
Company:
Address:

Project Number:
Project Name:
Project Location:
Turn Around (check one): ☐ Normal ☐ 5 BDs ☐ 3 BDs ☐ 2 BDs ☐ 24 hrs
If Rush, Report Due Date:
Sampled By (Print):

Preservation Codes
Analyses Requested

Sample Description	Collection Date	Collection Time	Matrix	Total # of Containers							Comments	Lab ID	Lab Receipt Time
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			
					☐	☐	☐	☐	☐	☐			

Preservation Codes
A=None B=HCL C=H_2SO_4
D=HNO_3 E=EnCore F=Methanol
G=NaOH O=Other (Indicate)

Matrix Codes
A=Air S=Soil W=Water O=Other

Rush TAT Multipliers
5 Business Days = 1.5x
3 Business Days = 2x
2 Business Days = 2.25x
24 Hours = 2.5x
must be pre-arranged

Relinquished By:	Date:	Time:	Received By:	Date:	Time:
Relinquished By:	Date:	Time:	Received By:	Date:	Time:

Custody Seal: ☐ Present ☐ Absent ☐ Intact ☐ Not Intact Seal #s:
Shipped Via:
Receipt Temp:
Temp Blank: ☐ Y ☐ N

Download this form at www.eccsmobilelab.com

WHITE - REPORT COPY YELLOW - LABORATORY COPY PINK - SAMPLER/SUBMITTER Rev. 5/11

QEC
Quality Environmental Containers

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

QEC
Quality Environmental Containers

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

QEC
Quality Environmental Containers

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

QEC
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P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

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Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

QEC
Quality Environmental Containers

P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	_____ GRAB _____ COMPOSITE

ANALYSIS REQUESTED

CUSTODY SEAL
DATE
SIGNATURE

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

GZA
GeoEnvironmental, Inc.
Engineers and Scientists

Lockheed Martin Corp., Inc.
Wedron Silica Co.
Wedron, Illinois

Boring No.: MW-1
Page: 1 **of** 1
File No.: 20.0151178.51
Check:

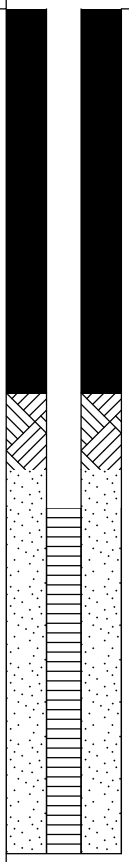
Contractor:
Foreman:
Representative:
Date Start/Finish:
Boring Location:
GS Elev.: **Datum:**

	Auger/ Casing	Sampler
Type:		
O.D. / I.D.:		
Hammer Wt.:		
Hammer Fall:		
Other:		

GROUNDWATER READINGS

Date	Time	Depth	Casing	Stab

Depth, (ft)	Sample Information: No.	Pen./ Rec., (in)	Depth, (ft)	Blows, (/6")	Field Test Data	Sample Description & Classification	Stratum Desc.	Remarks	Equipment Installed
1–24						END OF BORING AT 22'			



REMARKS

Stratification lines represent approximate boundary between soil types, transitions may be gradual. Water level readings have been made at times and under conditions stated. Fluctuations of groundwater may occur due to other factors than those present at the time measurements were made.

Boring No.: MW-1

WELL_BOR_NEW 151178.51 BORING LOG FOR QAPP - WELL_BOR_NEW.GPJ GZADEPTH.GDT 10/2/13

Field Equipment Calibration Documentation

Date	GZA Employee (Name)	Equipment Type and Model	Calibration Performed	Notes

APPENDIX G

Data Validation Procedures and Level 3 and 4 Data Validation Elements

EPA Level 4 Data Validation

EPA Level 4 data validation will be performed on the summary and raw data packages for analyses of sediment, sea water, and tissue samples analyzed by EPA and non-EPA methods. The data reviewer will request any missing information from the laboratory and facsimile a copy of this request to the client's project chemist when missing information is requested. The data reviewer will validate all components of the data package even when an individual QC element has rejected the data. All data will continue through the validation process and be qualified and requalified as many times as they fail to meet established criteria. An overall final qualification of results will encompass the impact of individual findings and will be determined using the professional judgement of a senior data reviewer. For organic analyses, 100% of all detects must be recalculated. For inorganic analyses, 10% of all detects must be recalculated.

Data summary packages provided by the contract laboratory should consist of sample results and quality assurance and quality control (QA/QC) summaries (equivalent to CLP Forms 1 through X for organic analyses and Forms 1 through XIV for inorganic analyses), and all raw data associated with the sample results and QA/QC summaries.

All data validation procedures will be in accordance with EPA Functional Guideline requirements and industry standards.

Data Validation Elements

The QC elements to be reviewed for Level 4 validation are identified in the following subsections.

Organic Analyses

- Holding times
- Initial calibration
- Continuing calibration
- Blanks
- Surrogate recovery
- Matrix spike and matrix spike duplicate recovery
- Laboratory control sample recovery
- Internal standard performance
- Field duplicate sample analysis RPD
- Compound identification
- Compound quantitation and detection limits
- Tentatively identified compound verification (GC/MS)
- System performance
- Overall assessment of data in the SDG

Inorganic Analyses

- Holding times
- Initial calibration
- Continuing calibration
- Blanks
- Surrogate recovery
- Matrix spike recovery
- Duplicate sample RPD
- Laboratory control sample recovery
- ICP interference check
- MSA and serial dilution checks
- Field duplicate sample analysis RPD
- Analyte identification
- Analyte quantitation and detection limits
- System performance
- Overall assessment of data in the SDG

EPA Level 3 Data Validation

EPA Level 3 data validation will be performed on the summary (i.e., no raw data) packages for analyses of sediment, sea water, and tissue samples analyzed by EPA and non-EPA methods. The data reviewer will request any missing information from the laboratory and facsimile a copy of this request to the client's project manager when missing information is requested. The data reviewer will validate all components of the data package even when an individual QC element has rejected the data. All data will continue through the validation process and be qualified and requalified as many times as they fail to meet established criteria. An overall final qualification of results will encompass the impact of individual findings and will be determined using the professional judgement of a senior data reviewer.

Data summary packages provided by the contract laboratory should consist of sample results and quality assurance and quality control (QA/QC) summaries (equivalent to CLP Forms 1 through X for organic analyses and Forms 1 through XIV for inorganic analyses), and QA/QC summaries.

All data validation procedures will be in accordance with EPA Functional Guideline requirements and industry standards.

Data Validation Elements

The QC elements to be reviewed for Level 3 validation are identified in the following subsections.

Organic Analyses

- Holding times
- Initial calibration
- Continuing calibration
- Blanks
- Surrogate recovery
- Matrix spike and matrix spike duplicate recovery
- Laboratory control sample recovery
- Internal standard performance
- Field duplicate sample analysis RPD
- Reporting limits
- Overall assessment of data in the SDG

Inorganic Analyses

- Holding times
- Initial calibration
- Continuing calibration
- Blanks
- Surrogate recovery
- Matrix spike recovery
- Duplicate sample RPD
- Laboratory control sample recovery
- ICP interference check
- MSA and serial dilution checks
- Field duplicate sample analysis RPD
- Reporting limits
- Overall assessment of data in the SDG

LDC Submittal for:

Wedron Community Groundwater, Wedron, IL Project

SUBMITTED TO:

GZA GeoEnvironmental, Inc.
20900 Swenson Drive, Suite 150
Waukesha, WI 53186
Attn: Mr. Bernard G. Fenelon

PRESENTED BY:

Laboratory Data Consultants, Inc.
7750 El Camino Real, Suite 2L
Carlsbad, CA 92009

September 23, 2013

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LDC Company Profile

Laboratory Data Consultants, Inc. (LDC) is a quality assurance and environmental chemistry company focused in data validation, data quality assessment, database implementation, and data usability. Our primary services include data validation, electronic transfer of data, oversight of Quality Assurance/Quality Control (QA/QC) programs, laboratory audits, technical support for litigation, and database management. Our corporate office in Carlsbad, California is directed by Mr. Richard Amano, principal chemist, who has over twenty-five years experience in the environmental laboratory and data validation industry.

Laboratory Data Consultants, Inc. (LDC) is recognized as one of the leaders in the data validation industry through participation on major DOD projects such as:

- Navy CLEAN II Pacific Division (Earth Tech)
- Navy CLEAN II Southwest Division (Bechtel National)
- Navy CLEAN Atlantic Division (EA Engineering)
- Navy CLEAN II EFA West (Tetra Tech EMI)
- Navy RAC Southwest Division (OHM Remediation/IT Group/Shaw)
- AFCEE, Mather AFB (Montgomery Watson)
- AFCEE/Army Corps of Engineers, Travis AFB (CH2M Hill)
- AFCEE, McClellan and Castle AFB (Jacobs Engineering Group)
- AFCEE, Beale AFB (Law/Crandall, Inc.)
- AFCEE, Andrews AFB (URS)
- Army Corps of Engineers, Various projects, 8a Contract (Sacramento District)
- Army Corps of Engineers, Various projects (New Mexico)
- Army Corps of Engineers, DuPont Chambers (Philadelphia District)
- Army Corps of Engineers, Camp Navajo (Tetra Tech)

LDC has successfully performed over 500 data validation projects worth over 10 million dollars for prime contractors servicing Air Force (AFCEE), Army Corps, Navy, and industrial activities.

With our data validation experience on high profile projects over the past 20 years, LDC has developed well-documented procedures which support all facets of the data validation process. This includes critical steps such as:

- Project tracking.
- Peer review for all data validation activities.
- Internal training programs.
- Internal and external audits.
- Strict documentation.
- Electronic deliverables.

LDC as the "Best Value" Subcontractor

Laboratory Data Consultants, Inc. (LDC) will provide the client with a data validation subcontractor with an impeccable track record for timeliness, quality, technical expertise, and the ability to mitigate complex data quality issues. Our clients will confirm our current and past performance under DOD (including Navy), DOE, and commercial programs. We have experience and a proven track record demonstrated by the following:

- Data validation experience as a subcontractor for more than 200 Navy (NAVFAC) sites, over 120 AFCEE sites, more than 80 Army Corps sites, multiple EPA and Superfund sites, and under 6 Navy CLEAN and RAC contracts. This experience includes thorough understanding of the DOD and AFCEE validation guidelines. Work totaled over 10 million dollars in the past five years.

- Thorough secondary QA review program and **the staffing capacity (33 full time chemistry staff) to handle a project of this magnitude and importance.** This significantly reduces additional work the consultant will need to do when receiving LDC reports.

- Navy data validation experience on projects at Kaneohe NAS/MCB, Hunter's Point Naval Shipyard, Long Beach Naval shipyard, Mare Island, MCB Camp Pendleton, Miramar NAS and over 100 other bases in the past 20 years.

- On-time delivery record of greater than 99% with the ability to expedite turnaround as needed with our large experienced staff as noted above.

- Successful completion of two DOE and five DOD audits to approve LDC's internal data validation procedures, QA program and documentation systems.

- LDC is a small business and has been self certified under the SBA.

Data Validation Capacity

Laboratory Data Consultants, Inc. (LDC) is continually evaluating and monitoring its capacity to meet client needs. It is company policy to not accept a project unless the service can be completed on-time with our expected quality of performance. This policy has proved very successful in meeting past project deadlines. Our client references can confirm our performance of on-time delivery.

Because of the versatile capabilities of our staff, personnel can also support multiple areas which are high in backlog. Our training program documents personnel approvals for all data review activities. All of our staff actively participate in the LDC training program.

Laboratory Data Consultants, Inc. (LDC) has met its contractual turnaround time requirement on over 99% of the projects completed. LDC has successfully completed projects which required data review capacity of as many as 2000 samples in one month.

Project Team

Laboratory Data Consultants, Inc. (LDC) personnel have hands-on experience in the areas of data validation, laboratory QA/QC, CLP SOWs, and environmental laboratory analyses. As documented in the resumes of our staff, the project team has significant experience with DOD protocols, EPA NFG, SW-846, and all appropriate procedures and protocols associated with this RFP.

Laboratory Data Consultants, Inc. is presenting the following staff to perform key roles for this contract. The key staff of the project team are as follows:

- **Richard M. Amano, Principal Chemist**
  Project Role: Program Manager/Senior Technical Reviewer
  Data Validation Experience: 20 years
  Overall Laboratory and Data Validation Experience: 30 years
  B.S. Biochemistry, UCLA, 1979

  Mr. Amano will perform the role of program manager and primary point of contact for this subcontract. He will actively participate in the final technical review of all data validation reports. Mr. Amano will closely work with Client personnel to assure that all elements of the SOW have been met.

  Mr. Amano has over 29 years of environmental laboratory, QA/QC, and data validation experience. He has managed data validation projects using the DOD data validation guidelines for the past twenty years. Prior to founding LDC in 1991, he directed to two major laboratories, Analytical Technologies, Inc. and Brown and Caldwell, from 1983 to 1991. His data validation experience includes oversight and direction of major efforts for Superfund sites, DOE sites, Navy RI/FS projects, Army Corps of Engineers investigations, and AFCEE projects. He also has overseen several laboratory audits for major analytical testing programs for the Navy, Texaco, and Hewlett-Packard. His laboratory experience includes hands-on CLP and SW-846 GC/MS analysis, direction of GC/MS (including TO-14 air analyses) and radiochemistry groups, dioxins method development, and complex GC data interpretation of Aroclors. He has performed expert witness support for litigation purposes.

- **Erlinda T. Rauto, Senior Chemist**
  Project Role: Project Manager
  Data Validation Experience: 17 years
  Overall Laboratory and Data Validation Experience: 25 years
  B.S Chemical Engineering, Feati University, Manila, 1967

  Ms. Rauto will perform the role of day to day Project Manager for this project. She will monitor schedules, compliance of the validation to the appropriate standards, perform routine surveillance activities such as generation of nonconformance reports, validator training, and QA reports to management.

  Ms. Rauto has over 25 years of environmental laboratory and data validation experience. She has worked under the DOD and EPA data validation guidelines for the past 10 years. Her experience includes performance of data validation in the GC, trace metals, and wet chemistry areas for major Federal projects. Her laboratory experience includes hands-on CLP and SW-846 ICP/GFAA analysis, pesticide/PCBs and wet chemistry analysis.

  Stella Cuenco, Senior Chemist
  Project Role: Senior Organic Data Validator
  Data Validation Experience: 16 years
  Overall Laboratory and Data Validation Experience: 22 years
  B.S. Chemistry, University of the Philippines, 1991

  Ms. Cuenco will perform the role of senior organic data validator for this project. She will perform data validation for GC/MS and gas chromatography analyses and serve as a peer reviewer in the initial validation review process.

  Ms. Cuenco has over 22 years of environmental laboratory and data validation experience under DOD and EPA guidelines. Her experience includes performance of data validation in trace metals, methyl mercury, gas chromatography for volatile organics and extensive CERCLA, and EPA data review and data verification for all organic and inorganic analyses. Her laboratory experience includes hands-on CLP and SW-846 GC/MS methods.

- **Pei Geng, Senior Chemist**
  Project Role: Organic Data Validator
  Data Validation Experience: 11 years
  Overall Laboratory and Data Validation Experience: 18 years
  M.S. Chemistry, Sam Houston University, 1989

  Ms. Geng will perform the role of organic data validator for this project. She will perform data validation for GC/MS and gas chromatography analyses and serve as a peer reviewer in the initial validation review process.

Ms. Geng has 18 years of environmental laboratory and data validation experience. Her experience includes performance of data validation in the gas chromatography area for volatile and semivolatile organics and extensive AFCEE and Navy data review and data verification for all organic analyses. Her laboratory experience includes hands-on CLP and SW-846 GC/MS methods.

- **Ming Hwang, Senior Chemist**
  Project Role: Senior Inorganic Validator
  Data Validation Experience: 10 years
  Overall Laboratory and Data Validation Experience: 18 years
  PhD, Chemistry, Boston College, 1990

  Ms. Hwang will perform the role of senior inorganic data validator for this project. She will perform data validation for trace metals, serve as a peer reviewer for final validation reports, and provide technical expertise in the inorganic area to other validators.

  Ms. Hwang has over 18 years of environmental laboratory and data validation experience. She has worked under EPA data validation guidelines for the past eight years for the various DOD including Navy, Army Corps, AFCEE and commercial projects. Her experience includes performance of data validation in the trace metals and wet chemistry areas for major Federal projects. Her laboratory experience includes hands-on CLP and SW-846 ICP/GFAA analysis, direction of trace metals and inorganic chemistry groups, and overall technical review of CLP data deliverables. She is certified as a “Radiometric Data Validation Specialists” through course work and testing by the Radiochemistry Society.

- **Andrew Kong, EDD Manager**
  Project Role: EDD Manager
  B.S. Environmental Toxicology, University of California at Davis 2006

  Mr. Kong will perform the role of EDD manager for this project. He will perform EDD population and verification and coordinate all back-up of electronic information.

  Mr. Kong has 6 years experience in various EDD format population and verification. He has experience in formatting of EDD validation deliverables including ERPIMS.

- **Tony Rommelfanger, Data Control Manager**
  Project Role: Data Custodian
  Data Custodian Experience: 17 years
  Overall Laboratory and Data Custodian Experience: 24 years

  Mr. Rommelfanger will perform the role of data custodian for this project. He will perform the log-in of all data packages into the LDC tracking system. This system will generate spreadsheets for identifying all samples, their collection date, analysis performed, matrix, and report due date. Upon the completion of each delivery order, he will archive and catalog all reports and data in a secured storage area.

  Mr. Rommelfanger has 13 years experience in laboratory and data management experience. He has experience in organizing, logging in, and tracking data packages for technical staff.

Technical / Management Approach

Laboratory Data Consultants, Inc. (LDC) has established data validation and data management procedures which enable the thorough, consistent, timely, and efficient review of analytical data. Mr. Richard Amano, principal chemist/program manager, is responsible for all data validation related activities and has final authority for the company. Ms. Linda Rauto, senior chemist, will coordinate the day to day data validation activities and interface with the Client project chemist or PM as necessary. For day to day data review activities, data validators report directly to the senior chemist in the section of the review. Senior chemists report to the technical project manager. Data validation will be performed following the Client project specific-requirements as stated in the Scope of Work (SOW).

As the client alerts LDC of in-coming SDGs, the LDC project manager will log the order into the up-coming project list identified as the "LDC Project Backlog" form. This is now considered a booked order and is reserved a place in the schedule. The project manager will allocate and schedule staff resources for the project. She will generate a project specific summary which will detail the expected receipt date of the order, the due date, special method or QC requirements, and the data quality objectives (DQOs) of the project.

Once the data packages arrive, the packages are received and logged in according to LDC SOP 6.0.0 "Standard Operating Procedure for Sample Data Log-in". Computer generated worksheets for each SDG along with the "LDC SDG Table" and "LDC Sample Validation" spreadsheet are distributed to all project staff.

The "LDC SDG Table" spreadsheet is specific to each order and provides the project manager with an overview of individual SDGs and their associated analyses. Progress of projects is indicated daily on the spreadsheet. The "LDC Sample Validation" spreadsheet identifies individual analyses requested for all samples and supports the data reviewer in verifying that appropriate samples have been reviewed in each data package. The previously discussed tools and processes have been used to successfully meet deadlines and estimate project completion dates. Meetings are held routinely to assess the status of each project.

Throughout the data review process, the project manager monitors project status as described in a previous paragraph. If any scheduling or technical complications arise such that the quality of the review will be impacted, data review staff notify the project manager immediately for resolution. The project manager will keep the Client Project Manager current on the progress of all validation activities on a routine basis via e-mail and telephone. Once the data review worksheets have been completed and approved by a secondary review, the final technical reports are written. All final technical reports are reviewed by at least two senior staff. This may include the Technical Project Manager, QA Director, or Lead Chemist. Upon shipping the final report, the Client project manager will be contacted by phone. Invoices shall be submitted in the Client approved format.

Data Review/Validation Process

The data review and validation level of effort required for the Scope of Work outlined for this project will encompass several activities. The steps can be categorized in the following manner:

1) Sample Log-in

All samples submitted for data validation are entered into the LDC Log-in system. The system generates various spreadsheets for sample tracking, listings of laboratory and client identifications, sampling dates, analysis requested, matrix, and project due date. These tracking documents are distributed to all data validation, QA and project management staff.

2) Pre-screening of Data Packages

The pre-screening is performed concurrently with the sample log-in process. This task verifies sample chain-of-custody, data package completeness, and concurrence with the authorized delivery order.

3) Data Validation

The execution of the data review task requires the highest level of effort. The review process will be handled in a stepwise fashion including manual and automated data review. The validator will use manual review to document each finding on a Validation Findings form. Along with the finding, the reviewer will document the date of the occurrence, the lab reference identification, the validation criteria, the associated samples, and the qualification of the data. A Validation Checklist form is marked noting if validation criteria was met or exceeded. These checklists are used as an inventory sheet to assure all samples were reviewed for each criteria. The findings documented on the Validation Findings form will be transcribed into the final summary report. Examples of recalculation and findings worksheets used for Level 4 validation are available for review upon request.

All initial validation performed by Laboratory Data Consultants, Inc. has a secondary peer review. All final reports will be reviewed by a Senior Chemist or Principal Chemist.

4) First Report Review

The first review of the data validation report verifies that all findings and data qualification has been accurately transferred from the data validation worksheets. All sample identifications, methods, formatting, and general text are reviewed.

5) Senior Report Review

The senior review of the data validation report verifies that all findings, data qualification, and professional judgments previously integrated into the reports reflect the overall quality of the data. Any additional comments required to enhance the usability of the report will be inserted at this time.

6) QA Report Review

A QA check of selected data validation reports within an individual delivery order will be reviewed by the QA department. A formal nonconformance report will be generated for any identified deficiencies. The deficiency will be addressed with the appropriate staff and corrected prior to submittal to senior management for final review and signature.

7) Senior Management Review

The program/technical manager will perform an overall review of the final reports. He will sign the report cover letter and submit the report to the sample custodian for shipment to the client.

8) Electronic Data Deliverables (EDD)

This process will be initiated at step 1 with the receipt of disks from the client or loading EDDs to LDC's secured Internet portal. After automated verification of the EDD format, content, and fields, the EDD will be populated with the manual review for importing of the final data qualifiers. The final approval of qualifiers will occur after step 5.

To perform the above tasks, LDC will not use subcontractors since they would reduce the quality and control of the work product.

LDC Corporate Resources

Laboratory Data Consultants, Inc. (LDC) personnel have experience and formal training in the areas of data validation, electronic data deliverables and laboratory QA/QC. As documented in the resumes. LDC personnel have performed data validation in all analytical disciplines. These include, but are not limited to, GC/MS volatiles, GC volatiles, GC/MS semivolatiles, GC pesticides, ICP metals, ICP/MS metals, GFAA metals, GC petroleum hydrocarbons, GC/MS dioxins, explosives, radiochemistry, and wet chemistry. This versatility allows our organization to adapt to workload changes and allows for an excellent secondary review system. The organization is structured to allow direct communication between project managers, data validators, and clerical staff occurs on a daily basis.

With LDC's 20 plus years as a national leader in the data validation industry and extensive experience for projects with multiple EPA regions, DoD and DOE facilities, LDC is confident it will provide data validation services which will successfully meet all project requirements.

The validation group is managed by Ms. Linda Rauto, senior chemist, who has over 25 years of experience, and the overall operations are directed by Mr. Richard Amano.

The validation group is divided into chemists by discipline, organics (GC/MS, GC and HPLC) and inorganics (wet chemistry, IC, ICP, ICP/MS). All chemists report to senior group leads. A separate group does data package log-in into the LDC tracking system. Another group performs the EDD population and verification. Andrew Kong, database manager, leads this group and he has over 5 years performing EDD work.

The majority of the data validation staff at LDC have been employed by the company over 10 years with some senior staff over 15 years. This level of stability and experience will give the project stability and consistency of our work product.

In addressing LDC's financial status, LDC has an excellent Dunn & Bradstreet report and has been profitable for the past 18 years. LDC has grown at approximately 10% in each of the past 5 years and continues to be a leader in our environmental sector of the data quality business. References are available from our vendors and clients to confirm our business success. Our annual revenue of approximately $6,000,000 per year in data validation work makes LDC on of the largest independent data validation firms in the nation.

LDC will commit the resources and materials to successfully complete this project with the required time period and with a high level of quality.

Relevant Project Experience

Laboratory Data Consultants, Inc. (LDC) has performed data validation and Quality Assurance services for contaminated sites overseen by AFCEE, Navy Southwest Division, DOE, EPA Superfund projects overseen by EPA Region III, IV, IX, X, Army Corps projects reviewed by the Baltimore, Louisville, Albuquerque, Seattle, Philadelphia, and Sacramento Districts, and Navy projects reviewed by NFESC.

As noted previously in this proposal, LDC has experience on a wealth of DOD projects and specifically LDC has direct project experience with EA engineering at Andersen AFB in Guam and LDC also has previous experience at Kirtland AFB for MWH Global.

LDC has validated over 1,000,000 samples for analyses such as volatile organics (CLP, EPA Method 8240/8260), semivolatile organics (CLP, EPA Method 8270), organochlorine pesticides/PCBs (CLP, EPA Method 8081/8082), chlorinated herbicides (EPA Method 8151), purgeable halocarbons and aromatics (EPA Method 8021), trace metals (CLP, EPA Method 6010/6020/7000), PAHs by EPA 8310 and 8270,TOC analyses, hexavalent chromium, total petroleum hydrocarbons (EPA Method 8015/CDOHS LUFT), radiochemical constituents including gross alpha/beta, alpha spec, gamma spec, tritium, and uranium, and general minerals.

Laboratory Data Consultants, Inc. (LDC) has met their contractual turnaround time and quality requirements on over 99% of the projects completed.

The following table lists representative projects in which LDC has provided services along with project-specific references. Please contact them at your leisure or contact LDC for more references if required.

Project References/Experience

Name and Address, Contact Person, Telephone	Work Description and Location	Requested Deliverables	Number of samples/ Matrix	Value ($)	Start/Stop
AECOM (Earth Tech) 700 Bishop Street Honolulu, HI 96813 Contact: Scott Lewis (808) 523-8874 Scott.Lewis@aecom.com	Data validation per EPA level "3/C" and "4/D" guidelines for volatile organic, semivolatile organic, pesticides/PCBs, herbicides, phenols, phosphorus pesticides, dioxin, radiochemical, and trace metal analyses in soil, water, and tissue matrices. (Navy PACDIV CLEAN, Honolulu, HI)	LDC worksheets and validation reports	>10, 000 samples Water/Soil/Air	$750,000	4/98-present
TetraTech EC 17885 Von Karman Ave, Suite 500 Irvine, CA 92614 Attn: Lisa Bienkowski (949) 809-5028 Lisa.Bienkowski@tetratech.com	Tetra Tech Hunter's Point CA LDC performed EPA Level III and IV equivalent data validation for a full suite of analyses on more than 5,000 soil and water samples. Analyses included tritium, isotopic thorium, uranium and plutonium, and gross alpha/beta. Expedited turnaround times were included (5 day TAT)	EPA Level III and IV data validation reports. Work conducted under US Navy RAC program, Southwest Div.	>5,000 Soil and Water	$490,000	2006-present
Shaw E&I 3347 Michelson Drive, Ste 200 Irvine, CA 92612 Contact: Mr. Dwayne Ishida Phone: (949) 660-7561 Dwayne.Ishida@shawgrp.com	Data validation per EPA level "3" and "4" and AFCEE guidelines for volatile organic, semivolatile organic, pesticides/PCBs, herbicides, phenols, phosphorus pesticides, dioxin, radiochemical, and trace metal analyses in soil, water, and tissue matrices. (Navy Southwest Division RAC, San Diego, CA and various AFCEE projects)	LDC worksheets and validation reports	>5000 samples Water/Soil/Air	$350,000	6/06-present

Note: All above projects were 100% self-performed by LDC