MULTI-SITE QUALITY ASSURANCE PROJECT PLAN, REVISION 0
USEPA BROWNFIELDS HAZARDOUS SUBSTANCES AND PETROLEUM ASSESSMENT GRANTS (OCTOBER 2010)
CITY OF CHICAGO DEPARTMENT OF ENVIRONMENT
BROWNFIELDS ASSESSMENT PROJECT

USEPA Cooperative Agreement No. BF-00E683-01
Terracon Project No. A2107017
~~**December 21, 2010**~~
Rev. 1 - Sept. 16, 2011

Prepared for:

City of Chicago
Chicago, Illinois

Prepared by:

Terracon
Chicago, Illinois

MULTI-SITE QUALITY ASSURANCE PROJECT PLAN

Revision 1

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Terracon Consultants, Inc.
Chicago, Illinois

Offices Nationwide Established in 1965
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Geotechnical ■ Environmental ■ Construction Materials ■ Facilities

QUALITY ASSURANCE PROJECT PLAN APPROVAL SHEET

U.S. EPA BROWNFIELDS ASSESSMENT GRANT-
HAZARDOUS SUBSTANCE AND/OR PETROLEUM
CHICAGO, ILLINOIS
AWARD DATE: August 2008
USEPA Cooperative Agreement No. BF-00E683-01
Terracon Project No. A2107017

On behalf of the City of Chicago, this Quality Assurance Project Plan (QAPP) was prepared by Terracon Consultants, Inc. (Terracon) for the Brownfields Assessment Project. The QAPP was developed following the guidance presented in the United States Environmental Protection Agency (U.S. EPA) document QA/R-5 *Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan*, dated June 2000.

Kelley A. Moore 8/12/11
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8/12/11
Jan Pels, U.S. EPA QA Reviewer

Dave Graham, City of Chicago, Grantee Project Manager

Linda Yang, PG, Terracon Senior Project Manager

Dave Koch, Terracon QA/QC Manager

Pinaki Banerjee, STAT Analysis, Laboratory Quality Assurance Manager

Kate Grams, PACE, Laboratory Quality Assurance Manager

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QUALITY ASSURANCE PROJECT PLAN DISTRIBUTION LIST

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

Jan Pels, U.S. EPA QAPP Reviewer
Kelley Moore, U.S. EPA Project Manager
Dave Graham, City of Chicago Project Manager
Linda Yang, P.G., Terracon Senior Project Manager
Dave Koch, Terracon QA Manager
Pinaki Banerjee, STAT Analysis, Laboratory Quality Assurance Manager
Kate Grams, PACE, Laboratory Quality Assurance Manager

ACRONYM LIST

ACM – Asbestos-Containing Material

AHERA – Asbestos Hazard Emergency Response Act

ASTs – Aboveground Storage Tanks

ASTM – American Society for Testing and Materials

Terracon – Terracon Consultants, Inc.

CFR – Code of Federal Regulations

CNS – Covenant Not to Sue

COC – Chain-of-Custody

DI – Deionized

DQOs – Data Quality Objectives

DRO – Diesel-Range Organic Compounds

GRO – Gasoline-Range Organic Compounds

HASP – Health and Safety Plan

HUD – U.S. Department of Housing and Urban Development

IEPA – Illinois Environmental Protection Agency

LCSs – Laboratory Control Samples

MDLs – Method Detection Limits

MS/MSD – Matrix Spike/Matrix Spike Duplicate

O&M – Operation and Maintenance

OSHA – Occupational Safety and Health Administration

PARCCS – Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity

PCBs – Polychlorinated Biphenyls

PE – Performance Evaluation

PID – Photoionization Detector

PPE – Personal Protective Equipment

QA – Quality Assurance

QAPP – Quality Assurance Project Plan

QA/QC – Quality Assurance/Quality Control

ACRONYM LIST cont.

QC – Quality Control

QLs – Quantitation Limits

RPD – Relative Percent Difference

RSD – Relative Standard Deviation

SAP – Sampling and Analysis Plan

SOPs – Standard Operating Procedures

SVOCs – Semi-volatile Organic Compounds

TPH – Total Petroleum Hydrocarbons

USEPA – United States Environmental Protection Agency

USTs – Underground Storage Tanks

IEPA – Illinois Environmental Protection Agency

VOCs – Volatile Organic Compounds

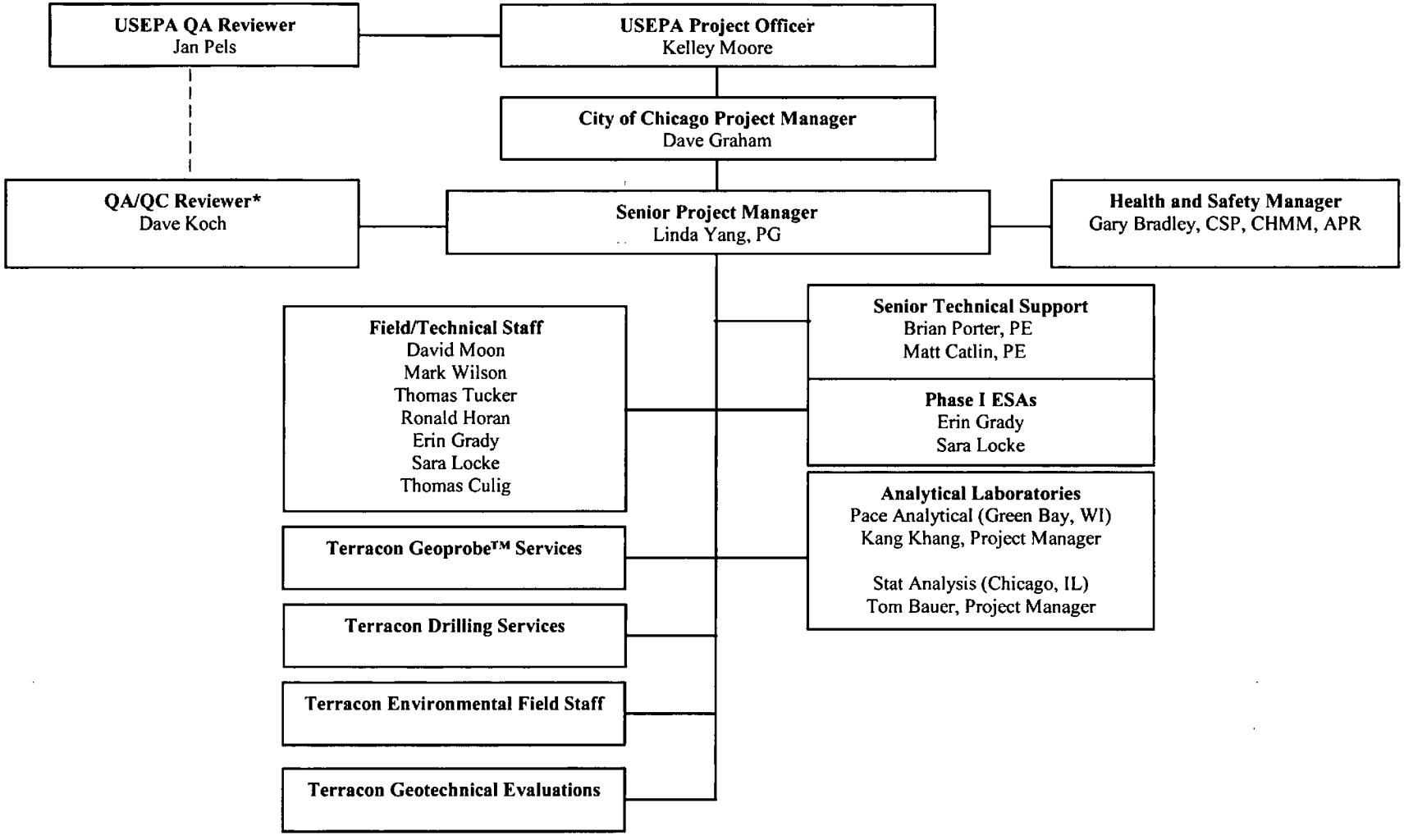
1.0 PROJECT MANAGEMENT

The purpose of this document is to describe the personnel, procedures, and methods for ensuring the quality, accuracy, and precision of data associated with the City of Chicago Brownfield Assessment Project. The City of Chicago Brownfield Assessment Project received a $400,000 U.S. Environmental Protection Agency (U.S. EPA) 'hazardous substance and/or petroleum' Grant. The purpose of this grant is to assess properties potentially impacted by hazardous substances and/or petroleum. Following the procedures outlined in this Quality Assurance Project Plan (QAPP) will ensure that the data collected meets the project objectives. This QAPP will be valid for up to 5 years, and it will be reviewed annually (from the date of approval) to insure that it is up to date. This annual review will be documented (letter format is acceptable) and sent to all recipients of the QAPP with any 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.), a call will be arranged with all parties that reviewed this QAPP to determine how to revise this document.

1.1 Project Organization and Responsibility

Figure 1 presents the organizational structure for the City of Chicago Brownfields Assessment Project. All lines of communication, management activities, and technical direction within this project team will follow this organization arrangement. Any directions or communications from the U.S. EPA will be given to the City of Chicago Project Manager. The Project Manager will subsequently communicate directions to the Terracon Consultants, Inc. (Terracon) project manager. The U.S. EPA project manager will be notified of all proposed changes in personnel.

Responsibilities of key project personnel are outlined below.



*Brownfields may require specialized knowledge to assist in data generation or reporting. Where this overlaps, the alternate Approved Project Reviewer not involved in data generation will review to maintain independence of the QA function.

Figure 1

U.S. EPA Project Officer

- Direct, review, and approve QAPP and Sampling and Analysis Plans (SAPs).
- Provide technical consultation services to the Project Manager and Terracon project manager.
- Review progress reports detailing work accomplished.
- Review all final reports.

Ms. Kelley Moore is the USEPA project manager.

U.S. EPA Quality Assurance Reviewer

- Review and approve the QAPP.
- Assist in review of the SAPs.

Ms. Jan Pels is the regional QA manager for the USEPA.

City of Chicago (Grantee) Project Manager

- Direct project activities.
- Prepare and submit progress reports detailing work accomplished, funds spent, and the project status.
- Responsible for review of project deliverables, development of project planning, and the overview of project strategies.
- Review site reports for consistency with objectives stated in work plans.
- Provide final signature on all assessments.

Mr. Dave Graham is the Project Manager for the City of Chicago (Grantee).

Terracon Senior Project Manager

- Responsible for planning, coordinating, monitoring, and evaluating of project field activities.
- Before sampling, meet with the Project Manager, quality assurance (QA) manager, and field staff to discuss and establish sampling purposes, sampling methodology, number of samples, size of samples, sample

preservation methods, chain-of-custody (COC) requirements, analyses required, and which samples will be duplicated in the field.
- Resolve technical problems.
- Conducts Field Audits.
- Meet with team members to discuss and review analytical results prior to completion of reports.
- Responsible for environmental reports and documents.

Ms. Linda Yang, PG acts as the senior project manager.

Senior Technical Support

- Can correspond with the USEPA, IEPA, City, or other parties, as needed, with or without the senior project manager.
- Assists with evaluation and coordination of environmental reports and documents.
- Can coordinate support from other Terracon offices on an as-needed basis and in response to a request from the senior project manager.
- Provides peer review of technical documents to the senior project manager.

Mr. Matt Catlin, PE and Mr. Brian Porter, PE, provide senior technical support.

Terracon Quality Assurance Manager

- Oversee assessment activities to ensure that sampling methodology, sample preservation methods, and COC procedures are being followed.
- Assist in any QA issues with field or laboratory questions, as needed.
- Maintain a record of samples submitted to the laboratory, the analyses being performed on each sample, the final analytical results, and data validation reports.
- Prepares Data Assessment Report (DAR).
- Annual review of QAPP.

Mr. Dave Koch acts as the QA reviewer.

Health and Safety Manager

- Responsible for Terracon's corporate safety program.

- Manages the Terracon surveillance, respiratory protection, and hazardous waste site training programs.
- Responsible for workplace safety at all Terracon facilities and for corporate compliance with federal and state OSHA, DOT, and DOE regulations.
- Oversees and coordinates workers compensation issues for Terracon and performs facility safety and regulatory compliance audits at Terracon facilities on a routine basis.

Mr. Gary K. Bradley, CSP, CHMM, is the Health and Safety Manager.

Terracon Field Technical Staff

- Before sampling, meet with Terracon project manager to discuss and establish sampling purposes, sampling methodology, number of samples, size of samples, sample preservation methods, COC requirements, analyses required, and which samples will be duplicated in the field.
- 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.
- Oversee drilling and soil boring work to ensure that proper procedures are followed during monitoring well installation and soil sample collection from borings.
- Monitor hazardous conditions while conducting field operations.
- Submit COC records and field paperwork to field team leader.

The field technical staff includes Messers. Mark Wilson, Thomas R. Tucker, Jr., Thomas Culig, and Ronald Horan as well as Mrs. Erin Grady and Ms. Sara Locke, although other supporting staff from Terracon may be assigned on an as-needed basis. Resumes for proposed Terracon personnel are included in Appendix B.

Phase I ESA

These staff are responsible for the following:

- Schedules and completes Phase I ESAs as requested by the senior project manager.
- Corresponds with Phase I ESA field team(s) and third-party Phase I ESA vendors.

- Provides Phase I ESAs to the senior project manager.
- Participates in outreach events as needed and upon request of the senior project manager.

Mrs. Erin Grady and Ms. Sara Locke will provide Phase I ESA services at the request of the senior project manager.

PACE Analytical (PACE) Project Manager

- Responsible for samples submitted to PACE, including those released to a subcontracted laboratory.
- Responsible for summarizing quality assurance/quality control (QA/QC) requirements for the project, including those samples analyzed by subcontracted laboratories.
- Maintain laboratory schedule and ensure that technical requirements are understood by laboratory personnel.
- Provide technical guidance to Terracon project manager.
- Ensure accuracy of the laboratory data.

Kang Khang will act as the Laboratory Project Manager.

PACE Analytical (PACE) QA Manager

- Responsible for evaluating adherence to policies and ensuring that systems are in place to provide QA/QC as defined in the QAPP.
- Initiate and oversee audits of corrective action procedures.
- Perform data reviews.
- Maintain documentation of training.

Kate Grams will act as the Laboratory QA Manager.

STAT Analysis (STAT) Project Manager

- Responsible for samples submitted to STAT, including those released to a subcontracted laboratory.

- Responsible for summarizing quality assurance/quality control (QA/QC) requirements for the project, including those samples analyzed by subcontracted laboratories.
- Maintain laboratory schedule and ensure that technical requirements are understood by laboratory personnel.
- Ensure accuracy of the laboratory data.

Tom Bauer will act as the Laboratory Project Manager.

STAT Analysis (STAT) QA Manager

- Responsible for evaluating adherence to policies and ensuring that systems are in place to provide QA/QC as defined in the QAPP.
- Initiate and oversee audits of corrective action procedures.
- Perform data reviews.
- Maintain documentation of training.

Pinaki Banerjee will act as the Laboratory QA Manager.

All Terracon site personnel will be trained as mandated by the Occupational Safety and Health Administration (OSHA) Act regulations (29 Code of Federal Regulations [CFR] 1910.120). Additionally, all site personnel will be properly trained in the procedures for collecting, labeling, packaging, and shipping of liquid and solid environmental samples. Persons conducting asbestos surveys will be certified by the Illinois Department of Public Health (IDPH). The Terracon project manager will maintain personnel training records. Field personnel will be trained to use all monitoring devices and other equipment used in the field.

The laboratories selected for the analytical work required for this project are Pace Analytical Services, Inc. (PACE) located in Green Bay, Wisconsin and STAT Analysis Corporation (STAT) located in Chicago, Illinois. Both laboratories have been certified by the IEPA under the National Environmental Laboratory Accreditation Program (NELAP), which is administered by the US EPA. STAT and Pace IEPA Accreditation numbers for their Chicago, Illinois and Green Bay, Wisconsin laboratories are 100445 and 200050, respectively. As IEPA-accredited laboratories, STAT and Pace have undergone performance evaluations administered by the State of Illinois for method accuracy and precision. These

evaluations meet the standards required by U.S. EPA. Nils Melberg is the Pace laboratory director and Don Cortes is the STAT laboratory director. The QA Manager for Pace will be Kate Grams and Pinaki Banarjee for STAT.

For analysis of asbestos-containing materials (ACM), STAT Analysis of Chicago, Illinois will do the analyses. They are NVLAP (National Voluntary Laboratory Accreditation Program) certified NVLAP # 101202-0.

The drilling subcontractor has not yet been selected for this project. However, all on-site drilling personnel shall have completed the applicable OSHA training. Additionally, drilling personnel will be required to comply with all site safety regulations covered in the site-specific HASP, provided under separate cover to this QAPP.

The geophysical survey subcontractor has not yet been selected for this project. However, all geophysical survey personnel shall have completed the applicable OSHA training. Additionally, geophysical survey personnel will be required to comply with all site safety regulations covered in the site-specific HASP, provided under separate cover to this QAPP.

The excavation/test pit subcontractor has not yet been selected for this project. However, all on-site excavation/test pit personnel shall have completed the applicable OSHA training. Additionally, excavation/test pit personnel will be required to comply with all site safety regulations covered in the site-specific HASP, provided under separate cover to this QAPP.

1.2 Facility History/Background Information

The City of Chicago's Brownfield Assessment Grant is a communitywide project, meaning that specific sites have not been identified for Phase II Property Assessments. Therefore, once the Brownfield sites have been identified for Phase II work, property-specific information will be provided with the SAPs.

1.3 Project Description and Schedule

The City of Chicago's Brownfield Assessment Grants are community-wide grants. The City has a list of potential Brownfield sites to be evaluated consisting of 'hazardous substance and/or petroleum' contamination, and will prioritize the sites

based on access to the properties, potential environmental issues, and redevelopment potential. The sites with the highest priorities will have Phase I and Phase II ESAs conducted as described in the cooperative agreement to understand the extent of environmental problems on a property. All Phase I assessments conducted will be compliant with U.S. EPA's All Appropriate Inquiry (AAI) rule of Nov. 1, 2005. Note that the ASTM E1527-05 Phase I Environmental Site Assessment Standard is consistent and compliant with the AAI rule, including the Environmental Professional Statement (E1527-05 Sections 12.8, 12.8.1, and 12.8.2.). Once the environmental assessments are completed, the City will pursue cleanup and redevelopment, which is not part of these grant projects.

The City of Chicago has retained Terracon as their consultant to perform Phase I and Phase II ESAs. The Phase I and Phase II ESAs are designed to provide City of Chicago and the U.S. EPA with data to facilitate potential redevelopment of each property investigated. These data will be used to determine whether there is a threat from potential contaminants, solutions for any remedial activities, and estimated costs for site redevelopment. The Phase I ESA is predominantly a fact-finding investigation. The Phase II ESAs may consist of one or more of the following tasks:

- Collection and analysis of soil samples
- Collection and analysis of sediment samples
- Collection and analysis of groundwater samples
- Collection and analysis of surface water samples
- Collection and analysis of ACM and LBP, potentially
- Installation of groundwater monitoring wells
- Aquifer testing and evaluation of aquifer characteristics
- Test pits or trenching
- Evaluation of geophysical survey results
- Evaluation of natural bioattenuation processes
- Evaluation of active remedial technologies

Details of the property-specific sampling activities will be addressed in the individual SAPs. The findings of each Phase II will be presented in a Phase II ESA report, which is discussed in later sections of this QAPP.

Terracon estimates that it will take approximately 6 to 9 weeks to perform a Phase II for each property. In general, it will take 1 to 2 weeks to prepare a SAP and HASP, 2 to 3 weeks to perform fieldwork including laboratory analyses, 2 weeks to gather any additional necessary data, and 1 to 2 weeks to prepare a Phase II report. The time is dependent on field conditions and laboratory data requirements. Once all of the assessments are complete, the Phase II ESA report will be prepared.

For the project schedule, please note that Phase I ESAs and Phase II ESAs may be conducted concurrently for different sites. The Phase I and Phase II work will not follow in succession. Based on an ongoing evaluation of City of Chicago's Brownfield program and the priorities established by the City of Chicago Brownfield Task Force, additional properties may have Phase I ESAs started while Phase II activities have already begun on other properties.

An IEPA-accredited laboratory will be used to ensure overall analytical quality. STAT Analysis or Pace Analytical will be the primary laboratories used for lab analyses. Copies of their IEPA certificates are included in Appendix G.

1.4 Data Quality Objectives (DQOs)

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.

1.4.1 Analytical Quality Objectives

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

1.4.1.1 Field Screening

Field-screening instruments provide a lower quality of analytical data compared to laboratory equipment in a controlled environment. However, field methods 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 detailed assessment, and preliminary comparison of remedial objectives. This type of field-screening data can include measurements of pH, temperature, conductivity, turbidity, or similar monitoring data. Field measurements of pH, temperature, conductivity, and turbidity will be collected during groundwater and surface water sampling activities. During sampling and other property assessment activities, the breathing space of site personnel will be monitored for the presence of organic vapors using a photoionization detector (PID). The PID will also be used to perform field screening of soil and sediment samples in order to assist in the selection of samples to be submitted for laboratory analysis. Generally, the soil interval with the highest PID readings at a boring or sampling location will be submitted to the laboratory. If no volatile organic vapors are detected by the PID, samples will be selected for laboratory analysis based on the following:

- Obvious discoloration, odor, or other visible signs of contamination.
- If no visible or odorous signs of contamination are evident, a sample from the zone directly above the water table will be submitted.
- A sample from a depth corresponding to the zone in the subsurface expected to contain the greatest concentration of contaminants will be submitted. This selection will be based on the type of release and the history of the area being investigated and will be determined by the Terracon project manager.

1.4.1.2 IEPA Analyses

The laboratories selected for the analytical work required for this project are Pace Analytical Services, Inc. (PACE) located in Green Bay, Wisconsin and STAT Analysis Corporation (STAT) located in Chicago, Illinois. Both laboratories have been accredited by the IEPA under the NELAP, which is administered by the US EPA. STAT and Pace IEPA Accreditation numbers for their Chicago, Illinois and Green Bay, Wisconsin laboratories are 100445 and 200050, respectively. As an IEPA-accredited laboratory, STAT and Pace have undergone performance evaluations administered by the State of Illinois for method accuracy and precision. These evaluations meet the standards required by U.S. EPA.

1.4.2 Project Quality Objectives

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

1.4.2.1 Problem Statement

City of Chicago intends to use the U.S. EPA Brownfields Assessment Grant funds to investigate properties listed for redevelopment, and possibly several others as identified by members of the community. Based on the prioritization, the balance of the funds will be used to conduct Phase I and Phase II ESAs. The intent of the Phase I ESAs will be to identify recognized environmental conditions (RECs) associated with the site. The property-specific work plans will detail the proposed methods for identifying contaminants, assessing the hazards posed by these contaminants, and managing or remediating contaminants for property redevelopment.

Exposure assessments and proposed redevelopment use of each of the properties will be discussed in the Phase II ESA reports.

1.4.2.2 Decision Identification

Available information will be used to determine if the subject properties have been contaminated. To assess the feasibility of property redevelopment, City of Chicago will ask the following questions:

- Do contaminant levels exceed applicable standards such as IEPA limits?
- Can the contaminants be managed by eliminating exposure pathways through engineering and institutional controls?
- Will the property require remediation prior to redevelopment?
- If remediation is too costly based on the expected land use, can the property be developed for another use?

1.4.2.3 Decision Inputs

Samples of soil, sediment, groundwater, and/or surface water will be collected for analysis as described in the SAPs in order to assess the level of contamination. Samples will be collected to either assess the data gaps identified from work previously completed or assess Recognized Environmental Conditions (RECs) noted during the Phase Is. An REC is the presence or likely presence of any hazardous substance and/or petroleum products on a property under conditions that indicate an existing release, a past release, or a material threat of a release of any hazardous substance and/or petroleum products into structures on the property or into the ground, groundwater, or surface water of the property or nearby properties. Such data gaps or environmental conditions may answer the following:

- What is the level of potential exposure to surface or subsurface soils at the property?
- What is the level of potential exposure to surface water and associated sediments at the property?
- What is the level of potential exposure to groundwater at the property?
- Have past uses of the property (or adjacent properties) impacted the soil, sediment, surface water, or groundwater?
- Did past 'hazardous substance and/or petroleum' handling or storage activities, if any, impact the property?
- If any former underground storage tanks (USTs) existed on the property, does contamination exist near the area of the identified tank?
- Have former aboveground storage tanks (ASTs) impacted the surrounding media at the property?
- Does fill material (such as slag) used at the property contain contaminants that may impact soil, sediment, surface water, or groundwater?
- Have uncontrolled dumping or landfilling activities occurred at the property, and if so, have they impacted the soil, sediment, surface water, or groundwater?

1.4.2.4 Assessment Boundary

A site map showing the assessment boundary will be provided in each SAP. Because target properties will be selected based on the results of Phase I ESAs and the nature of environmental impacts will be property-specific, detailed information

regarding the assessment boundaries cannot be determined currently. However, once the target properties are identified, information regarding the assessment boundaries will be included in the associated SAPs. The assessment boundary information in each SAP will include the property boundaries, potential exposure areas, and sample locations and depths. It should be noted that the assessment boundary will not necessarily be the property boundary.

The vertical assessment boundary will vary depending on the contaminant conditions identified and the end use of the subject property.

1.4.2.5 City of Chicago Decision Process

IEPA generic numerical standards may be the applicable State standards for cleanup criteria. Soils and sediment will be compared to the applicable Tiered Approach to Corrective Action Objectives (TACO) soil remediation objectives as detailed in Title 35 Illinois Administrative Code Chapter 742. The IEPA soil objectives for residential properties and commercial/industrial properties are presented in Table 8 (located in Appendix A). Groundwater results will be compared with the IEPA TACO remediation objectives for Class I Groundwater. These are also presented in Table 8. If sample results collected as part of the property assessment are all below the applicable IEPA objectives, then a redevelopment project can proceed without special consideration other than those normally employed in redeveloping properties of similar type and land use.

If sample results exceed the applicable IEPA objectives, City of Chicago will consider the following options:

- If contaminant levels exceed the IEPA Tier 1 criteria for residential land use, the City could enroll the property in the SRP to pursue formal regulatory closure. However, based on a site-specific evaluation of the redevelopment "value" of an NFR letter, the City may opt to forego enrollment of a property in the SRP depending on the following factors.
    - Is environmental impact limited to a specific media?
    - Are the soil concentrations below non-residential ROs and is the property planned for non-residential redevelopment?

- Can the City easily impose limitations on future land use sufficient to allow redevelopment?

- Other factors as yet unknown

If this occurs, the technical report documenting the investigation activities can be used at a later date to enroll the property in the SRP in pursuit of an NFR letter.

- If soil or groundwater contaminant levels exceeding IEPA standards are associated only with a specific exposure pathway, City of Chicago may then conduct a property-specific evaluation and pursue management of exposure pathways through the use of engineering and institutional controls. These controls may be implemented through an Operation and Maintenance (O&M) Agreement with IEPA.

- If an exposure pathway cannot be managed through engineering or institutional controls, then the City of Chicago may develop a Remedial Action Plan to meet the needs of the proposed future use of the property.

1.5 Quality Assurance Objectives for Measurement

The overall QA objective for each project is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting using IEPA protocol. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventative maintenance of field equipment, and corrective action are described in other sections of this QAPP.

Data quality objectives for measurements during this project will be addressed in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). The numerical PARCCS parameters will be determined from the project DQOs to ensure that they are met. 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.

1.5.1 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.

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.

1.5.1.1 Field Precision Objectives

Field precision will be assessed through the collection and analysis of duplicate samples. Water matrix samples can be readily duplicated due to their homogeneous nature; however, the duplication of solid (e.g., soil, fill, sediment) samples is much more difficult due to their non-homogeneous nature. Consequently, target water and soil (or other solids) RPDs will be within 35% and 50%, respectively, of the original result. Duplicate recoveries within these ranges will be considered precise without further discussion. Duplicate recoveries beyond these ranges may require further qualification of associated data, but data will not be rejected unless determined unusable through the data validation process. It should be noted that the RPD of two measurements can be very high when the data approach the PQL. For this reason, the RPD will only be calculated for analytes detected in both investigative and field duplicate samples at concentrations greater than or equal to five times the PQL.

The RPD will be calculated using equation (1).

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100 \qquad (1)$$

D1 = First sample value
D2 = Second sample value (duplicate)

Duplicate samples will generally be collected and analyzed to achieve 5% duplicate data for each medium sampled. At least one duplicate sample will be collected and

analyzed for each medium sampled during each individual Phase II assessment. Table 4 in Appendix B further outlines duplicate sampling frequency. Additional duplicate sampling protocols may be outlined in the Site-Specific Sampling and Analysis Plans.

Field duplicate samples will be provided for each matrix (soil, groundwater, etc.) sampled. The minimum number of field duplicate samples required for each round of sampling is one for every 20 samples. If there are fewer than 20 samples per matrix, one field duplicate per matrix will be submitted.

Field sampling for ACM will follow AHERA sampling protocols. ACM sampling procedures are documented in Terracon's SOP for bulk asbestos sampling included in Appendix C of this QAPP.

1.5.1.2 Laboratory Precision Objectives

The precision of laboratory analyses will be based upon laboratory MS/MSD analyses performed in accordance with laboratory-specific procedures set forth in Appendices D and E. If insufficient sample volume is available for MS/MSD analysis, LCS/LCSD sample will be used to determine laboratory precision. The laboratory will perform MS/MSD analyses at a rate of one for every 20 investigative samples.

RPD or matrix precision will be calculated using the equation in Section 4.3.1 of this QAPP. Results will be calculated and compared to the laboratory-specific RPD objectives set forth in Appendices D and E. Except for PLM analysis for asbestos, RPD values within 20% will be considered precise without further discussion. For PLM analysis for asbestos, an RPD limit of 1% will apply. Relative Percent Difference values beyond these limits may require further qualification of associated data, but data will not be rejected unless determined unusable through the data validation process.

Precision QC limits for ACM duplicate samples are 1%. The precision QC limit for lead testing of paint chips is 20%.

1.5.2 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.

1.5.2.1 Field Accuracy Objectives

The objective for accuracy of the field sample collection procedures will be to ensure that samples are not 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. Trip blanks will be submitted at the rate of one trip blank per shipping container containing field 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.

Equipment blanks will be collected by pouring laboratory-prepared water or distilled water over or through reusable 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 U.S. EPA is one per 20 field samples per matrix or, if less than 20 samples are collected, one equipment blank per day per sample matrix.

Trip and equipment blanks will be analyzed during assessment activities in order to assess potential problems as they occur.

1.5.2.2 Laboratory Accuracy Objectives

STAT and Pace 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 accuracy of the organics analyses also will be monitored through analysis of surrogate compounds. Surrogate compounds are added to each sample, 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.

In addition, please see Table 8 and Appendix D and E of this QAPP, for the laboratory's QA objectives.

1.5.3 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.

1.5.3.1 Measures to Ensure 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. It will also be achieved by ensuring that sampling locations are properly selected. Representativeness is dependent upon the proper design of the sampling program and will be accomplished by ensuring that this QAPP, the property-specific SAPs, and standard procedures are followed. 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 prior to groundwater sampling will ensure that representative samples are collected. Soil intervals will be homogenized for all analyses except VOCs to help ensure that representative soil samples are collected. Suspected ACM and lead based paint samples will be collected to ensure enough material is collected to accurately represent the bulk sample.

1.5.3.2 Measures to Ensure 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 ensure that the laboratory data is representative.

1.5.4 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 percent is desirable, an overall completeness goal of 90 percent may be realistically achieved under normal field sampling and laboratory analysis conditions.

1.5.4.1 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 percent of all samples be valid data. The equation for calculating completeness is presented in Section 4.3.5.1.

1.5.4.2 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 percent 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.

1.5.5 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.

1.5.5.1 Measures to Ensure Comparability of Field Data

Ensuring that this QAPP and the property-specific SAPs 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.

1.5.5.2 Measures to Ensure 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 to help ensure that the data will produce comparable results. Table 8 summarizes the laboratory reporting limits.

1.5.6 Sensitivity

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

1.5.6.1 Measures to Ensure Field Sensitivity

The sensitivity of the field instruments selected to measure temperature, conductivity, turbidity, and the dissolved oxygen (DO) 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 photoionization detector (PID) used to screen samples for organic vapors is relative to background readings in ambient air.

1.5.6.2 Measures to Ensure Laboratory Sensitivity

The sensitivity requirements for laboratory analyses are to be such to an extent as to meet IEPA standards for both soil and groundwater, EPA ACM standard of 1%, IEPA SRP, and the U.S Department of Housing and Urban Development (HUD) standard for lead-based paint of 0.5 percent by weight. If analytical methods are deemed to be insufficiently sensitive, alternative analytical methods may be utilized.

Additionally, minimum laboratory detection limits which exceed IEPA standards will be evaluated in the following manner:

- Is the compound expected to be a chemical of concern, or, if the reporting limit exceeds IEPA groundwater standards, was the compound detected in the surrounding soils? If the compound is not an expected COC or detected in the soils, then the compound will be considered nondetect. If the compound is considered a COC or was detected in the surrounding soils, the compound will be evaluated using half the detection limit.
- If the reported detection limit exceeds IEPA groundwater standards, does the compound have an established Federal maximum contaminant level (MCL), and if so, does the reporting limit meet the MCL? If the reporting limit is at or below the MCL, the compound will be considered nondetect. If the reporting limit exceeds the MCL, the compound will be evaluated using half the reported laboratory detection limit.

Table 8 in Appendix A presents the laboratory reporting limits.

1.6 Documentation and Records

Records generated during Phase II activities are a critical part of any property assessment. Terracon 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. City of Chicago will retain the records generated during assessment activities for a minimum of 10 years following the completion of this project.

At a minimum, the draft and final Phase II Site Assessment report submittal packages 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.
- Tables comparing all laboratory data to the applicable standards.
- Tables summarizing QA/QC analytical results.
- Complete laboratory data reports, including copies of all COC records.

- Copies of soil boring, groundwater, sediment, and surface water sampling logs.
- Other relevant material needed to support property redevelopment.
- Data Assessment Report 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.

2.0 DATA GENERATION AND ACQUISITION

The purpose of the QAPP is to produce reliable data that will be generated throughout the assessment 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.

2.1 Sampling Process Design

Sample locations, analytical parameters, and frequency of sampling will be discussed in the property-specific SAPs. The laboratory SOPs for these analytical parameters are presented in Appendix D and E.

Analytical parameters will be chosen based on representative contaminants most commonly associated with the former activities and/or identified areas at each property.

Sampling will occur as a stepwise process. During initial sampling activities, it is expected that a variety of chemicals of concern will be analyzed. The initial results may indicate that only certain chemicals of concern are present. Therefore, later rounds of sampling will include only those specific compounds or class of compounds present in the initial sampling events.

QA/QC samples will be submitted in accordance with the QAPP protocols presented in the following sections.

Sampling and data acquisition designs for Phase II assessment will be dependent on Phase I ESA findings and previous assessment results, if applicable. As indicated, individual sampling designs will be documented in the Site-Specific Sampling and Analysis Plans to be approved by USEPA. The following considerations will be made in development of sample collection and analysis designs.

- property and facility history
- adjacent and nearby property and facility history
- state and federal regulatory history
- potential source areas
- types and quantities of contaminants
- potential human and/or ecological targets
- surface topography/drainage and adjacent water bodies
- floodplain information
- geology, hydrogeology, and other relevant physical setting information
- site accessibility to the public
- site accessibility for sampling relative to on-site obstructions
- existing records relative to site conditions and previous contaminant releases
- current and prospective land use associated with redevelopment
- project budget

In conjunction with professional judgment, these considerations will be the primary basis behind property-specific sampling designs. The approach will focus on relevant contaminants of concern and areas of concern, while maximizing sampling efficiency. Repeated evaluations may be performed to generate three-dimensional designs. Additional considerations will be documented in the Site-Specific Sampling and Analysis Plans.

Sampling approaches will be applied to assess known or likely source areas. Contaminated areas may be non-point source or point source. The following sampling designs may be relevant for investigation activities.

IEPA Criteria (non-point source) – Non-point source sites will be assessed at equally spaced sampling locations placed in a grid pattern. The sample density will be no less than the SRP-required minimum of one soil boring location per quarter-acre.

Judgmental Sampling (point source) - Sampling locations are selected based on known or potential sources area and professional judgment. Locations are selected in response to specific indicators such as surface staining, stressed vegetation, waste storage locations, or other relevant indicators and RECs.

One or a combination of the approaches discussed above may be applicable and applied during any specific investigation. These approaches may also be applied in conjunction with composite sampling, a process in which two or more sample aliquots are homogenized to generate a single composite sample. Composite sampling will generally be applied as a screening tool, particularly in the case of IDW disposal characterization, and to assess nonvolatile impacts (e.g., metals) to soils encompassing large areas. With the exception of IDW samples, composite soil/sediment samples for VOC analysis or composite water samples for analysis will not be collected.

Three general categories of data will be collected during the project: 1) screening data without definitive confirmation results; 2) screening data with definitive confirmation results; and 3) definitive data. These data measurement categories are based on the level of precision and accuracy confidence of the results. The following paragraphs describe the three data categories in detail.

Screening Data Without Definitive Confirmation - This data is considered to be of uncertain precision and accuracy and is therefore considered qualitative in nature. This data will generally be collected as a preliminary screening tool to evaluate probability of environmental impact and the need for more definitive results. This category usually involves rapid measurement with limited QA/QC procedures. Examples include PID, colorimetric, parametric immuno-assay tests, and unconfirmed XRF screening using various portable field instruments.

Screening Data With Definitive Confirmation - This data is considered to be of higher precision and accuracy quality than data without definitive confirmation. This category is generally of known quality that is verified through calibration and comparison to definitive data. A relevant example is XRF data with certain levels of high-quality laboratory verification data in support. Generally, 5% to 10% of screening data need be supported by definitive data, as outlined in USEPA analytical methods. A higher level of confidence may be attributed to these screening data if strong verification data correlations are observed.

Definitive Data - This data relies on USEPA-approved analytical methods and specific QA/QC procedures to verify the precision and accuracy of the results. Definitive data is generally of the highest confidence and quality and is analyte-specific. Analyte identities and concentrations are verified according to the analytical method and associated QA/QC procedures. Typically, definitive data

will be generated by an accredited, off-site laboratory. Certain field data (e.g., XRF) may be considered definitive in nature if strong correlations are observed through statistical analysis. The degree of correlation required to be considered definitive is identified by specific USEPA analytical methods.

Under the SRP, the IEPA can issue an NFR letter based only on definitive data produced by an accredited laboratory. However, the two "lesser" categories are used to support the definitive data/NFR status.

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 STAT and Pace SOPs (Appendix D and E). Laboratories will coordinate all analytical services for this assessment. The specific analytical method and reporting limits for each parameter are presented in Table 8. Preparatory methods for analytical parameters are discussed in the laboratory SOPs included in Appendix D and E.

Proper sample containers, preservation, holding times, and volumes for each analytical parameter are outlined in Appendix D and E. STAT and Pace will provide all sample containers and preservatives for this project. Sample containers for groundwater VOC analysis will be pre-preserved with hydrochloric acid (HCL) by the laboratory. In addition, sample containers for groundwater cyanide analysis will be pre-preserved with NaOH. Soil containers for VOCs will be pre-preserved with methanol (5mLs for 5g soil) and sodium bisulfate solution (5mLs for 5g soil) and integral stir bars to allow the laboratory to analyze high and low levels as necessary.

All sample containers supplied by STAT and/or Pace will be cleaned according to U.S. EPA 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 STAT and Pace. Additionally, STAT and Pace shall provide the field team with trip blanks for VOC analysis.

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 procedure will be used to document the authenticity of data collected during the City of Chicago Brownfield Assessment Project. 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 City of Chicago Brownfield Assessment Project. Entries will be dated and signed by personnel recording the data. The entries will be made in ink. 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 any other information pertinent to the assessment.

2.3.1.2 Field Identification System

Each sample collected during property assessments will be given a unique identification code. Each unique sample identification will consist of the following:

- *Project Identification Code.*

A two-letter designation will be used to identify the property from which the sample was collected. Examples of this include the following:

SG – Smitty's Gas Station
BF – Bulk facility

- *Sample Matrix Code.*

Each sample will be further identified by a code corresponding to the sample matrix:

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

- *Location Code*

Each sample will be identified by a location code and interval as follows (note that surface water, sediment, and surface soil samples will be numbered consecutively and not given an additional location identifier):

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

- *Depth Code*

Multiple samples from a single boring will be identified by a depth code corresponding to the sample interval as follows.

2-4 = Two to four foot sample interval

- *Examples.*

SG-GW-MW-01 = groundwater sample from Monitoring Well 1 Smitty's Gas Station property

SG-GW-MW-01-FD = duplicate groundwater from MW 1

SG-SB-GP-01-2-4 = subsurface soil sample collected from Geoprobe boring 1 from 2-4 feet at Smitty's Gas Station property.

Sample bottle labels appropriate for the size and type of containers shall be provided by the Laboratory. 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
- Type of preservative.

All labels will be completed in waterproof ink. An example of 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. Terracon field personnel are responsible for ensuring that COC procedures are followed. Field personnel will maintain custody of all samples until they are relinquished to another custodian, the laboratory, or to the freight shipper.

All samples must be catalogued on a COC form using sample identification codes. 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 located in Appendix C.

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 couriered or shipped via overnight mail to the respective laboratory. The laboratory will be contacted in advance to expect shipment. The COC forms will be sealed in a plastic bag and transported inside the sample cooler. In addition, any 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. Any 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. Upon relinquishing the sample cooler to the laboratory, Terracon 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 Terracon data manager and a second copy will be retained by the laboratory. The integrity of the custody seals shall be noted by STAT or PACE on the COC form upon arrival. In addition, the shipping label will be included with the COC form retained by the Terracon data manager.

2.3.1.5 Field Documentation

Field COC procedures will ensure the proper documentation of each sample from collection in the field to delivery at the laboratory. Custody of samples shall 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
- Shipping documents

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

2.3.2 Laboratory Chain-of-Custody

The laboratory 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 custody protocol is maintained. Pace Analytical's SOP for sample custody is presented in Section 2.0 of the Laboratory QA Manual (Appendix D). STAT Analysis's SOP for sample custody is presented in Section 8 of the Laboratory QA Manual (Appendix E).

2.3.3 Final Evidence Files Custody Procedure

Terracon 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. Terracon will retain this file for a period of 10 years after completion of the assessment.

2.4 Quality Control Requirements

Project QC will be maintained by performing assessment activities in general accordance with this QAPP, Property-Specific Sampling and Analysis Checklists (SAPs), referenced SOPs, and other relevant guidance documents. Laboratory samples will be analyzed and reported in accordance with the specific analytical methods applied and the lab QA Manual or other laboratory quality management plans. To ensure generation of valid and defensible data, sampling and analysis procedures will adhere to the specific QC and data validation requirements discussed herein. In addition, project activities will be conducted by trained and experienced personnel under the oversight of the senior project manager and the QA reviewer.

2.4.1 QC Sample Summary

Table 4 in Appendix A summarizes specific quality control samples to be collected during the project, associated sampling/analysis frequency, and additional details regarding the purpose of the QC samples. The SAP will provide additional information regarding QC sampling and specific analytical methods.

Soil samples that are submitted for laboratory MS/MSD analysis will have an additional set of samples collected from the sample location (e.g., the number of containers submitted will be doubled). For water, two additional sets of samples will be submitted (e.g., the number of containers submitted will be tripled). The field crew will select soil and water MS/MSD samples from relatively not impacted (based on field screening and visual observations) sample locations.

Quality control samples will be assigned sample identifiers exclusive to the sample, but consistent with labeling of additional samples to prevent laboratory bias. These samples will be handled, preserved, and stored consistent with procedures outlined in this QAPP to ensure data quality. Quality control sample collection will be documented in the field logbook.

2.5 Instrument Calibration and Frequency

The calibration procedures to be employed for both the field and laboratory instruments used during the City of Chicago Brownfield Assessment Project are referenced in this section. Measuring and test 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 the respective laboratories. Terracon field personnel are responsible for the calibration of Terracon field equipment and field equipment provided by subcontractors.

Documented and approved procedures will be used for calibrating measuring and testing equipment. Widely accepted procedures, such as those published by U.S. EPA and 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 Terracon 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 Terracon 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 subcontractor field equipment and Terracon equipment used only for this specific project will be kept in the project files. STAT and Pace 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, pH meters, conductivity meters, and temperature probes. 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 located in Appendix C.

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,
- Any corrective action.

Trained personnel will operate field measurement equipment in accordance with the appropriate standard procedures or manufacturer's specifications. Terracon field technical staff members will examine field measurement equipment used during field sampling to verify that they are in operating condition. The Terracon field team leader will periodically audit the calibration and field performance of the field equipment to ensure 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 U.S. EPA-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 U.S. EPA. 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 quality control, calibration procedures, and corrective action procedures are discussed in Sections 4, 6, and 9 in the Pace QA Manual and in Sections 4, 5, and 10 in the STAT QA Manual. Instrument preventative maintenance is discussed in Section 6 in the PACE QA Manual and in Section 7 in the STAT Manual. Pace and STAT manuals are included in Appendices D and E of this QAPP, respectively.

2.6 Data Management

Terracon field technical staff members will manage raw data during field activities. Data such as geologic profiles and monitoring well construction details will be

recorded on the appropriate field forms (examples of which are located in Appendix F) or in field logbooks. The Terracon data manager will periodically collect data gathered during assessment activities in order to maintain results. As appropriate, the Terracon data manager will coordinate transfer of raw data to computer formats such as Microsoft® Excel or Microsoft® Access to better organize and track incoming data. This will enable the Terracon data manager to identify any data gaps. Any flaws in field QA/QC will be brought to the attention of the Terracon QA manager.

The laboratory project manager will be responsible for laboratory data management. Laboratory procedures for data review and data reporting are discussed in Section 7 of Pace's Manual and in Section 9 of STAT's manual, located in Appendix D and E of this QAPP, respectively. Analytical data reports generated by the laboratory will present all sample results, including all 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, explanation of all lab applied qualifiers; all sample results including the % moisture content for soil samples, the spike and duplicate analysis results (or MS/MDS results) including the % recoveries and RPDs. The following data must be available upon request from the lab on a case by case basis, if data issues arise:

- summaries of daily calibration check samples (including notation of any outliers)
- calibration blank results
- surrogate results including % recoveries (as applicable per analysis)
- the method blank results
- lab control sample (LCS) results including % recoveries

Soil results will be reported on a dry weight basis. All data, including QA/QC results, will become part of the project files and will be maintained by the Terracon data manager. Upon report delivery, Terracon personnel will analyze laboratory data in accordance with accepted statistical methodologies and will be supervised by the Terracon data manager.

3.0 ASSESSMENT/OVERSIGHT

Performance and system audits will be completed to ensure that the field sampling activities and laboratory analyses are performed following the procedures established in this QAPP, including the attached SOPs, and the property-specific SAPs. 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 Terracon geologist will be present at the site during sampling activities. The geologist will provide the on-site supervision required during the project. The geologist will be in daily contact with the Terracon 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 Project Manager and U.S. EPA project manager. Terracon field technical staff members will report modifications to the Terracon 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 to verify reproducibility. Equipment blanks and trip blanks will be measured for quality control purposes.

3.1.2 Field Screening Instruments

Terracon field technical staff members will audit and maintain the performance of field-screening instruments. Instruments will be calibrated according to the standard procedures located in Appendix C, and regular preventative maintenance will be performed as described in Table 5 in Appendix A.

3.1.3 Report Preparation

Prior to submittal to City of Chicago and U.S. EPA, all reports will undergo a peer review conducted by a project team within Terracon. All components of the report will be checked by a designated team member. City of Chicago will also review all reports prior to submittal to U.S. EPA.

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 Terracon Senior Project Manager will conduct audits of field activities. U.S. EPA may also conduct an independent field audit. At least one field audit will be completed near the beginning of the sample collection activities for each assessment. If a second phase of field activities is necessary and the second phase starts more than 6 months following the initial phase, then a second field audit will be completed. A copy of the QC Field Audit Corrective Action Form is included in Appendix H.

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. A copy of the Field Audit Form is included in Appendix H.

3.2.2 Laboratory Audits

STAT and/or Pace will perform many, if not all, of the analytical services required during the assessments. As discussed in Section 1.4.1.2, both laboratories are IEPA accredited laboratories, and a copy of their IEPA accreditation is located in

Appendix D and E. In addition, if any asbestos sampling is performed, analysis will be performed by STAT, an NVLAP-certified laboratory. A copy of their NVLAP certificate is also contained in Appendix E. The Laboratory QA manager will be responsible for ensuring that the laboratory data precision and accuracy are maintained in accordance with specifications and laboratory SOPs. As IEPA-accredited labs, STAT and Pace are routinely audited by the State of Illinois and/or the NELAP Accrediting Authority.

3.3 Reports to Management

For the duration of the project, monthly reports will be prepared by the Terracon project manager and submitted to the City Project Manager and U.S. EPA project manager. These reports will serve to inform the Project Manager and U.S. EPA of the project progress and any significant interim findings that have been identified. This will streamline the process of addressing issues as they arise and adjusting the program to better define the environmental concerns. At the completion of the assessment, the project reports will be issued.

To note, a Property-Specific Sampling and Analysis Plan (SAP), dated June 31, 2011, and associated Health and Safety Plan for the site located at 545 North Central Park Avenue (Proposed Ujima Gardens) was previously submitted under a separate cover.

4.0 DATA VALIDATION/USABILITY

This section describes the QA activities that will be performed to ensure that the collected data are scientifically defensible, properly documented, and of known quality, and meet project objectives. All analytical data collected for the City of Chicago Brownfield Assessment Project will be validated.

The following three steps will be followed to ensure 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. The group that generates the data 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 purpose of the project (i.e., to answer the environmental questions or to make environmental decisions). 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 all 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 all field samples, standards, QC checks and QC samples, and all 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. Terracon 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. Terracon will note deviations from the specifications, and discuss them with the U.S. EPA project manager.

- *Sample Collection Procedures* – Sample collection procedures identified in this QAPP will be followed. If field conditions require deviations, they will be discussed with the U.S. EPA project manager.

- *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 occur during sample handling affecting the integrity of the samples.

Terracon field technical staff members will evaluate the sample containers and the preservation methods used and ensure 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 to ensure 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 ensure 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 specified in an earlier section of 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 ensure 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; and
  - Had acceptable linearity checks and other checks to ensure 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 rekeying of data to identify data entry errors. Section 7 of Pace's QA Manual and Section 9 of STAT QA Manual (Appendix D and E, respectively) discusses their 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 Terracon QA manager by reviewing field documentation and chain-of-custody records. Data from direct-reading instruments 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 STAT and Pace are discussed in the QA Manuals (Appendix D and E), 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. All 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 Terracon consistent with the procedure identified in Section 1.5 of this QAPP. The data verification/validation procedure will identify data as being acceptable, of limited usability qualified or estimated, or rejected. The conditions that result in data being qualified or estimated or rejected are identified in Section 1.5 of this QAPP. The results of the data verification/validation will be provided in data validation memoranda that are provided to Terracon's Project Manager and are included in the Quality Assurance Management Reports. All sampling, handling, field analytical data, and fixed-laboratory data will be validated by entities external to the data generator. The

validation procedure will specify the verification process of every quality control measure used in the field and laboratory. Data validation procedures followed by STAT and Pace are discussed in the QA Manuals (Appendix D and E).

Each analytical report will be reviewed for compliance with the applicable method and for the quality of the data reported.

Data determined to be unusable may require that corrective action be taken. Potential types of corrective action may include resampling by the field team or reanalysis of the samples by the laboratory. The corrective actions taken are depending upon the ability to mobilize the field team and whether the data are critical for the project DQOs to be achieved. Should Terracon's QA Officer identify a situation requiring corrective action during data verification/validation, Terracon'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 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 1.5 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 formula:

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

where: R_1 = value of first result
R_2 = value of second result

Overall precision for the sampling programs will be determined by calculating the mean RPD for all 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
- 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 Data Assessment Report. It should be noted that the Data

Validation Report is considered to be the QA/QC report supplied by the analytical laboratory, and the Data Assessment Report will be prepared by Terracon and submitted as part of the Phase II document.

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 Data Assessment Report should address how this problem will be resolved and discuss the need for resampling.

4.3.2 Accuracy/Bias

In order to meet the needs of the project, data must meet the measurement performance criteria for accuracy. The methods discussed in Section 1.5.2 will be implemented so that the data is accurate.

Sample Contamination

Equipment and trip blank results will be reviewed to evaluate the potential for contamination during field and/or transport activities. If the potential for field and/or transport contamination is identified, the Data Assessment Report section of the investigation reports will evaluate the impact of the contamination on data usability. 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.

Analytical Accuracy

The laboratory will evaluate accuracy by reviewing the results of LCS, MS, MSD, or surrogate analysis. Laboratory analytical deliverables will flag results that are outside the designated acceptance limits. These notes will provide an explanation as to the potential impact on data quality. If the laboratory analytical reports indicate 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 Data Assessment Report section of the investigation reports.

Overall Accuracy

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 procedure 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 (ten 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 will 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 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.

The Data Assessment Report section of the investigation reports will discuss and compare overall accuracy data from multiple data sets collected for the project for each matrix, analytical parameter, and concentration level. The reports will describe the limitations on the use of the project data if extensive inaccuracy exists or when it is limited to a specific sampling or laboratory analytical group, data set, matrix, analytical parameter, or concentration level. The investigation reports will identify qualitative and/or quantitative bias trends for each matrix, analytical parameter, and concentration level. The impact of qualitative and/or quantitative trends in bias on the sample data will be discussed.

When the required level of accuracy is not achieved and project data is not usable to adequately address environmental questions and to support project decision making, then the investigation reports will address how this problem will be resolved and will discuss the 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 1.5.3.

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 this may trigger additional scoping meetings and subsequent resampling in order to collect data that are more representative of a nonhomogeneous site. Overall sample representativeness will be determined by calculating the percent of field duplicate sample data that achieved the RPD criteria specified in Section 1.5.3 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 percent of the samples.

The Data Assessment Report will discuss and compare overall representativeness for each matrix, parameter, and concentration level. Data Assessment Reports will describe the limitations on the use of project data when overall nonrepresentative sampling has occurred or when nonrepresentative 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 Data Assessment Report will address how this problem will be resolved and discuss potential need for resampling.

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. Document the procedures for calculating method detection limits (MDLs) and quantitation limits (QLs).

The quantitation limits for the sample data will be reviewed to ensure that the sensitivity of the analyses was sufficient to achieve IEPA standards. 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 1.5.6 of this QAPP.

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 samples are less than the acceptable evaluation criteria (i.e., IEPA standards).

It should be noted that quantitation limits may be elevated as a result of high concentrations of target compounds, nontarget 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 Data Validation Reports indicate that sensitivity and/or QLs were not achieved, then the impact of that lack of sensitivity and/or higher QLs on data usability will be discussed in the Data Assessment Report.

The Data Assessment Report will discuss and compare overall sensitivity and QLs from multiple data sets collected for the project for each matrix, analytical parameter, and concentration level. The Data Assessment Report will describe the limitations on the use of the project data if project-required sensitivity and QLs 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 QLs are not achieved and project data are not usable to adequately address environmental questions and to support project decision making, then the Data Assessment Report will address how this problem will be resolved and discuss the potential need for resampling. In this case, the Data Assessment Report 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.

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 Phase II will be calculated when all sampling and

analysis is concluded. Overall completeness will be considered acceptable if at least 90 percent of the data are determined to be valid.

The Data Assessment Report will discuss and compare overall completeness of multiple data sets collected for the project for each matrix, analytical parameter, and concentration level. The Data Assessment Report 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 Data Assessment Report will address how this problem will be resolved and discuss the potential need for additional resampling.

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 and any approved QAPP revisions or amendments are used for generating the soil, groundwater, sediment, and surface water data.

The Data Assessment Report will discuss and compare overall comparability between multiple data sets collected for the project for each matrix, analytical parameter, and concentration level. The Data Assessment Report 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.

For long-term monitoring projects, data comparability is extremely important. Project data will be compared to previously generated data to determine the possibility of false positives and/or false negatives. Variations detected in the data may reflect a changing environment or indicate sampling and/or analytical error. Comparability

criteria will be established to evaluate these data sets in order to identify statistical outliers to trigger resampling as verified.

If it is determined that long-term monitoring data are not comparable, the Data Assessment Report will address whether the data indicate a changing environment or the anomalies are a result of sampling and/or analytical error. If data are not usable to adequately address environmental questions and/or support project decision making, then the Data Assessment Report will address how this problem will be resolved.

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 Data Assessment Report 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 Data Assessment Report 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 Data Assessment Report will address how this problem will be resolved and discuss potential need for resampling.

Overall comparability of data from the groundwater monitoring program will be assessed by evaluating analyte concentrations over time. The data from monitoring events will be evaluated for trends, if necessary, using the Mann-Kendall test described in Section 4.3.4.1 of EPA QA/G-9. Suspected outliers will be assessed using the Extreme Value Test described in Section 4.4.3 of EPA QA/G-9. As the groundwater database becomes larger, it may be necessary to use different statistical methods to determine trends and outliers. Any changes to the statistical

methods used for this project will be communicated to the U.S. EPA prior to initiating the change.

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 within the Phase II. 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.

5.0 REFERENCES

United States Environmental Protection Agency. 1993. *Data Quality Objectives Process for Superfund: Interim Final Guidance*. EPA 540-R-93-071, Office of Research and Development, Washington DC.

United States Environmental Protection Agency. 1994. *Guidance for Data Quality Assessments*. EPA QA/G-5, Office of Research and Development, Washington DC.

United States Environmental Protection Agency. 1996. *Guidance for the Data Quality Assessment Process: Practical Methods for Data Analysis*. EPA QA/G-9, Office of Research and Development, Washington DC.

Appendix A

Appendix A - Tables

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- STAT PNAs EPA (8270C SIM) - Groundwater
- STAT VOCs EPA (8260) - Groundwater
- STAT Pesticides EPA (8081) - Groundwater
- STAT PCBs EPA (8082) - Groundwater

Table 1 - QA Objectives for Field Measurements

Media	Parameter	Method Reference	Precision	Accuracy	Completeness
Water	Standing Water Level	Solinst Water Level Indicator, TSOP E.1820	±0.01 feet	.001 feet	90%
	Temperature	Horiba U-22 Water Quality Instrument	±0.3°C	1.0°C	90%
	Conductivity	Horiba U-22 Water Quality Instrument	0.10%	0.30%	90%
	pH	Horiba U-22 Water Quality Instrument	±0.02 pH Units	0.1 pH Units	90%
	DO	Horiba U-22 Water Quality Instrument	±0.1 mg/L	0.2 mg/L	90%
	Turbidity	Horiba U-22 Water Quality Instrument	±3%	5% NTU	90%
Soil	pH	HACH Instrument, TSOP E.530	Not Part of Method	0.05 pH Units	90%
	PID	Mini Rae Instrument	0.1 ppm	0.1 ppm	90%

Table 2 - Laboratory Summary

Laboratory / Location	Contaminant of Concern	USEPA Analytical Method[1]
Pace Analytical Services (Green Bay, WI) STAT Analysis Corporation (Chicago, IL)	VOCs	5035/8260B
	SVOCs	8270C
	PAHs (groundwater)	8270 SIM
	PCBs	8082
	Pesticides	8081A
	RCRA Metals (except mercury)	6010B/6020
	Mercury	7471A
	Cyanide	9012
	TCLP Extraction	1311
	SPLP Extraction	1312
	TPH (DRO/GRO/ERO)	8015
	pH	9045C
	TOC	9060 / 415.1
	foc	3550
	ORP	SM[2] 2580
	Paint Filter	9095A
	Total Organic Matter	ASTM D 2974-00
	VOCs	8260B
	SVOCs (groundwater)	8270C
	PAHs (groundwater)	8270C/ 8310
	PCBs	8082
	Pesticides	8081A
	RCRA Metals (except mercury)	6010B/6020
	Mercury	7470A
	Cyanide	9012
	pH	9040B
Pace Analytical Services (Green Bay, WI)	Flashpoint	1010A
STAT Analysis Corporation (Chicago, IL)	PAHs (soil)	8270 SIM
	Reactive Cyanide	7.3.3.2
	Reactive Sulfide	7.3.4.2
	Total Phenol	9065
	Hexavalent Chromium	7196A
	Bulk Asbestos Samples	EPA-66/M4-82-020
	Paint Chips for Lead Testing	7420

[1] = Extraction/Analysis Holding Times

[2] = Standard Methods for the Examination of Water and Wastewater. Measurement of ORP is required when analyzing soil/sediment samples for hexavalent chromium.

Table 3 - Sample Container, Preservation and Holding Time Requirements

Matrix	Contaminant of Concern	Packaging and Preservation	Maximum Holding Times
Soil	VOCs	(1) 40-mL vial with 5 mL Methanol, (2)-40mL vials with 5 mL Sodium Bisulfate, 2oz CWM[1], 4°C	ASAP/14 days
	SVOCs (including phenols)	4oz CWM, 4°C	14/40 days
	PAHs	4oz CWM, 4°C	14/40 days
	PCBs	4oz CWM, 4°C	14/40 days
	Pesticides	4oz CWM, 4°C	14/40 days
	RCRA Metals (except mercury)	4oz CWM, 4°C	None/180 days
	Hexavalent Chromium	4oz CWM, 4°C	None/28 days
	Mercury	4oz CWM, 4°C	None/28 days
	Cyanide	4oz CWM, 4°C	None/14 days
	TCLP Extraction (Mercury)	4oz CWM, 4°C	28 days, then method-specific hold times
	SPLP Extraction (Mercury)	4oz CWM, 4°C	
	TCLP Extraction (Other Inorganics)	4oz CWM, 4°C	180 days, then method-specific hold times
	SPLP Extraction (Other Inorganics)	4oz CWM, 4°C	
	TCLP Organic (VOCs)	4oz CWM, 4°C	14 days
	TCLP Organic (SVOCs)	4oz CWM, 4°C	21 days
	foc	4oz CWM, 4°C	28 days
	Flashpoint	4oz CWM, 4°C	30 days
	Paint Filter	4oz CWM, 4°C	None
	Total Phenol	4oz CWM, 4°C	28 days
	Reactive Cyanide	4oz CWM, 4°C	14 days
	Reactive Sulfide	4oz CWM, 4°C	7 days
	TCLP Organics	4oz CWM, 4°C	14 days
	TPH (DRO/GRO/ERO)	4oz CWM, 4°C	10 days
	ORP	4oz CWM, 4°C	None/ASAP
	pH	4oz CWM, 4°C	None/ASAP
	Total Organic Matter	CWM, minimum 500 g of sample	None
Groundwater	VOCs	(3) 40-mL glass vials with HCl, pH<2, 4°C	None/14 days
	SVOCs (including phenols)	(2) 1-L Amber Glass with Sodium Thiosulfate, 4°C	7/40 days
	TPH (DRO/GRO/ERO)	(2) 1-L Amber Glass, 4°C	7/14 days
	PAHs	(2) 1-L Amber Glass, 4°C	7/40 days
	PCBs	(2) 1-L Amber Glass, 4°C	7/40 days
	Pesticides	(2) 1-L Amber Glass, 4°C	7/40 days
	RCRA Metals (except mercury)	250-mL HDPE[2] with HNO3, pH<2, 4°C	None/180 days
	Hexavalent Chromium	250-mL HDPE, No Preservative, 4°C	None/24 hours
	Mercury	250-mL HDPE with HNO_3, pH<2, 4°C	None/28 days
	Cyanide	250-mL HDPE with NaOH, pH>12, 4°C	None/14 days
	pH	125-mL, HDPE, No Preservative, 4°C	None/ASAP
Bulk Suspect ACM	Bulk Asbestos Samples	Sealed container (e.g, Whirl-Pak® sample bag or equivalent)	None
Paint Chips	Paint Chips for Lead Testing	Sealed container (e.g, Whirl-Pak® sample bag or equivalent), minimum 0.05 g of sample	None

[1] = Clear, Wide-Mouth glass jar

[2] = High-Density Polyethylene

Table 4 - Quality Control Sample Collection

QC Sample Type	Frequency of Sample / Analysis	Details
Equipment Blanks (rinsate)	One per field mobilization (per site) per media sampled; not applicable for ACM or LBP	Distilled water placed into contact with sampling equipment; used to assess quality of data from field sampling and decontamination procedures
Trip Blanks	One per sample cooler used to store and transport VOC samples	Laboratory prepared organic-free blank to assess potential contamination during sample container handling, shipment, and storage
Field Duplicate Samples	One per 20 investigative samples per site for each media sampled; a minimum of one duplicate sample for each media sampled each sampling phase will be collected	Duplicate sample collected by the same methods and at the same time as original sample; used to verify sampling and analytical reproducibility
Field Blanks	In the event that all simple-use equipment is being used and no rinsate blanks will be collected a field blank will be collected	Distilled water will be poured into blank glassware provided by the laboratory at the location the samples are collected
MS/MSD	Per Laboratory QA Manuals; will generally include a frequency of one per every 20 laboratory analyses	Laboratory spiked sample to evaluate matrix and measurement methodology
Method Blanks	Per Laboratory QA Manuals; will generally include a frequency of one per every 20 laboratory analyses	Laboratory blank sample to assess potential contamination from laboratory instruments/procedures
LCS and Duplicates	Per Laboratory QA Manuals; will generally include a frequency of one per every 20 laboratory analyses	Evaluates laboratory reproducibility

Table 5 - Field Equipment Mantenance Schedule

Instrument	Maintenance Procedures / Schedule	Spare Parts in Stock
Photoionization Detector	1. Calibrate beginning and end of each day and as necessary during use. 2. Check battery, and recharge when low. 3. Clean lamp and dust filter as needed. 4. Replace water traps if they become wet.	Battery charger Spare Lamps
pH Meter	1. Calibrate beginning and end of each day, and as necessary during use. 2. Replace electrodes as needed.	pH buffers Batteries Spare electrodes
Conductivity Meter	1. Calibrate beginning and end of each day, and as necessary during use. 2. Check redline and replace batteries if does not calibrate.	Batteries

Table 6 - Project Oversight and Audit Procedure

Assessment Activity	Agency Name	Project Position	Frequency of Activity
Project Field Audit	Terracon	Senior Project Manager	A minimum of one audit during project fieldwork
Third-Party Audit	IEPA	SRP Project Manager or QC Representative	Discretionary, will coincide with project fieldwork
Project QA Review	Terracon	QA/QC Reviewer	One audit before development of final report
Laboratory System Audit	Pace and Stat Analysis	Laboratory QA Officers	As discussed in Appendices C, E, F, and G

Table 7 - Project Reporting Documents

Document	Receiving Party	Prepared By	Submittal Frequency
QAPP	City, USEPA	Terracon	One submittal, with annual Terracon review for continued applicability
Property-Specific Sampling and Analysis Checklists	City, USEPA	Terracon	One submittal per assessment property; date contingent upon Phase I ESA schedule
Property-Specific Health and Safety Plans - Attachment to Property-Specific Sampling and Analysis Checklists	City, USEPA	Terracon	One submittal per assessment property; date contingent upon Phase I ESA schedule
Final Site Investigation Reports	City, IEPA*, USEPA	Terracon	One submittal per assessment property; schedule based on City review of draft
Property Profile Forms	USEPA	City Grant Director	For each property: Once at start and following assessment activities and/or significant changes in property conditions
Terracon Audit Reports	Terracon	Terracon	Within 30 days of field audit; minimum of one audit to be performed
IEPA Audit Reports	IEPA, Terracon	IEPA	Discretionary
Laboratory Audit Reports	As requested	Laboratory	As requested following laboratory audit
Laboratory Reports	Terracon and City (if requested)	Laboratory	Various, within 10 business days of each laboratory sample submittal
Field Analytical Reports	Terracon and City (if requested)	Various Parties	Various, within 10 business days of completion of field activities
Quarterly Progress Reports	USEPA	City Grant Director	Within 30 days of end of each federal fiscal quarter

* = For sites enrolled in the Illinois EPA Site Remediation Program.

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA RESIDENTIAL CRITERIA

Illinois Soil													
ANALYTE (& EPA METHOD)	CAS	Pace-GB	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
TOTAL METALS (6010)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/l)				
ALUMINUM	7429-90-5	50	-	-	-	-	410000	870000	3.5	80-120	20	75-125	20
ANTIMONY	7440-36-0	2	31	-	820	-	82	—	0.006	80-120	20	75-125	20
ARSENIC	7440-38-2	2	13	750	-	1200	61	25000	0.05	80-120	20	75-125	20
BARIUM	7440-39-3	0.5	5500	690000	140000	910000	14000	870000	2	80-120	20	75-125	20
BERYLLIUM	7440-41-7	0.4	160	1300	4100	2100	410	44000	0.004	80-120	20	75-125	20
CADMIUM	7440-43-9	0.5	78	1800	2000	2800	200	59000	0.005	80-120	20	75-125	20
CALCIUM	7440-70-2	20	-	-	-	-	—	—	—	80-120	20	75-125	20
CHROMIUM	7440-47-3	0.5	230	270	6100	420	4100	690	0.1	80-120	20	75-125	20
COBALT	7440-48-4	0.5	4700	-	120000	-	12000	—	1	80-120	20	75-125	20
COPPER	7440-50-8	1	2900	-	82000	-	8200	—	0.65	80-120	20	75-125	20
CYANIDE (9012A)	57-12-5	0.6	1600	-	41000	-	4100	—	0.2	80-120	20	80-120	20
HEXAVALENT CHROMIUM	18540-29-9	NA	230	270	6100	420	4100	690	—	NA	NA	NA	NA
IRON	7439-89-6	10	-	-	-	-	140000	—	5	80-120	20	75-125	20
LEAD	7439-92-1	1	400	-	800	-	700	—	0.0075	80-120	20	75-125	20
MAGNESIUM	7439-95-4	20	325000	-	-	-	730000	—	—	80-120	20	75-125	20
MANGANESE	7439-96-5	0.5	1600	69000	41000	91000	4100	8700	0.15	80-120	20	75-125	20
MERCURY (7471B)	7439-97-6	0.01	23	10	610	16	61	0.1	0.002	85-115	20	85-115	20
NICKEL	7440-02-0	1	1600	13000	41000	21000	4100	440000	0.1	80-120	20	75-125	20
POTASSIUM	7440-09-7	20	-	-	-	-	—	—	—	80-120	20	75-125	20
SELENIUM	7782-49-2	2	390	-	10000	-	1000	—	0.05	80-120	20	75-125	20
SILVER	7440-22-4	1	390	-	10000	-	1000	—	0.05	80-120	20	75-125	20
SODIUM	7440-23-5	100	-	-	-	-	—	—	—	80-120	20	75-125	20
THALLIUM	7440-28-0	4	6.3	-	160	-	160	—	0.002	80-120	20	75-125	20
VANADIUM	7440-62-2	0.5	550	-	14000	-	1400	—	0.049	80-120	20	75-125	20
ZINC	7440-66-6	4	23000	-	610000	-	61000	—	5	80-120	20	75-125	20

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B
TCLP/SPLP values are used for MIG TO GW.

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS AND IEPA SOIL CRITERIA

Illinois Soil													
ANALYTE (& EPA METHOD)	CAS	Pace-GB	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
SEMIVOLATLES (8270)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)				
1,2-Dichlorobenzene	95-50-1	0.167	7000	560	180000	560	18000	310	17	54-130	20	47-130	20
1,2,4-Trichlorobenzene	120-82-1	0.167	780	3200	20000	3200	2000	920	5	59-130	20	48-130	24
1,3-Dichlorobenzene	541-73-1	0.167	70	570	1800	570	180	570	0.2	51-130	20	40-130	21
1,4-Dichlorobenzene	106-46-7	0.167	-	11000	-	17000	—	340	2	51-130	20	45-130	21
2,2'oxybis (1-Chloropropane)	108-60-1	0.167	3100	1300	82000	1300	8200	1300	2.4	34-130	20	31-130	25
2,4,6-Trichlorophenol	95-95-4	0.167	58	200	520	390	11000	540	0.2	10-130	20	36-+130	29
2,4,5-Trichlorophenol	88-06-2	0.167	7800	-	200000	-	200000	—	270	67-130	20	36-130	24
2,4-Dichlorophenol	120-83-2	0.167	230	-	6100	-	610	—	1	69-130	20	46-130	28
2,4-Dimethylphenol	105-67-9	0.167	1600	-	41000	-	41000	—	9	61-130	20	30-130	32
2,4-Dinitrophenol	51-28-5	0.667	160	-	4100	-	410	—	0.2	29-130	20	10-130	40
2,4-Dinitrotoluene	121-14-2	0.167	0.9	-	8.4	-	180	—	0.0008	70-130	20	32-130	30
2,6-Dinitrotoluene	606-20-2	0.167	0.9	-	8.4	-	180	—	0.0007	70-130	20	48-130	26
2-Chloronaphthalene	91-58-7	0.167	6300	-	160000	-	160000	—	49	68-130	20	53-130	20
2-Chlorophenol	95-57-8	0.167	390	53000	10000	-	10000	53000	4	53-130	20	45-130	24
2-Methylnaphthalene	91-57-6	0.167	310	-	8200	-	820	—	7.2	66-130	20	52-130	20
2-Methylphenol (o-Cresol)	95-48-7	0.167	3900	-	100000	-	100000	—	15	51-130	20	45-130	24
2-Nitroaniline	88-74-4	0.167	230	35	6100	56	610	3.6	0.14	55-130	20	31-130	20
2-Nitrophenol	88-75-5	0.167	-	-	-	-	—	—	-	57-130	20	30-130	28
3-Nitroaniline	99-09-2	0.167	23	250	610	400	61	26	0.01	65-130	20	15-137	36
3,3-Dichlorobenzidine	91-94-1	0.167	1	-	13	-	280	—	0.007	52-139	20	10-169	50
3&4-Methylphenol (m&p-cresol)	106-44-5	0.167	390	-	10000	-	1000	—	0.2	53-130	20	43-130	25
4,6Dinitro-2-methylphenol	534-52-1	0.167	7.8	-	200	-	820	—	-	54-130	20	10-130	50
4-Bromophenylphenyl ether	101-55-3	0.167	-	-	-	-	—	—	-	65-130	20	36-130	25
4-Chloro-3-methlyphenol	59-50-7	0.167	5500	-	14000	-	41000	—	24	63-130	20	43-130	27
4-Chloroaniline	106-47-8	0.333	310	-	8200	-	820	—	0.7	49-130	20	18-130	28
4-Chlorophenylphenyl ether	7005-72-3	0.167	-	-	-	-	—	—	-	70-130	20	51-130	20
4-Nitroaniline	100-01-6	0.167	230	1000	6100	1600	610	110	0.1	55-140	20	10-142	50
4-Nitrophenol	100-02-7	0.167	630	-	16000	-	16000	—	-	46-134	20	10-130	50
Acenaphthene	83-32-9	0.167	4700	-	120000	-	120000	—	570	70-130	20	52-130	21
Acenaphthylene	208-96-8	0.167	2300	-	61000	-	61000	—	85	69-130	20	50-130	20
Anthracene	120-12-7	0.167	23000	-	610000	-	610000	—	12000	70-130	20	45-130	25
Benzo(a)anthracene	56-55-3	0.167	0.9	-	8	-	170	—	2	70-130	20	43-130	41
Benzo(a)pyrene	50-32-8	0.167	0.09	-	0.8	-	17	—	8	70-130	20	34-130	38
Benzo(b)fluoranthene	205-99-2	0.167	0.9	-	8	-	170	—	5	66-130	20	32-130	36
Benzo(ghi)perylene	191-24-2	0.167	2300	-	61000	-	61000	—	27000	43-149	20	16-148	50
Benzo(k)fluoranthene	207-08-9	0.167	9	-	78	-	1700	—	49	66-130	20	45-130	41
bis(2-Chloroethoxy)methane	111-91-1	0.167	-	-	-	-	—	—	-	59-130	20	54-130	21
bis(2-Chloroethyl)ether	111-44-4	0.167	0.6	0.2	5	0.47	75	0.66	0.0004	40-130	20	42-130	26
bis(2-Ethylhexyl)phthalate	117-81-7	0.167	46	31000	410	31000	4100	31000	3600	64-133	20	35-155	23
Butylbenzylphthalate	85-68-7	0.167	16000	930	410000	930	410000	930	930	60-136	20	31-154	27
Carbazole	86-74-8	0.167	32	-	290	-	6200	—	0.6	67-130	20	29-135	34
Chrysene	218-01-9	0.167	88	-	780	-	17000	—	160	70-130	20	46-130	30
Dibenzo(a,h)anthracene	53-70-3	0.167	0.09	-	0.8	-	17	—	2	45-139	20	21-132	50
Dibenzofuran	132-64-9	0.167	160	-	4100	-	4100	—	6.1	70-130	20	53-130	20
Diethylphthalate	84-66-2	0.167	63000	2000	1000000	2000	1000000	2000	470	70-130	20	51-130	22
Dimethylphthalate	131-11-3	0.167	780000	1300	1000000	1300	1000000	1300	380	70-130	20	63-130	25
Di-n-butylphthalate	84-74-2	0.167	7800	2300	200000	2300	200000	2300	2300	70-130	20	43-130	29
Di-noctylphthalate	117-84-0	0.167	1600	10000	41000	10000	4100	10000	10000	60-133	20	44-136	29
Fluoranthene	2063-44-0	0.167	3100	-	82000	-	82000	—	4300	59-130	20	22-130	38
Fluorene	86-73-7	0.167	3100	-	82000	-	82000	—	560	70-130	20	50-130	20
Hexachlorobenzene	118-74-1	0.167	0.4	1	4	1.8	78	2.6	2	68-130	20	44-130	20
Hexachloro-1,3-butadine	87-68-3	0.167	16	1000	410	1000	41	180	2.9	55-130	20	49-130	23
Hexachlorocyclopentadiene	77-47-4	0.167	550	10	14000	16	14000	1.1	400	13-130	20	10-130	50
Hexachloroethane	67-72-1	0.167	78	-	2000	-	2000	—	0.5	47-130	20	31-130	23
Indeno(1,2,3-cd)pyrene	193-39-5	0.167	0.9	-	8	-	170	—	14	37-150	20	16-142	50
Isophorone	78-59-1	0.167	15600	4600	410000	4600	410000	4600	8	17-130	20	19-130	20
Naphthalene	91-20-3	0.167	1600	170	41000	270	4100	1.8	12	60-130	20	51-130	21
Nitrobenzene	98-95-3	0.167	39	92	1000	140	1000	9.4	9.4	48-130	20	46-130	24
N-Nitroso-di-n-propylamine	621-64-7	0.167	0.09	-	0.8	-	18	—	0.00005	48-130	20	42-130	23
N-Nitrosodiphenylamine	86-30-6	0.167	130	-	41000	270	4100	1.8	1	70-135	20	42-151	36
Pentachlorophenol	87-86-5	0.167	3	-	24	-	520	—	0.03	47-130	20	10-130	38
Phenanthrene	85-01-8	0.167	2300	-	61000	-	61000	—	200	70-130	20	44-130	27
Phenol	108-95-2	0.167	23000	-	610000	-	61000	—	100	46-130	20	41-130	28
Pyrene	129-00-0	0.167	2300	-	61000	-	61000	—	4200	41-146	20	21-156	42

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B
Bolded number indicates value is less than laboratory reporting limit

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA RESIDENTIAL CRITERIA

Illinois Soil													
ANALYTE (& EPA METHOD)	CAS	Pace-GB	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
VOLATILES (8260)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)				
1,1,1-Trichloroethane	71-55-6	0.05	-	1200	-	1200	---	1200	2	67-130	20	55-159	20
1,1,2,2-Tetrachloroethane	79-34-5	0.05	4700	2000	120000	2000	12000	2000	3.3	70-130	20	59-143	25
1,1,2-Trichloroethane	79-00-5	0.05	310	1800	8200	1800	8200	1800	**0.02**	70-130	20	70-135	20
1,1-Dichloroethane	75-34-3	0.025	7800	1300	200000	1700	200000	130	23	70-130	20	67-133	20
1,1-Dichloroethene	75-35-4	0.05	3900	290	100000	470	10000	3	0.06	70-130	20	49-151	20
1,2-Dichlorobenzene	95-50-1	0.05	7000	560	180000	560	1400	0.99	17	70-130	20	70-130	20
1,2-Dichloroethane	107-06-2	0.05	7	0.4	63	0.7	1800	0.5	**0.02**	70-130	20	60-145	20
2-Butanone (MEK)	78-93-3	0.25	47000	25000	1000000	25000	120000	710	17	50-150	20	50-150	20
2-Hexanone	591-78-6	0.25	3100	70	82000	110	8200	0.72	1.3	50-150	20	50-150	20
4-Methyl-2-pentanone (MIBK)	108-10-1	0.05	-	3100	-	3100	---	340	-	50-150	20	46-150	20
Acetone	67-64-1	0.25	70000	100000	-	100000	---	100000	25	45-150	20	50-150	20
Benzene	71-43-2	0.025	12	0.8	100	1.6	2300	2.2	0.03	70-130	20	66-130	20
Bromodichloromethane	75-27-4	0.05	10	3000	92	3000	2000	3000	0.6	70-130	20	52-150	20
Bromoform	75-25-2	0.05	81	53	720	100	16000	140	0.8	68-130	20	43-143	20
Bromomethane	74-83-9	0.05	110	10	2900	15	1000	3.9	0.2	52-160	20	23-178	20
Carbon Disulfide	75-15-0	0.05	7800	720	200000	720	20000	9	32	66-130	20	36-146	20
Carbon tetrachloride	56-23-5	0.05	5	0.3	44	0.64	410	0.9	0.07	70-130	20	50-169	20
Chlorobenzene	108-90-7	0.05	1600	130	41000	210	4100	1.3	1	70-130	20	70-134	20
Chloroethane	75-00-3	0.05	31000	1500	820000	1500	170	1.1	15	38-172	20	10-200	20
Chloroform	67-66-3	0.25	100	0.3	940	0.54	2000	0.76	0.6	70-130	20	63-145	20
Chloromethane	74-87-3	0.05	310	110	8200	170	820	1.1	0.14	68-130	20	33-130	20
cis-1,2-Dichloroethene	156-59-2	0.05	780	1200	20000	1200	20000	1200	0.4	70-130	20	69-132	20
cis-1,3-Dichloropropene	542-75-6	0.05	6.4	1.1	57	2.1	1200	0.39	0.004	70-130	20	60-133	20
Dibromochloromethane	124-48-1	0.05	1600	1300	41000	1300	41000	1300	0.4	70-130	20	49-150	20
Ethylbenzene	100-41-4	0.05	7800	400	200000	400	20000	58	13	70-130	20	62-154	20
Methyl-tert-butyl ether	1634-04-4	0.05	780	8800	20000	8800	2000	140	0.32	70-130	20	70-130	20
Methylene Chloride	75-09-2	0.05	85	13	760	24	12000	34	**0.02**	70-130	20	57-146	20
Styrene	100-42-5	0.05	16000	1500	410000	1500	41000	430	4	66-130	20	63-130	20
Tetrachloroethene	127-18-4	0.05	12	11	110	20	2400	28	0.06	70-130	20	61-152	20
Toluene	108-88-3	0.05	16000	650	410000	650	410000	42	12	70-130	20	64-147	20
trans-1,2-Dichloroethene	156-60-5	0.05	1600	3100	41000	3100	41000	3100	0.7	70-130	20	64-132	20
1,3-Dichloropropane	10061-02-6	0.05	-	-	-	-	-	-	-	70-130	20	70-130	20
Trichloroethene	79-01-6	0.05	58	5	520	8.9	1200	12	0.06	70-130	20	70-130	20
Vinyl Chloride	75-01-4	0.05	0.46	0.28	7.9	1.1	170	1.1	**0.01**	70-130	20	36-130	20
Total Xylene	1330-20-7	0.075	16000	320	410000	320	41000	5.6	150	70-130	20	70-131	20

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B
Bolded number indicates value is less than laboratory reporting limit

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois Soil													
ANALYTE (& EPA METHOD)	CAS	Pace-GB	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
PESTICIDES (8081)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)				
4,4'-DDD	72-54-8	0.00333	3	-	24	-	370	---	16	46-130	20	29-130	21
4,4'-DDE	72-55-9	0.00333	2	-	17	-	370	---	54	48-130	20	14-140	44
4,4'-DDT	50-29-3	0.00333	2	-	17	1500	100	2100	32	46-430	20	29-130	32
Aldrin	309-00-2	0.00167	0.04	3	0.3	6.6	6.1	9.3	0.5	44-130	20	30-130	45
a-BHC	319-84-6	0.00167	0.1	0.8	0.9	1.5	20	2.1	**0.0005**	49-130	20	37-130	27
a-Chlordane	5103-71-9	0.00167	1.8	72	16	140	100	22	10	52-130	20	34-130	41
b-BHC	319-85-7	0.00167	-	-	-	-	---	---	-	49-130	20	17-130	50
d-BHC	319-86-8	0.00167	-	-	-	-	---	---	-	22-135	20	10-137	32
Dieldrin	60-57-1	0.00333	0.04	1	0.4	2.2	7.8	3.1	**0.004**	46-130	20	31-130	30
Endosulfan I	959-98-8	0.00167	470	-	12000	-	1200	---	18	40-130	20	16-130	32
Endosulfan II	33213-65-9	0.00333	470	-	12000	-	1200	---	18	47-130	20	35-130	28
Endosulfan sulfate	1031-07-8	0.00333	470	-	12000	-	1200	---	18	44-130	20	21-130	36
Endrin	72-20-8	0.00333	23	-	610	-	61	---	1	53-130	20	38-130	28
Endrin Aldehyde	7421-93-4	0.00333	23	-	610	-	61	---	1	41-130	20	16-130	45
Endrin ketone	53494-70-5	0.00333	23	-	610	-	61	---	1	48-130	20	16-130	45
g-BHC	58-89-9	0.00167	0.5	-	4	-	96	---	**0.009**	43-130	20	32-130	28
g-Chlordane	5566-34-7	0.00167	1.8	72	16	140	100	22	10	44-130	20	20-130	34
Heptachlor	76-44-8	0.00167	0.1	0.1	1	11	28	16	23	51-130	20	40-130	27
Heptachlor Epoxide	1024-57-3	0.00167	0.07	5	0.6	9.2	2.7	13	0.7	44-130	20	34-130	34
4,4'-Methoxychlor	72-43-5	0.0167	390	-	10000	-	1000	---	160	48-130	20	43-130	36
Toxaphene	8001-35-2	0.1	0.6	89	5.2	170	110	240	31	48-130	20	10-161	50

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B
Bolded number indicates value is less than laboratory reporting limit

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois Soil													
ANALYTE (& EPA METHOD)	CAS	Pace-GB)	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
PCBs (8082)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)				
Aroclor 1016	12674-11-2	0.1	-	-	-	-	-	-	-	60-130	20	46-130	20
Aroclor 1221	11104-28-2	0.1	-	-	-	-	-	-	-	60-130	20	46-130	20
Aroclor 1232	11141-16-5	0.1	-	-	-	-	-	-	-	60-130	20	46-130	20
Aroclor 1242	53469-21-9	0.1	-	-	-	-	-	-	-	60-130	20	46-130	20
Aroclor 1248	12672-29-6	0.1	-	-	-	-	-	-	-	60-130	20	46-130	20
Aroclor 1254	11097-69-1	0.1	-	-	-	-	-	-	-	60-130	20	46-130	20
Aroclor 1260	11096-82-5	0.1	-	-	-	-	-	-	-	60-130	20	46-130	20
PCBs (total)	1336-36-3		1.00	-	1.00	-	-	-	-	-	-	-	-

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois Soil											
ANALYTE (& EPA METHOD)	CAS	STAT	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	MS/MSD % R	MS/MSD % RPD
TOTAL METALS (6020)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/l)		
ALUMINUM	7429-90-5	2	-	-	-	-	410000	870000	3.5	75-125	20
ANTIMONY	7440-36-0	0.2	31		820	-	82	---	0.006	75-125	20
ARSENIC	7440-38-2	0.1	13	750	-	1200	61	25000	0.05	75-125	20
BARIUM	7440-39-3	0.1	5500	690000	140000	910000	14000	870000	2	75-125	20
BERYLLIUM	7440-41-7	0.05	160	1300	4100	2100	410	44000	0.004	75-125	20
CADMIUM	7440-43-9	0.05	78	1800	2000	2800	200	59000	0.005	75-125	20
CALCIUM	7440-70-2	6	-	-	-	-	---	---	---	75-125	20
CHROMIUM	7440-47-3	0.1	230	270	6100	420	4100	690	0.1	75-125	20
COBALT	7440-48-4	0.1	4700	-	120000	-	12000	---	1	75-125	20
COPPER	7440-50-8	0.25	2900	-	82000	-	8200	---	0.65	75-125	20
CYANIDE (9012)	57-12-5	0.25	1600	-	41000	-	4100	---	0.2	75-125	20
HEXAVALENT CHROMIUM (7196A	18540-29-9	0.4	230	270	6100	420	4100	690	---	75-125	20
IRON	7439-89-6	3	-	-	-	-	140000	---	5	75-125	20
LEAD	7439-92-1	0.05	400	-	800	-	700	---	0.0075	75-125	20
MAGNESIUM	7439-95-4	3	325000	-	-	-	730000	---	---	75-125	20
MANGANESE	7439-96-5	0.1	1600	69000	41000	91000	4100	8700	0.15	75-125	20
MERCURY (7471A)	7439-97-6	0.02	23	10	610	16	61	0.1	0.002	75-125	20
NICKEL	7440-02-0	0.1	1600	13000	41000	21000	4100	440000	0.1	75-125	20
POTASSIUM	7440-09-7	3	-	-	-	-	---	---	---	75-125	20
SELENIUM	7782-49-2	0.1	390	-	10000	-	1000	---	0.05	75-125	20
SILVER	7440-22-4	0.1	390	-	10000	-	1000	---	0.05	75-125	20
SODIUM	7440-23-5	6	-	-	-	-	---	---	---	75-125	20
THALLIUM	7440-28-0	0.1	6.3	-	160	-	160	---	0.002	75-125	20
VANADIUM	7440-62-2	0.1	550	-	14000	-	1400	---	0.049	75-125	20
ZINC	7440-66-6	0.5	23000	-	610000	-	61000	---	5	75-125	20

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B
TCLP/SPLP values are used for MIG TO GW.

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois Soil											
ANALYTE (& EPA METHOD)	CAS	STAT	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	MS/MSD % R	MS/MSD % RPD
SEMIVOLATLES (8270C)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)		
1,2-Dichlorobenzene	95-50-1	0.17	7000	560	180000	560	18000	310	17	30-130	50
1,2,4-Trichlorobenzene	120-82-1	0.17	780	3200	20000	3200	2000	920	5	55-106	23
1,3-Dichlorobenzene	541-73-1	0.17	70	570	1800	570	180	570	0.2	30-130	50
1,4-Dichlorobenzene	106-46-7	0.17	-	11000	-	17000	---	340	2	27-95	43
2,2'oxybis (1-Chloropropane)	108-60-1	0.17	3100	1300	82000	1300	8200	1300	2.4	30-130	50
2,4,6-Trichlorophenol	95-95-4	0.17	58	200	520	390	11000	540	0.2	30-130	50
2,4,5-Trichlorophenol	88-06-2	0.33	7800	-	200000	-	200000	---	270	30-130	50
2,4-Dichlorophenol	120-83-2	0.17	230	-	6100	-	610	---	1	30-130	50
2,4-Dimethylphenol	105-67-9	0.17	1600	-	41000	-	41000	---	9	30-130	50
2,4-Dinitrophenol	51-28-5	0.8	160	-	4100	-	410	---	0.2	30-130	50
2,4-Dinitrotoluene	121-14-2	0.17	0.9	-	8.4	-	180	---	0.0008	52-127	37
2,6-Dinitrotoluene	606-20-2	0.17	0.9	-	8.4	-	180	---	0.0007	30-130	50
2-Chloronaphthalene	91-58-7	0.17	6300	-	160000	-	160000	---	49	30-130	50
2-Chlorophenol	95-57-8	0.17	390	53000	10000	-	10000	53000	4	21-102	49
2-Methylnaphthalene	91-57-6	0.17	310	-	8200	-	820	---	7.2	30-130	50
2-Methylphenol (o-Cresol)	95-48-7	0.17	3900	-	100000	-	100000	---	15	30-130	50
2-Nitroaniline	88-74-4	0.8	230	35	6100	56	610	3.6	0.14	30-130	50
2-Nitrophenol	88-75-5	0.17	-	-	-	-	---	---		30-130	50
3-Nitroaniline	99-09-2	0.8	23	250	610	400	61	26	0.01	30-130	50
3,3-Dichlorobenzidine	91-94-1	0.66	1	-	13	-	280	---	0.007	30-130	50
3&4-Methylphenol (m&p-cresol)	106-44-5	0.8	390	-	10000	-	1000	---	0.2	30-130	50
4,6Dinitro-2-methylphenol	534-52-1	0.17	7.8	-	200	-	820	---	-	30-130	50
4-Bromophenylphenyl ether	101-55-3	0.17	-	-	-	-	---	---	-	30-130	50
4-Chloro-3-methlyphenol	59-50-7	0.17	5500	-	14000	-	41000	---	24	28-121	88
4-Chloroaniline	106-47-8	0.17	310	-	8200	-	820	---	0.7	30-130	50
4-Chlorophenylphenyl ether	7005-72-3	0.17	-	-	-	-	---	---	-	30-130	50
4-Nitroaniline	100-01-6	0.8	230	1000	6100	1600	610	110	0.1	30-130	50
4-Nitrophenol	100-02-7	0.8	630	-	16000	-	16000	---	-	10-156	56
Acenaphthene	83-32-9	0.025	4700	-	120000	-	120000	---	570	30-130	50
Acenaphthylene	208-96-8	0.025	2300	-	61000	-	61000	---	85	30-130	50
bis(2-Chloroethoxy)methane	111-91-1	0.17	23000	-	610000	-	---	---	12000	30-130	50
bis(2-Chloroethyl)ether	111-44-4	0.17	0.9	-	8	-	75	0.66	2	30-130	50
bis(2-Ethylhexyl)phthalate	117-81-7	0.33	0.09	-	0.8	-	4100	31000	8	30-130	50
Butylbenzylphthalate	85-68-7	0.17	0.9	-	8	-	410000	930	5	30-130	50
Carbazole	86-74-8	0.17	2300	-	61000	-	6200	---	27000	30-130	50
Dibenzofuran	132-64-9	0.17	9	-	78	-	4100	---	49	30-130	50
Diethylphthalate	84-66-2	0.17	-	-	-	-	1000000	2000	-	30-130	50
Dimethylphthalate	131-11-3	0.17	0.6	0.2	5	0.47	1000000	1300	0.0004	30-130	50
Di-n-butylphthalate	84-74-2	0.17	46	31000	410	31000	200000	2300	3600	30-130	50
Di-noctylphthalate	117-84-0	0.17	16000	930	410000	930	4100	10000	930	30-130	50
Hexachlorobenzene	118-74-1	0.17	32	-	290	-	78	2.6	0.6	30-130	50
Hexachloro-1,3-butadine	87-68-3	0.17	88	-	780	-	41	180	160	30-130	50
Hexachlorocyclopentadiene	77-47-4	0.17	0.09	-	0.8	-	14000	1.1	2	30-130	50
Hexachloroethane	67-72-1	0.17	160	-	4100	-	2000	---	6.1	30-130	50
Isophorone	78-59-1	0.17	63000	2000	1000000	2000	410000	4600	470	30-130	50
Nitrobenzene	98-95-3	0.17	780000	1300	1000000	1300	1000	9.4	380	30-130	50
N-Nitroso-di-n-propylamine	621-64-7	0.33	7800	2300	200000	2300	18	---	2300	30-130	50
N-Nitrosodiphenylamine	86-30-6	0.17	1600	10000	41000	10000	4100	1.8	10000	30-130	50
Pentachlorophenol	87-86-5	0.8	3100	-	82000	-	520	---	4300	10-204	47
Phenol	108-95-2	0.17	3100	-	82000	-	61000	---	560	20-103	66

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois Soil											
ANALYTE (& EPA METHOD)	CAS	STAT	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	MS/MSD % R	MS/MSD % RPD
SEMIVOLATLES (8270C SIM)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)		
Acenaphthene	83-32-9	0.025	4700	-	120000	-	120000	—	570	30-130	50
Acenaphthylene	208-96-8	0.025	2300	-	61000	-	61000	—	85	30-130	50
Anthracene	120-12-7	0.025	23000	-	610000	-	610000	—	12000	30-130	50
Benzo(a)anthracene	56-55-3	0.025	0.9	-	8	-	170	—	2	30-130	50
Benzo(a)pyrene	50-32-8	0.025	0.09	-	0.8	-	17	—	8	30-130	50
Benzo(b)fluoranthene	205-99-2	0.025	0.9	-	8	-	170	—	5	30-130	50
Benzo(ghi)perylene	191-24-2	0.025	2300	-	61000	-	61000	—	32000	30-130	50
Benzo(k)fluoranthene	207-08-9	0.025	9	-	78	-	1700	—	49	30-130	50
Chrysene	218-01-9	0.025	88	-	780	-	17000	—	160	30-130	50
Dibenzo(a,h)anthracene	53-70-3	0.025	0.09	-	0.8	-	17	—	2	30-130	50
Fluoranthene	2063-44-0	0.025	3100	-	82000	-	82000	—	4300	30-130	50
Fluorene	86-73-7	0.025	3100	-	82000	-	82000	—	560	30-130	50
Indeno(1,2,3-cd)pyrene	193-39-5	0.025	0.9	-	8	-	170	—	14	30-130	50
Naphthalene	91-20-3	0.025	1600	170	41000	270	4100	1.8	12	30-130	50
Phenanthrene	85-01-8	0.025	2300	-	61000	-	61000	—	200	30-130	50
Pyrene	129-00-0	0.025	2300	-	61000	-	61000	—	4200	30-130	50

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois Soil											
ANALYTE (& EPA METHOD)	CAS	STAT	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	MS/MSD % R	MS/MSD % RPD
VOLATILES (8260B)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)		
1,1,1-Trichloroethane	71-55-6	0.005	-	1200	-	1200	—	1200	2	70-130	25
1,1,2,2-Tetrachloroethane	79-34-5	0.005	4700	2000	120000	2000	12000	2000	3.3	70-130	25
1,1,2-Trichloroethane	79-00-5	0.005	310	1800	8200	1800	8200	1800	0.02	70-130	25
1,1-Dichloroethane	75-34-3	0.005	7800	1300	200000	1700	200000	130	23	70-130	25
1,1-Dichloroethene	75-35-4	0.005	3900	290	100000	470	10000	3	0.06	70-130	25
1,2-Dichloroethane	107-06-2	0.005	7	0.4	63	0.7	1400	0.99	0.02	70-130	25
1,2-Dichloropropane	78-87-5	0.005	9	15	84	23	1800	0.5	0.03	70-130	25
2-Butanone (MEK)	78-93-3	0.01	47000	25000	1000000	25000	120000	710	17	70-130	25
2-Hexanone	591-78-6	0.01	3100	70	82000	110	8200	0.72	1.3	70-130	25
4-Methyl-2-pentanone (MIBK)	108-10-1	0.01	-	3100	-	3100	—	340	-	70-130	25
Acetone	67-64-1	0.05	70000	100000	-	100000	—	100000	25	70-130	25
Benzene	71-43-2	0.005	12	0.8	100	1.6	2300	2.2	0.03	70-130	25
Bromodichloromethane	75-27-4	0.005	10	3000	92	3000	2000	3000	0.6	70-130	25
Bromoform	75-25-2	0.005	81	53	720	100	16000	140	0.8	70-130	25
Bromomethane	74-83-9	0.01	110	10	2900	15	1000	3.9	0.2	70-130	25
Carbon Disulfide	75-15-0	0.005	7800	720	200000	720	20000	9	32	70-130	25
Carbon tetrachloride	56-23-5	0.005	5	0.3	44	0.64	410	0.9	0.07	70-130	25
Chlorobenzene	108-90-7	0.005	1600	130	41000	210	4100	1.3	1	70-130	25
Chloroethane	75-00-3	0.005	31000	1500	820000	1500	170	1.1	15	70-130	25
Chloroform	67-66-3	0.005	100	0.3	940	0.54	2000	0.76	0.6	70-130	25
Chloromethane	74-87-3	0.01	310	110	8200	170	820	1.1	0.14	70-130	25
cis-1,2-Dichloroethene	156-59-2	0.005	780	1200	20000	1200	20000	1200	0.4	70-130	25
cis-1,3-Dichloropropene	10061-01-5	0.002	6.4	1.1	57	2.1	1200	0.39	0.004	70-130	25
Dibromochloromethane	124-48-1	0.005	1600	1300	41000	1300	41000	1300	0.4	70-130	25
Ethylbenzene	100-41-4	0.005	7800	400	200000	400	20000	58	13	70-130	25
Methyl-tert-butyl ether	1634-04-4	0.005	780	8800	20000	8800	2000	140	0.32	70-130	25
Methylene Chloride	75-09-2	0.01	85	13	760	24	12000	34	0.02	70-130	25
Styrene	100-42-5	0.005	16000	1500	410000	1500	41000	430	4	70-130	25
Tetrachloroethene	127-18-4	0.005	12	11	110	20	2400	28	0.06	70-130	25
Toluene	108-88-3	0.005	16000	650	410000	650	410000	42	12	70-130	25
trans-1,2-Dichloroethene	156-60-5	0.005	1600	3100	41000	3100	41000	3100	0.7	70-130	25
1,3-Dichloropropane	10061-02-6	0.005	-	-	-	-	-	-	-	70-130	25
Trichloroethene	79-01-6	0.005	58	5	520	8.9	1200	12	0.06	70-130	25
Vinyl Chloride	75-01-4	0.005	0.46	0.28	7.9	1.1	170	1.1	0.01	70-130	25
Total Xylene	1330-20-7	0.015	16000	320	410000	320	41000	5.6	150	70-130	25

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois Soil											
ANALYTE (& EPA METHOD)	CAS	STAT	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	MS/MSD % R	MS/MSD % RPD
PESTICIDES (8081)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)		
4,4'-DDD	72-54-8	0.0016	3	-	24	-	370	—	16	30-150	25
4,4'-DDE	72-55-9	0.0016	2	-	17	-	370	—	54	30-150	25
4,4'-DDT	50-29-3	0.0016	2	-	17	1500	100	2100	32	30-150	25
Aldrin	309-00-2	0.0016	0.04	3	0.3	6.6	6.1	9.3	0.5	30-150	25
a-BHC	319-84-6	0.0016	0.1	0.8	0.9	1.5	20	2.1	**0.0005**	30-150	25
a-Chlordane	5103-71-9	0.0016	1.8	72	16	140	100	22	10	30-150	25
b-BHC	319-85-7	0.0016	-	-	-	-	—	—	-	30-150	25
d-BHC	319-86-8	0.0016	-	-	-	-	---	---	-	30-150	25
Dieldrin	60-57-1	0.0016	0.04	1	0.4	2.2	7.8	3.1	0.004	30-150	25
Endosulfan I	959-98-8	0.0016	470	-	12000	-	1200	---	18	30-150	25
Endosulfan II	33213-65-9	0.0016	470	-	12000	-	1200	---	18	30-150	25
Endosulfan sulfate	1031-07-8	0.0016	470	-	12000	-	1200	---	18	30-150	25
Endrin	72-20-8	0.0016	23	-	610	-	61	---	1	30-150	25
Endrin Aldehyde	7421-93-4	0.0016	23	-	610	-	61	---	1	30-150	25
Endrin ketone	53494-70-5	0.0016	23	-	610	-	61	---	1	30-150	25
g-BHC	58-89-9	0.0016	0.5	-	4	-	96	---	0.009	30-150	25
g-Chlordane	5566-34-7	0.0016	1.8	72	16	140	100	22	10	30-150	25
Heptachlor	76-44-8	0.0016	0.1	0.1	1	11	28	16	23	30-150	25
Heptachlor Epoxide	1024-57-3	0.0016	0.07	5	0.6	9.2	2.7	13	0.7	30-150	25
4,4'-Methoxychlor	72-43-5	0.0016	390	-	10000	-	1000	---	160	30-150	25
Toxaphene	8001-35-2	0.033	0.6	89	5.2	170	110	240	31	30-150	25

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B
Bolded number indicates value is less than laboratory reporting limit

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
SOIL METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois Soil											
ANALYTE (& EPA METHOD)	CAS	STAT	TACO RES	TACO RES	TACO IND/COM	TACO IND/COM	TACO CONST. WORKER	TACO CONST. WORKER	TACO SOIL	MS/MSD % R	MS/MSD % RPD
PCBs (8082)		RL (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	Ingest. (mg/kg)	Inhal. (mg/kg)	MIG TO GW (mg/kg)		
Aroclor 1016	12674-11-2	0.08	-	-	-	-	-	-	-	30-150	25
Aroclor 1221	11104-28-2	0.08	-	-	-	-	-	-	-		
Aroclor 1232	11141-16-5	0.08	-	-	-	-	-	-	-		
Aroclor 1242	53469-21-9	0.08	-	-	-	-	-	-	-		
Aroclor 1248	12672-29-6	0.08	-	-	-	-	-	-	-		
Aroclor 1254	11097-69-1	0.08	-	-	-	-	-	-	-		
Aroclor 1260	11096-82-5	0.08	-	-	-	-	-	-	-	30-150	25
PCBs (total)	1336-36-3		1	-	1	-	-	-	-		

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER								
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
METALS (6010)		mg/L	mg/L	Cleanup Critiria mg/L				
ALUMINUM	7429-90-5	0.0061	0.5	3.5	80-120	20	75-125	20
ANTIMONY	7440-36-0	0.00145	0.02	0.006	80-120	20	75-125	20
ARSENIC	7440-38-2	0.00055	0.02	0.05	80-120	20	75-125	20
BARIUM	7440-39-3	0.00027	0.005	2	80-120	20	75-125	20
BERYLLIUM	7440-41-7	0.00013	0.004	0.004	80-120	20	75-125	20
CADMIUM	7440-43-9	0.00026	0.005	0.005	80-120	20	75-125	20
CALCIUM	7440-70-2	0.01912	0.2	-	80-120	20	75-125	20
CHROMIUM	7440-47-3	0.00044	0.005	0.1	80-120	20	75-125	20
COBALT	7440-48-4	0.00049	0.005	1	80-120	20	75-125	20
COPPER	7440-50-8	0.00027	0.01	0.65	80-120	20	75-125	20
IRON	7439-89-6	0.00426	0.1	5	80-120	20	75-125	20
LEAD	7439-92-1	0.00139	0.0075	0.0075	80-120	20	75-125	20
MAGNESIUM	7439-95-4	0.0154	0.2	-	80-120	20	75-125	20
MANGANESE	7439-96-5	0.00059	0.005	0.15	80-120	20	75-125	20
MERCURY (7470A)	7439-97-6	0.0001	0.2	0.002	85-115	20	85-115	20
NICKEL	7440-02-0	0.00043	0.01	0.1	80-120	20	75-125	20
POTASSIUM	9/7/7440	0.02335	0.2	-	80-120	20	75-125	20
SELENIUM	7782-49-2	0.00211	0.02	0.05	80-120	20	75-125	20
SILVER	7440-22-4	0.00046	0.01	0.05	80-120	20	75-125	20
SODIUM	7440-23-5	0.01249	1		80-120	20	75-125	20
THALLIUM	7440-28-0	0.0022	0.04	0.002	80-120	20	75-125	20
VANADIUM	7440-62-2	0.00049	0.005	0.049	80-120	20	75-125	20
ZINC	7440-66-6	0.00181	0.04	5	80-120	20	75-125	20
CYANIDE (9012)	57-12-5	6.12	2	0.2	90-110	20	90-110	20
HEXAVALENT CHROMIUM (SM3500CR-B)	18540-29-9	3.4	2		90-110	20	90-110	20

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER								
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
SEMIVOLATIES (8270)		mg/L	mg/L	Cleanup Critiria mg/L				
1,2-Dichlorobenzene	95-50-1	0.00071	0.005	0.6	51-130	24	42-130	26
1,2,4-Trichlorobenzene	120-82-1	0.00087	0.005	0.07	58-130	21	50-130	21
1,3-Dichlorobenzene	541-73-1	0.00083	0.005	0.0063	45-130	26	39-130	29
1,4-Dichlorobenzene	106-46-7	0.00086	0.005	0.075	46-130	26	42-130	29
2,2'-oxybis(1-Chloropropane)	108-60-1	0.00082	0.005	0.28	44-130	20	43-135	20
2,4,5-Trichlorophenol	95-95-4	0.001	0.005	0.7	70-130	20	62-130	20
2,4,6-Trichlorophenol	88-06-2	0.00107	0.005	0.01	70-130	20	58-130	20
2,4-Dichlorophenol	120-83-2	0.00115	0.005	0.021	63-130	20	50-130	20
2,4-Dimethylphenol	105-67-9	0.00113	0.005	0.14	14-130	20	10-151	32
2,4-Dinitrophenol	51-28-5	0.00206	0.01	0.014	45-130	20	45-133	20
2,4-Dinitrotoluene	121-14-2	0.0008	0.005	0.00002	70-130	20	56-138	20
2,6-Dinirotoluene	606-20-2	0.00107	0.005	0.00031	70-130	20	51-137	20
2-Chloronaphthalene	91-58-7	0.00084	0.005	0.56	70-130	20	57-130	20
2-Chlorophenol	95-57-8	0.0007	0.005	0.035	54-130	20	40-130	20
2-Methylnaphthalene	91-57-6	0.00135	0.005	0.028	70-130	20	58-130	20
2-Methylphenol (o-Cresol)	95-48-7	0.00097	0.005	0.35	45-130	20	29-130	20
2-Nitroaniline	88-74-4	0.00084	0.005	0.021	63-130	20	29-153	20
2-Nitrophenol	88-75-5	0.00136	0.005	-	65-130	20	46-130	20
3-Nitroaniline	99-09-2	0.00097	0.005	0.0021	69-130	20	24-131	20
3,3-Dichlorobenzidine	91-94-1	0.00111	0.005	0.02	39-138	21	10-138	44
3&4-Methylphenol (m&p-cresol)	106-44-5	0.00077	0.005	0.035	40-130	20	29-130	21
4,6Dinitro-2-methylphenol	534-52-1	0.00075	0.005	0.0007	56-130	20	52-130	20
4-Bromophenylphenyl ether	101-55-3	0.0013	0.005	-	63-130	20	62-130	20
4-Chloro-3-methlyphenol	59-50-7	0.00101	0.005	0.49	64-130	20	42-130	20
4-Chloroaniline	106-47-8	0.00081	0.005	0.028	48-130	23	29-130	28
4-Chlorophenylphenyl ether	7005-72-3	0.00119	0.005	-	70-130	20	69-130	20
4-Nitroaniline	100-01-6	0.0011	0.005	0.021	53-140	21	21-140	28
4-Nitroaniline	100-02-7	0.00087	0.005	-	53-140	21	21-140	28
bis(2-Chloroethoxy)methane	111-91-1	0.00119	0.005	-	70-130	20	68-130	20
bis(2-Chloroethyl)ether	111-44-4	0.00066	0.005	0.01	54-130	20	48-130	20
bis(2-Ethylhexyl)phthalate	117-81-7	0.0026	0.005	0.006	70-137	20	34-160	20
Butylbenzylphthalate	85-68-7	0.00109	0.005	1.4	66-143	20	37-158	20
Carbazole	86-74-8	0.00069	0.005	-	70-130	20	70-130	20
Dibenzofuran	132-64-9	0.00106	0.005	0.014	70-130	20	70-130	20
Diethylphthalate	84-66-2	0.00135	0.005	5.6	70-130	20	50-130	20
Dimetlphthalate	131-11-3	0.00104	0.005	70	70-130	20	70-130	20
Di-n-butylphthalate	84-74-2	0.0009	0.005	0.7	70-130	20	63-130	20
Di-n-octylphthalate	117-84-0	0.00153	0.005	0.14	70-135	20	51-150	20
Hexachlorobenzene	118-74-1	0.00111	0.005	0.00006	66-130	20	58-130	20
Hexachloro-1,3-butadine	87-68-3	0.00066	0.01	0.0014	47-130	21	39-130	26
Hexachlorocyclopentadiene	77-47-4	0.0011	0.005	0.05	10-130	29	10-130	50
Hexachloroethane	67-72-1	0.00058	0.005	0.007	39-130	30	24-130	45
Isophorone	78-59-1	0.00137	0.005	1.4	24-130	20	23-130	20
Naphthalene	91-20-3	0.0007	0.005	0.14	66-130	20	48-130	20
Nitrobenzene	98-95-3	0.00137	0.005	0.0035	62-130	20	52-130	20
N-Nitroso-di-n-propylamine	621-64-7	0.00106	0.005	0.0018	56-130	20	44-134	20
N-Nitrosodiphenylamine	86-30-6	0.00245	0.01	0.0032	35-130	20	24-130	20
Pentachlorophenol	87-86-5	0.00108	0.005	0.001	44-130	20	44-130	20
Phenol	108-95-2	0.00103	0.005	0.1	23-130	23	19-130	23

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER								
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
LOW LEVEL PAHs (8270C SIM)		mg/L	mg/L	Cleanup Critiria mg/L				
Acenaphthylene	83-32-9	4.8E-06	0.00005	0.21	43-130	46	25-130	41
Acenaphthene	208-96-8	3.82E-06	0.00005	0.42	33-130	47	22-130	40
Anthracene	120-12-7	6.08E-06	0.00005	2.1	33-130	50	22-130	36
Benzo(a)anthracene	56-55-3	3.84E-06	0.00005	0.00013	41-130	20	52-130	20
Benzo(a)pyrene	50-32-8	3.03E-06	0.00005	0.0002	59-130	20	52-130	20
Benzo(b)fluoranthene	2052-99-2	0.000003	0.00005	0.00018	53-130	20	51-130	20
Benzo(k)fluoranthene	207-08-9	4.63E-06	0.00005	0.21	64-133	20	55-130	22
Benzo(ghi)perylene	191-24-2	3.6E-06	0.00005	0.00017	55-130	20	46-130	20
Chrysene	218-01-9	3.69E-06	0.00005	0.0015	62-130	20	49-130	20
Dibenzo(a,h)anthracene	53-70-3	3.39E-06	0.00005	0.0003	37-130	20	43-130	20
Fluoranthene	206-44-0	4.67E-06	0.00005	0.28	48-130	37	41-130	28
Fluorene	86-73-7	5.06E-06	0.00005	0.28	42-130	48	21-130	32
Indeno(1,2,3-cd)pyrene	193-39-5	4.96E-06	0.00005	0.00043	46-130	20	42-130	20
Naphthalene	91-20-3	5.14E-06	0.00005	0.14	33-130	53	19-130	42
Phenanthrene	85-01-8	8.58E-06	0.00005	0.21	36-130	47	22-130	38
Pyrene	129-00-0	5.03E-06	0.00005	0.21	51-130	33	35-130	21

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER								
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
VOLATILES (8260)		mg/L	mg/L	Cleanup Critiria mg/L				
1,1,1-Trichloroethane	71-55-6	0.0009	0.001	0.2	70-132	20	70-132	20
1,1,2,2-Tetrachloroethane	79-34-5	0.00092	0.001	0.42	63-130	20	61-130	20
1,1,2-Trichloroethane	79-00-5	0.00042	0.001	0.005	70-130	20	70-130	20
1,1-Dichloroethane	75-34-3	0.00075	0.001	0.7	70-132	20	70-132	20
1,1-Dichloroethene	75-35-4	0.00057	0.001	0.007	70-137	20	70-137	20
1,2-Dichlorobenzene	95-50-1	0.00083	0.001	0.6	70-130	20	70-130	20
1,2-Dichloroethane	107-06-2	0.00036	0.001	0.005	70-130	20	70-130	20
2-Butanone (MEK)	78-93-3	0.0043	0.02	4.2	50-150	20	50-150	20
2-Hexanone	591-78-6	0.00197	0.005	0.28	50-150	20	50-150	20
4-Methyl-2-pentanone (MIBK)	108-10-1	0.0012	0.005	-	50-150	20	50-150	20
Acetone	67-64-1	0.0033	0.02	6.3	50-150	20	50-150	20
Benzene	71-43-2	0.00041	0.001	0.005	70-130	20	70-130	20
Bromodichloromethane	75-27-4	0.00056	0.001	0.0002	70-131	20	70-131	20
Bromoform	75-25-2	0.00094	0.001	0.001	70-130	20	68-130	20
Bromomethane	74-83-9	0.00091	0.001	0.0098	53-160	20	47-177	20
Carbon Disulfide	75-15-0	0.00066	0.001	0.7	70-130	20	60-130	29
Carbon tetrachloride	56-23-5	0.00049	0.001	0.005	70-130	20	70-149	20
Chlorobenzene	108-90-7	0.00041	0.001	0.1	70-130	20	70-130	20
Chloroethane	75-00-3	0.00097	0.001	2.8	70-147	20	66-147	20
Chloroform	67-66-3	0.0013	0.005	0.0002	70-130	20	70-130	20
Chloromethane	74-87-3	0.00024	0.001	0.028	41-137	20	41-137	20
cis-1,2-Dichloroethene	156-59-2	0.00083	0.001	0.07	70-130	20	70-130	20
cis-1,3-Dichloropropene	10061-01-5	0.0002	0.001	0.001	70-130	20	70-130	20
Dibromochloromethane	124-48-1	0.00081	0.001	0.14	70-130	20	70-130	20
Ethylbenzene	100-41-4	0.00054	0.001	0.7	70-130	20	70-130	20
Methyl-tert-butyl ether	1634-04-4	0.00061	0.001	0.07	70-130	20	70-130	20
Methylene Chloride	75-09-2	0.00043	0.001	0.005	70-130	20	70-130	20
Styrene	100-42-5	0.00086	0.001	0.1	70-130	20	13-149	20
Tetrachloroethene	127-18-4	0.00045	0.001	0.005	70-130	20	70-130	20
Toluene	108-88-3	0.00067	0.001	1	70-130	20	70-130	20
trans-1,2-Dichloroethene	156-60-5	0.00089	0.001	0.1	70-130	20	70-130	20
trans-1,3-Dichloropropene	10061-02-6	0.00019	0.001	0.001	70-130	20	70-130	20
Trichloroethene	79-01-6	0.00048	0.001	0.005	70-130	20	70-130	20
Vinyl Chloride	75-01-4	0.00018	0.001	0.002	47-131	20	46-131	20
Total Xylene	1330-20-7	0.0026	0.003	10	70-130	20	70-130	20

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER								
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
PESTICIDES (8081)		mg/L	mg/L	Cleanup Critiria mg/L				
4,4'-DDD	72-54-8	0.000023	0.0001	0.014	67-130	20	67-130	20
4,4'-DDE	72-55-9	0.000023	0.0001	0.01	56-133	20	56-133	20
4,4'-DDT	50-29-3	0.000026	0.0001	0.006	53-130	20	53-130	20
Aldrin	309-00-2	0.000012	0.00005	0.014	53-130	20	53-130	20
a-BHC	319-84-6	6.2E-06	0.00005	0.00011	66-130	20	66-130	20
a-Chlordane	5103-71-9	0.00001	0.00005	0.002	70-130	20	70-130	20
b-BHC	319-85-7	0.000013	0.00005	-	70-130	20	70-130	20
d-BHC	319-86-8	9.3E-06	0.00005	-	31-152	20	31-152	20
Dieldrin	60-57-1	0.000018	0.0001	0.009	70-130	20	10-171	20
Endosulfan I	959-98-8	0.000011	0.0001	0.042	58-130	20	55-130	20
Endosulfan II	33213-65-9	0.000023	0.0001	0.042	70-130	20	70-130	20
Endosulfan sulfate	1031-07-8	0.000017	0.0001	0.042	67-130	20	67-130	20
Endrin	72-20-8	0.000024	0.0001	0.002	66-130	20	66-132	20
Endrin Aldehyde	7421-93-4	0.000019	0.0001	0.002	42-132	20	42-132	20
Ednrin Ketone	53494-70-5	0.000016	0.0001	0.002	70-130	20	70-130	20
g-BHC (Lindane)	58-89-9	0.000008	0.0005	0.0002	62-130	20	43-141	20
g-Chlordane	5566-34-7	0.000013	0.00005	0.002	65-130	20	10-167	20
Heptachlor	76-44-8	9.1E-06	0.00005	0.0004	61-130	20	50-130	20
Heptachlor Epoxide	1024-57-3	8.4E-06	0.00005	0.0002	69-130	20	68-130	20
4,4'-Methoxychlor	72-43-5	0.000089	0.0005	0.04	59-130	20	42-133	20
Toxaphene	8001-35-3	0.00049	0.003	0.003	70-130	20	70-130	20

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
PACE ANALYTICAL
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER								
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	LCS/LCSD % R	LCS/LCSD % RPD	MS/MSD % R	MS/MSD % RPD
PCBs EPA (8082)		mg/L	mg/L	Cleanup Critiria mg/L				
Aroclor 1016	12674-11-2	0.0003	0.001	-	65-130	20	52-130	20
Aroclor 1221	11104-28-2	0.0003	0.001	-	65-130	20	52-130	20
Aroclor 1232	11141-16-5	0.0003	0.001	-	65-130	20	52-130	20
Aroclor 1242	53469-21-9	0.0003	0.001	-	65-130	20	52-130	20
Aroclor 1248	12672-29-6	0.0003	0.001	-	65-130	20	52-130	20
Aroclor 1254	11097-69-1	0.0003	0.001	-	65-130	20	52-130	20
Aroclor 1260	11096-82-5	0.0003	0.001	-	65-130	20	52-130	20
PCBs (total)	1336-36-3			0.0005				

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER						
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	MS/MSD % R	MS/MSD % RPD
METALS (6020)		mg/L	mg/L	Cleanup Critiria mg/L		
ALUMINUM	7429-90-5	0.015	0.02	3.5	75-125	20
ANTIMONY	7440-36-0	0.0019	0.003	0.006	75-125	20
ARSENIC	7440-38-2	0.00079	0.002	0.05	75-125	20
BARIUM	7440-39-3	0.00044	0.002	2	75-125	20
BERYLLIUM	7440-41-7	0.00026	0.01	0.004	75-125	20
CADMIUM	7440-43-9	0.00027	0.001	0.005	75-125	20
CALCIUM	7440-70-2	0.044	0.1	-	75-125	20
CHROMIUM	7440-47-3	0.00047	0.002	0.1	75-125	20
COBALT	7440-48-4	0.00008	0.002	1	75-125	20
COPPER	7440-50-8	0.0007	0.005	0.65	75-125	20
IRON	7439-89-6	0.00926	0.05	5	75-125	20
LEAD	7439-92-1	0.00035	0.01	0.0075	75-125	20
MAGNESIUM	7439-95-4	0.00539	0.05	-	75-125	20
MANGANESE	7439-96-5	0.00028	0.002	0.15	75-125	20
MERCURY	7439-97-6	1.1E-05	0.0002	0.002	75-125	20
NICKEL	7440-02-0	0.00019	0.002	0.1	75-125	20
POTASSIUM	2023695	0.023	0.05	-	75-125	20
SELENIUM	2023695	0.0011	0.002	0.05	75-125	20
SILVER	7440-22-4	0.00018	0.002	0.05	75-125	20
SODIUM	7440-23-5	0.046	0.15	-	75-125	20
THALLIUM	7440-28-0	0.00017	0.001	0.002	75-125	20
VANADIUM	7440-62-2	0.00062	0.002	0.049	75-125	20
ZINC	7440-66-6	0.00217	0.01	5	75-125	20
CYANIDE (9012)	57-12-5	0.0035	0.005	0.2	75-125	20
HEXAVALENT CHROMIUM (7196A)	18540-29-9	0.002	0.01		85-115	20

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TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER						
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	MS/MSD % R	MS/MSD % RPD
SEMIVOLATIES (8270C)		mg/L	mg/L	Cleanup Critiria mg/L		
1,2-Dichlorobenzene	95-50-1	0.0012	0.005	0.6	50-125	25
1,2,4-Trichlorobenzene	120-82-1	0.0013	0.005	0.07	48-90	25
1,3-Dichlorobenzene	541-73-1	0.0013	0.005	0.0063	50-125	25
1,4-Dichlorobenzene	106-46-7	0.0012	0.005	0.075	44-85	24
2,2'-oxybis(1-Chloropropane)	108-60-1	0.0012	0.005	0.28	50-125	25
2,4,5-Trichlorophenol	95-95-4	0.0018	0.01	0.7	50-125	25
2,4,6-Trichlorophenol	88-06-2	0.0012	0.005	0.01	50-125	25
2,4-Dichlorophenol	120-83-2	0.0017	0.005	0.021	50-125	25
2,4-Dimethylphenol	105-67-9	0.0013	0.005	0.14	50-125	25
2,4-Dinitrophenol	51-28-5	0.0007	0.025	0.014	66-106	21
2,4-Dinitrotoluene	121-14-2	0.00003	0.0001	0.00002	50-125	25
2,6-Dinirotoluene	606-20-2	0.00002	0.0001	0.00031	50-125	25
2-Chloronaphthalene	91-58-7	0.001	0.005	0.56	50-125	25
2-Chlorophenol	95-57-8	0.001	0.005	0.035	41-95	37
2-Methylnaphthalene	91-57-6	0.00003	0.0001	0.028	50-125	25
2-Methylphenol (o-Cresol)	95-48-7	0.0013	0.005	0.35	50-125	25
2-Nitroaniline	88-74-4	0.0018	0.005	0.021	50-125	25
2-Nitrophenol	88-75-5	0.001	0.005	-	50-125	25
3-Nitroaniline	99-09-2	0.0023	0.025	0.0021	50-125	25
3,3-Dichlorobenzidine	91-94-1	0.0021	0.01	0.02	50-125	25
3&4-Methylphenol (m&p-cresol)	106-44-5	0.0019	0.005	0.035	50-125	25
4,6Dinitro-2-methylphenol	534-52-1	0.0008	0.025	0.0007	50-125	25
4-Bromophenylphenyl ether	101-55-3	0.0011	0.005	-	50-125	25
4-Chloro-3-methlyphenol	59-50-7	0.0017	0.005	0.49	64-104	22
4-Chloroaniline	106-47-8	0.0008	0.005	0.028	50-125	25
4-Chlorophenylphenyl ether	7005-72-3	0.0007	0.005	-	50-125	25
4-Nitroaniline	100-01-6	0.0019	0.025	0.021	50-125	25
4-Nitrophenol	100-02-7	0.0019	0.025	-	22-77	31
bis(2-Chloroethoxy)methane	111-91-1	0.0011	0.005	-	50-125	25
bis(2-Chloroethyl)ether	111-44-4	0.0013	0.005	0.01	50-125	25
bis(2-Ethylhexyl)phthalate	117-81-7	0.00021	0.005	0.006	50-125	25
Butylbenzylphthalate	85-68-7	0.0022	0.005	1.4	50-125	25
Carbazole	86-74-8	0.00002	0.0001	-	50-125	25
Dibenzofuran	132-64-9	0.00003	0.0002	0.014	50-125	25
Diethylphthalate	84-66-2	0.0008	0.005	5.6	50-125	25
Dimetlphthalate	131-11-3	0.0017	0.005	70	50-125	25
Di-n-butylphthalate	84-74-2	0.0011	0.005	0.7	50-125	25
Di-n-octylphthalate	117-84-0	0.0015	0.005	0.14	50-125	25
Hexachlorobenzene	118-74-1	0.0012	0.005	0.00006	50-125	25
Hexachloro-1,3-butadine	87-68-3	0.0009	0.005	0.0014	50-125	25
Hexachlorocyclopentadiene	77-47-4	0.0004	0.005	0.05	50-125	25
Hexachloroethane	67-72-1	0.0014	0.005	0.007	50-125	25
Isophorone	78-59-1	0.0014	0.005	1.4	50-125	25
Naphthalene	91-20-3	0.00011	0.0002	0.14	50-125	25
Nitrobenzene	98-95-3	0.00003	0.0002	0.0035	50-125	25
N-Nitroso-di-n-propylamine	621-64-7	0.00009	0.0001	0.0018	47-93	29
N-Nitrosodiphenylamine	86-30-6	0.0009	0.005	0.0032	50-125	25
Pentachlorophenol	87-86-5	0.0018	0.025	0.001	50-151	20
Phenol	108-95-2	0.0008	0.005	0.1	27-52	33

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TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER

ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	MS/MSD % R	MS/MSD % RPD
LOW LEVEL PAHs EPA METHOD 8270C (SIM)		mg/L	mg/L	Cleanup Critiria mg/L		
Acenaphthylene	83-32-9	0.00003	0.0002	0.21	50-125	25
Acenaphthene	208-96-8	0.00005	0.0002	0.42	50-125	25
Anthracene	120-12-7	0.00002	0.0002	2.1	50-125	25
Benzo(a)anthracene	56-55-3	0.00002	0.00013	0.00013	50-125	25
Benzo(a)pyrene	50-32-8	0.00002	0.0002	0.0002	50-125	25
Benzo(b)fluoranthene	2052-99-2	0.00006	0.00018	0.00018	50-125	25
Benzo(k)fluoranthene	207-08-9	0.00008	0.00017	0.21	50-125	25
Benzo(ghi)perylene	191-24-2	0.00002	0.0001	0.00017	50-125	25
Chrysene	218-01-9	0.00002	0.0001	0.0015	50-125	25
Dibenzo(a,h)anthracene	53-70-3	0.00002	0.0001	0.0003	50-125	25
Fluoranthene	206-44-0	0.00002	0.0002	0.28	50-125	25
Fluorene	86-73-7	0.00003	0.0001	0.28	50-125	25
Indeno(1,2,3-cd)pyrene	193-39-5	0.00002	0.0002	0.00043	50-125	25
Naphthalene	91-20-3	0.00011	0.0002	0.14	50-125	25
Phenanthrene	85-01-8	0.00004	0.0002	0.21	50-125	25
Pyrene	129-00-0	0.00002	0.0002	0.21	50-125	25

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TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER

ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	MS/MSD % R	MS/MSD % RPD
VOLATILES (8260B)		mg/L	mg/L	Cleanup Critiria mg/L		
1,1,1-Trichloroethane	71-55-6	0.0002	0.005	0.2	70-130	15
1,1,2,2-Tetrachloroethane	79-34-5	0.0001	0.005	0.42	70-130	15
1,1,2-Trichloroethane	79-00-5	0.0001	0.005	0.005	70-130	15
1,1-Dichloroethane	75-34-3	0.0002	0.005	0.7	70-130	15
1,1-Dichloroethene	75-35-4	0.0004	0.005	0.007	70-130	15
1,2-Dichlorobenzene	95-50-1	0.0003	0.005	0.6	70-130	15
1,2-Dichloroethane	107-06-2	0.0002	0.005	0.005	70-130	15
2-Butanone (MEK)	78-93-3	0.0016	0.01	4.2	70-130	15
2-Hexanone	591-78-6	0.0002	0.01	0.28	70-130	15
4-Methyl-2-pentanone (MIBK)	108-10-1	0.0007	0.01	-	70-130	15
Acetone	67-64-1	0.0031	0.01	6.3	70-130	15
Benzene	71-43-2	0.0002	0.005	0.005	70-130	15
Bromodichloromethane	75-27-4	0.0002	0.005	0.0002	70-130	15
Bromoform	75-25-2	0.0003	0.005	0.001	70-130	15
Bromomethane	74-83-9	0.002	0.01	0.0098	70-130	15
Carbon Disulfide	75-15-0	0.0003	0.005	0.7	70-130	15
Carbon tetrachloride	56-23-5	0.001	0.005	0.005	70-130	15
Chlorobenzene	108-90-7	0.0002	0.005	0.1	70-130	15
Chloroethane	75-00-3	0.0005	0.01	2.8	70-130	15
Chloroform	67-66-3	0.0003	0.005	0.0002	70-130	15
Chloromethane	74-87-3	0.0003	0.01	0.028	70-130	15
cis-1,2-Dichloroethene	156-59-2	0.0002	0.005	0.07	70-130	15
cis-1,3-Dichloropropene	10061-01-5	0.0002	0.001	0.001	70-130	15
Dibromochloromethane	124-48-1	0.0002	0.005	0.14	70-130	15
Ethylbenzene	100-41-4	0.0003	0.005	0.7	70-130	15
Methyl-tert-butyl ether	1634-04-4	0.0003	0.005	0.07	70-130	15
Methylene Chloride	75-09-2	0.0003	0.005	0.005	70-130	15
Styrene	100-42-5	0.0003	0.005	0.1	70-130	15
Tetrachloroethene	127-18-4	0.0003	0.005	0.005	70-130	15
Toluene	108-88-3	0.0004	0.005	1	70-130	15
trans-1,2-Dichloroethene	156-60-5	0.0005	0.005	0.1	70-130	15
trans-1,3-Dichloropropene	10061-02-6	0.0001	0.001	0.001	70-130	15
Trichloroethene	79-01-6	0.0003	0.005	0.005	70-130	15
Vinyl Chloride	75-01-4	0.0003	0.002	0.002	70-130	15
Total Xylene	1330-20-7	0.001	0.015	10	70-130	15

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TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER						
ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	MS/MSD % R	MS/MSD % RPD
PESTICIDES (8081)		mg/L	mg/L	Cleanup Critiria mg/L		
4,4'-DDD	72-54-8	0.000022	0.00005	0.014	30-150	25
4,4'-DDE	72-55-9	0.000017	0.00005	0.01	30-150	25
4,4'-DDT	50-29-3	0.000008	0.00005	0.006	30-150	25
Aldrin	309-00-2	0.000011	0.00005	0.014	30-150	25
a-BHC	319-84-6	0.000013	0.00005	0.00011	30-150	25
a-Chlordane	5103-71-9	0.000017	0.00005	0.002	30-150	25
b-BHC	319-85-7	0.000018	0.00005	-	30-150	25
d-BHC	319-86-8	0.000012	0.00005	-	30-150	25
Dieldrin	60-57-1	0.000028	0.00005	0.009	30-150	25
Endosulfan I	959-98-8	0.000015	0.00005	0.042	30-150	25
Endosulfan II	33213-65-9	0.00004	0.00005	0.042	30-150	25
Endosulfan sulfate	1031-07-8	0.000019	0.00005	0.042	30-150	25
Endrin	72-20-8	0.000016	0.00005	0.002	30-150	25
Endrin Aldehyde	7421-93-4	0.000017	0.00005	0.002	30-150	25
Ednrin Ketone	53494-70-5	0.000014	0.00005	0.002	30-150	25
g-BHC (Lindane)	58-89-9	0.000014	0.00005	0.0002	30-150	25
g-Chlordane	5566-34-7	0.000017	0.00005	0.002	30-150	25
Heptachlor	76-44-8	0.00002	0.00005	0.0004	30-150	25
Heptachlor Epoxide	1024-57-3	0.000017	0.00005	0.0002	30-150	25
4,4'-Methoxychlor	72-43-5	0.000014	0.00005	0.04	30-150	25
Toxaphene	8001-35-3	0.0004	0.001	0.003	30-150	25

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

TABLE 8 PREPORTING LIMITS COMPARED TO TACO
STAT Analysis Corp.
WATER METHOD DETECTION LIMITS, REPORTING LIMITS, AND IEPA CRITERIA

Illinois GROUNDWATER

ANALYTE (& EPA METHOD)	CAS #	MDL	MRL	TACO GW	MS/MSD % R	MS/MSD % RPD
PCBs EPA (8082)		mg/L	mg/L	Cleanup Critiria mg/L		
Aroclor 1016	12674-11-2	0.00017	0.0005	-	30-150	25
Aroclor 1221	11104-28-2	0.0001	0.0005	-		
Aroclor 1232	11141-16-5	0.0001	0.0005	-		
Aroclor 1242	53469-21-9	0.0001	0.0005	-		
Aroclor 1248	12672-29-6	0.0001	0.0005	-		
Aroclor 1254	11097-69-1	0.0001	0.0005	-		
Aroclor 1260	11096-82-5	0.00008	0.0005	-	30-150	25
PCBs (total)	1336-36-3			0.0005		

- No toxicity criteria provided in 35 Ill. Admn. Part 742 Appendix B, Tables A and B

Appendix B

Appendix B - Terracon Information

Résumés of Key Terracon Staff

LINDA CHEN YANG, P.G.
ENVIRONMENTAL DEPARTMENT MANAGER

PROFESSIONAL EXPERIENCE

Ms. Yang is the Illinois Environmental Department Manager overseeing the environmental service line in the State of Illinois. Ms. Yang has over 17 years of experience in environmental consulting with a broad range of expertise serving clients with site investigation, remediation, risk assessment, negotiation, life-cycle liabilities assessment, and achieving closures within the regulatory framework in Illinois, Indiana, Ohio, Michigan, Wisconsin, Minnesota, and Pennsylvania. Ms. Yang has extensive knowledge of federal and state environmental programs and regulations, including CERCLA, RCRA, and regulations in multiple States (IEPA LUST, SRP, TACO, and IDEM RISC regulations).

Additionally, Ms. Yang specializes at major oil and gas clients' project management, project framing, milestone evaluation, life-cycle assessment, NPV calculation, and decision making support.

Being the Environmental Department Manager, Ms. Yang manages teams of scientists and engineers to provide environmental services to our regional and national clients. Services include environmental due diligence, subsurface investigation, remediation, compliance, and industrial hygiene. Clients include State, municipalities, commercial, and industrial.

Before joining Terracon, Ms Yang worked for a fortune 500 company managing an environmental group in the Chicago area. The work included remediation, environmental, health and safety consultancy, environmental due diligence, compliance, industrial hygiene, air, capital permitting, water and natural resources. She successfully established key relationships and developed regional and global accounts including Oil and Gas, Power Utility, and Manufactures.

PROJECT EXPERIENCE

Program Manager, Major Oil and Gas Company Legacy Sites Liability Reduction and Compliance Portfolio, Illinois, Indiana, Wisconsin, Minnesota, Ohio, Pennsylvania, West Virginia, New York, North Carolina, South Carolina, Tennessee, Florida, and Nebraska. Program manager for a nation-wide petroleum/agricultural portfolio including service stations, terminals, bulk plants and former agricultural facilities. Led a cross functional, cross-regional team to assist the client to reduce its contaminated assets. The Program was a long term program to assess, remediate, and receive regulatory closure of the sites. Responsible for management and technical oversight of up to 6 project managers and other support staff conducting assessment and remediation of 40 legacy sites. Remedial/site closure strategies and pilot testing employed to date have included multi-phase extraction and treatment, air sparging and soil vapor extraction, groundwater pump and treat, soil excavation and disposal, bioremediation, monitored natural attenuation, and risk assessment to close sites. Regulatory agency closure has been achieved on 20 sites in the eastern United States.

Education
Bachelor of Science, Geology, 1990, Peking University, China

Graduate Studies, Geology, 1994, The University of Illinois Chicago

Masters of Business Administration (MBA), 2005, Kellogg Graduate School of Management, Northwestern University

Registrations
Professional Geologist: Illinois, 196.000764

Certifications
OSHA 40-Hour Health and Safety Training

Loss Prevention System

Smith Driving

FA/CPR

Affiliations
Asian Women Professionals Network

Work History
Terracon, Environmental Department Manager, 2011-present

AECOM Technical Services, Inc., Department Manager 2005-2011; Strategic Account Manager; Regional Sales Director 2006-2009; Project Manager, 1999-2005; Project Geologist, 1994-1999

Terracon

Project Coordinator, Major Pipeline Company, Emergency Response, Illinois. During the pipeline spill emergency response activities, Ms. Yang coordinated the technical experts and field staff assisting client with multiple work plans preparation required by the EPA Region 5, onsite permitting for water and wetland, manifesting, various agencies communication, and other supporting activities.

Project Manager, Major Pipeline Company, Groundwater Monitoring and Remediation, Illinois, Indiana, Ohio. Project manager for several superfund sites to conduct groundwater monitoring and remediation system O & M. Responsibilities include, but not limited to: proposal preparation, work plan preparation, project coordination, scheduling field work and staffing, report preparations, and agency communication.

Account Manager, Major Utility Company, Illinois. Account Manager for a Utility Client. Led the team assisting client with its stormwater/wetland permitting program, industrial hygiene needs, and greenhouse gas and carbon footprint reduction initiatives. Responsible for all project execution, health and safety, meeting client business objectives, construction and activity schedules, and cost effectiveness. Spearheaded Client/Consultant joint community services including Annual Earth Day fund raising and local prairie development.

Project Manager, Major Utility Company, Leaking Underground Storage Tanks Program Management, Illinois. Project Manager for a portfolio of LUST sites. Responsibilities included, but not limited to: proposal preparation, project coordination, field work scheduling, work plan preparation, negotiation with the IEPA, report preparation, state fund reimbursement preparation, and project management. Achieved site closures utilizing conventional and innovative technologies.

Project Manager, Major Utility Company, Phase I/II Environmental Site Assessments, Illinois. Project Coordinator for a Phase I/II ESA portfolio during a major power plants property transaction. Responsibilities included, but not limited to project coordination, field work scheduling, ESA reports preparation, budget tracking and project management.

Project Director, Major Rail Road Company, Air Compliance Program. Assisted the client in its property acquisition environmental site assessment in the Chicago area, and its rail yards' air compliance, tracking, permitting condition monitoring and program management nationwide.

Project Manager, Retail Petroleum, Underground Storage Tanks, Illinois, Indian, Michigan, Ohio, Wisconsin. Project manager for petroleum underground storage tank projects. Responsibilities included, but not limited to: direct removal and closure of USTs, groundwater monitoring well installation, soil and groundwater sampling, feasibility tests, data evaluation, report generation, regulatory compliance, groundwater modeling, soil and groundwater remediation design, system installation, and operation and maintenance (O & M), project management. She is also responsible for risk-based corrective action evaluation for several Illinois sites. Negotiate with State regulators regarding "No Further Action" closures of leaking USTs and obtain site specific cleanup objectives through various risk assessment methods.

Project Manager, Major Petroleum Company, Soil Vapor Extraction and Air Sparging Remediation System, Indiana. Site manager for a project, which has a soil vapor extraction (SVE) and air sparging system. Responsibilities included performing SVE and air sparging pilot tests, data evaluation, assisting system design, system installation oversight, system operation and maintenance, project coordination, and report generation.

DAVID E. KOCH, R.B.P.
QUALITY ASSURANCE / SENIOR CONSULTANT

PROFESSIONAL EXPERIENCE

Mr. Koch is a senior principal of the firm providing specialized technical expertise and training in the fields of risk-based assessment and mitigation. Mr. Koch has worked in environmental assessment and remediation throughout the United States. He serves as a Senior Consultant providing senior technical support for major projects involving public or client exposures. Mr. Koch's emphasis is in evaluating and mitigating public health risk from chemical releases under Brownfields, Superfund (CERCLA/SARA), RCRA, RBCA and other voluntary programs.

Project specialties involve risk-based evaluation and solutions for redevelopment for brownfields restoration and industrial risk-based solutions. Mr. Koch develops and conducts risk-based strategies on behalf of municipal, public and industrial clients in restoration of impaired properties. Mr. Koch has supervised and conducted risk assessments for industrial, recreational and residential project sites. Mr. Koch has personally managed or participated actively in more than 90 EPA Brownfields assessment, cleanup and revolving loan fund grant projects. He has been on 6 of Terracon's 7 regional and national Phoenix Award Winner teams.

Mr. Koch actively engages in the redevelopment process of rural, urban and industrial properties from grant writing, through siting/ feasibility to corrective action and long-term post-development monitoring. He has coined the internal phrase "consult beyond the chemistry" in seeking environmental solutions that progress at the rate of business, not environmental regulation.

Dave routinely speaks and trains on behalf of state and federal Brownfield agencies as well as non-profit and community groups. Since 2005, he developed and implemented more than 40 Brownfield training workshops for the government and private sector in addition to dozens of other project-specific outreach and public engagement activities.

He serves on ASTM's E50 Environmental main and sub-committees to develop national environmental standards, recently engaging in the subcommittee formed for Green Cleanups. As a founding subcommittee member Dave helped develop the ASTM risk-based corrective action standards. Mr. Koch has assisted in risk-based environmental program rule-making and program development in several states.

SELECTED BROWNFIELDS PROJECT EXPERIENCE
DETAILS & OTHER RELATED EXPERIENCE AVAILABLE ON REQUEST

- **Project Manager or Senior Consultant conducting technical and programmatic work directly to the following projects.**
  - EPA4 Petroleum Assessment Grant 2005 – 2007, 1300 Acre Downtown Inventory & Assessment, Clarksville, TN
  - EPA4 Hazardous Cleanup Grant 2006, Scotland Hospital, Laurinburg, NC
  - EPA4 Petroleum Cleanup Grant 2006, Scotland Hospital, Laurinburg, NC

EDUCATION

Bachelor of Arts, Science/Geology, 1980, University of Northern Iowa

Graduate Study, Geology, Augustana College, IL

REGISTRATIONS

Registered Brownfields Professional #33 (2005-2010)

Certified Groundwater Professional Iowa #1200 (Former 1994-2004)

AFFILIATIONS

Institute of Brownfield Professionals (Former)

National Brownfields Association

National Association of Local Governmental Environmental Professionals (NALGEP)

American Wind Energy Association

Sigma Gamma Epsilon (National Honor Society for the Earth Sciences)

ASTM E50 & E50.04 RBCA Committees & Subcommittees

WORK HISTORY

Terracon Consultants, Inc., Sr. Consultant / National Sector Leader for Brownfields Services, 2002-Present National Sector Leader for Renewable Energy, 2007-2009 Principal/Office Manager, 1990-1994, 1998-2002; Special Projects Manager, 1996-1998; Operations Manager/Corporate Safety Officer, 1986-1989

Soil Testing Services of Iowa 1983-1986; Environmental Staff Geologist, 1980-1983; Quality Control Supervisor/Field Geologist, 1978-1980

Cedar County, Iowa – County Engineer's Office Survey and Construction Materials Technician 1973-1977

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PROJECT EXPERIENCE (continued)

- EPA5 Supplemental Assessment Grant 2001-2003, Riverfront Brownfields Landfill Redevelopment, East Moline, IL
- EPA4 Petroleum Assessment Grant 2005 – 2007, 1300 Acre Downtown Inventory & Assessment, Clarksville, TN
- EPA4 Hazardous Substance Cleanup Grant 2006, Former Scotland Memorial Hospital, Laurinburg, NC
- EPA4 Petroleum Cleanup Grant 2006, Former Scotland Memorial Hospital, Laurinburg, NC
- EPA4 Hazardous Substance Assessment Grant 2009-2011, Community-wide Assessments, Talladega, AL
- EPA4 Petroleum Assessment Grant 2009-2011, Community-wide Assessments, Talladega, AL
- EPA5 Supplemental Assessment Grant 2001-2003, Riverfront Brownfields Landfill Redevelopment, East Moline, IL
- EPA5 Assessment Demonstration Grant 2001-2003, Downtown Riverfront Corridor Project, Wausau, WI
- EPA5 Hazardous Substance Assessment 2006-2009, Community-wide Assessment Program, Moline, IL
- EPA5 Hazardous Substance Assessment 2006-2009, Community-wide Brownfields Assessments, Rock Island, IL
- EPA5 Petroleum Assessment Grant 2006-2009 Community-wide Brownfields Assessment Program, Rock Island, IL
- EPA6 Hazardous Substance Assessment 2006-2008, Ark-Tex Council of Governments,
- EPA6 Petroleum Grant Assessment 2007-200, Ark-Tex Council of Governments
- EPA6 Hazardous Substance Assessment 2004-2006, Community-wide Assessments, Helena & West Helena, AR
- EPA6 Petroleum Assessment Grant 2004-2006, Community-wide Brownfields Program, Helena & West Helena, AR
- EPA6 Assessment Demonstration Grant 2000-2003, Poultry Agribusiness Landfill Restoration, Cherokee Nation, OK
- EPA6 Assessment Demonstration Grant 2001-2003, Lee St. Corridor Restoration, Greenville, TX
- EPA6 Hazardous Substance Assessment 2004-2006, Lee St. Corridor Restoration, Greenville, TX
- EPA6 Petroleum Assessment Grant 2004-2006, Lee St. Corridor Restoration, Greenville, TX
- EPA6 Hazardous Substances Cleanup Grant 2005-2006, Smarthouse Way Property, North Little Rock, AR
- EPA6 Hazardous Substances Assessment 2008-2011, City of Arlington Brownfields Program, Arlington, TX
- EPA6 Petroleum Assessment Grant 2008-2011, City of Arlington Brownfields Program, Arlington, TX
- EPA7 Assessment Demonstration Grant 1999-2001, 220-Acre Inventory & Assessments, Coralville, IA
- EPA7 Supplemental Assessment Grant 2000-2002, 1st Avenue Revitalization, Coralville, IA
- EPA7 Supplemental Assessment Grant 2002-2004, 1st Avenue Revitalization, Coralville, IA
- EPA7 Assessment Demonstration Grant 1999–2000, Liberty Square Redevelopment Project, City of Clinton, IA
- EPA7 Supplemental Assessment Grant 2001 Liberty Square Redevelopment Project, City of Clinton, IA
- EPA7 Assessment Demonstration Grant 2001–2004, Riverfront Industrial Redevelopment, St. Joseph, MO
- EPA7 Petroleum Cleanup Grant 2004-2006, Flat Branch Creek Park Restoration, Columbia, MA
- EPA7 Hazardous Substance Assessment 2004-2007, Iowa River Landing Revitalization, Coralville, IA
- EPA7 Petroleum Assessment Grant 2004-2007, Iowa River Landing Revitalization, Coralville, IA
- EPA7 Cleanup Revolving Loan Fund 2004-2007, Iowa River landing Revitalization, Coralville, IA
- EPA7 Hazardous Substance Assessment 2003-2005, Jordan Creek Corridor Redevelopment, Springfield, MO
- EPA7 Petroleum Assessment Grant 2004-2007, Jordan Creek Corridor Redevelopment, Springfield, MO
- EPA7 Hazardous Substance Assessment 2004-2007, Jordan Creek Corridor Redevelopment, Springfield, MO
- EPA7 Hazardous Substance Cleanup Grant 2008-2011, West Meadows/ Locomotive Yard Restoration, Springfield, MO
- EPA7 Hazardous Substance Assessment 2005–2007, Southeast Iowa Regional Planning Commission, Burlington, IA
- EPA7 Petroleum Assessment Grant 2005–2007, Southeast Iowa Regional Planning Commission, Burlington, IA
- EPA7 Assessment Demonstration Grant 2000–2001, Southside Neighborhood Brownfields Project, Cedar Rapids, IA
- EPA7 Supplemental Assessment Grant 2001–2003, Southside Neighborhood Brownfields Project, Cedar Rapids, IA
- EPA7 Assessment Demonstration Grant 2003-2005, Port of Dubuque Brownfields Restoration, Dubuque, IA
- EPA7 Petroleum Cleanup Grant 2003-2005, Port of Dubuque Brownfields Restoration, Dubuque, IA
- EPA7 Hazardous Substance Assessment 2005–2006, Atchison Downtown & Riverfront Revitalization, Atchison, KS
- EPA7 Petroleum Assessment Grant 2006–2008, Blue Springs Inventory & Corridor Redevelopment, Blue Springs, MO
- EPA7 Petroleum Assessment Grant 2008-2011, Iowa River Landing, Coralville, IA, IA
- EPA7 Hazardous Substance Assessment 2008-2011, Iowa River Landing, Coralville, IA, IA
- EPA8 Assessment Demonstration Grant 2000–2002, Union Center and Railyard Restoration, Evanston, WY
- EPA8 Assessment Demonstration Grant 1999–2000, San Haven Former Mental Hospital Project, Rollette County, ND
- EPA8 Supplemental Assessment Grant 2001-2003, Union Center and Railyard Restoration, Evanston, WY
- EPA8 Supplemental Assessment Grant 2000-2001, San Haven Former Mental Hospital Project, Rollette County, ND
- EPA8 Assessment Demonstration Grant 2000-2004, 250 Acre Riverfront Business District, Yankton, SD
- EPA9 Assessment Petroleum Grant 2007-2008, Route 66 Central Corridor, City of Flagstaff, / Flagstaff, AZ
- EPA9 Assessment Demonstration Grant 2000-2003, Abandoned Lumber Mill, White Mountain Apache / McNary, AZ
- EPA10 Petroleum Assessment Grant 2007-2008, Medford Urban Renewal Agency, Medford, OR

- State of Kansas Brownfields Targeted Assessment Contractor, 2003 - 2010

Contract/Project Manager for state contract to assess Brownfields sites statewide for Kansas Department of Health and Environment, Brownfields program, State of Kansas 2003-2006. Senior Consultant 2007-2010.

• EPA Technical Assistance to Brownfields Communities (TAB) Contractor, 2008 - 2011
Contractor to assist Kansas State University/Centers for Hazardous Waste to implement EPA's TAB program by providing educational and technical services to communities affected by Brownfields in EPA Regions 5 and 7 in a 10-state area.

BRIAN PORTER, P.E.

Senior Technical Support

PROFESSIONAL EXPERIENCE

Mr. Porter manages the environmental department in Terracon's St. Louis office. He oversees all aspects of the department's operations. His experience ranges from simple Phase I Environmental Site Assessments (ESAs) to complex, mulit-site brownfield redevelopments. Mr. Porter has worked with a variety of media, including air, soil, and water.

Mr. Porter has extensive experience using the concept of risk-based evaluation for industrial and commercial property restoration. His experience includes projects in brownfields and voluntary cleanup programs administered by the State of Illinois, USEPA Region 5, and USEPA Region 7. His experience includes frequent use and contact with government agencies.

Mr. Porter also has experience with several other technical areas including regulatory compliance, landfill evaluation and design, agricultural assessments, indoor air quality, and limited geotechnical evaluation.

PROJECT EXPERIENCE

Brownfields Redevelopment

Project titles include Senior Project Manager, Phase I Coordinator, and Phase II Field Captain/Coordinator for managing and supervising field activities and data collection consistent with USEPA and state quality assurance/quality control requirements. Managed and directed project scope development, final report preparation and review, technical considerations, outreach efforts and materials, and financial stability of the project. Also worked with grant recipients to leverage additional funding.

- Moline, Illinois city-wide USEPA brownfield project. Served as Senior Project Manager responsible for overall management and performance of the project.
- Rock Island, Illinois city-wide USEPA brownfield project. Served as Senior Project Manager responsible for overall management and performance of the project.
- Multiple sites in Keokuk, Fort Madison, and Burlington, Iowa as part of the Southeast Iowa Regional Planning Commission Brownfields Initiative. Served as Senior Project Manager responsible for overall management and performance of this USEPA grant project.
- East Moline, Illinois site with concerns related to industrial fill activities (East Moline Riverfront and Downtown Redevelopment Project). Initially served as Phase II Field Coordinator, then as Senior Project Manager responsible for overall management and performance of this USEPA and State funded project.
- Galesburg, Illinois sites with concerns related to former paint manufacturing and former dry cleaning activities. Served as Senior Project Manager responsible for overall management and performance of this State funded project.

Education

Bachelor of Science, Civil Engineering (Environmental), 1997, University of Illinois

Registrations

Licensed Professional Engineer: Illinois No. 062-055804

Licensed Professional Engineer: Iowa No. 16643

Licensed Professional Engineer: Missouri No. 2008015913

Certifications

40-Hour OSHA HAZWOPER , #40-1351

USEPA Region 7 Quality Assurance Training in Systematic Planning Process and Quality Assurance Project Plans

Affiliations

American Society of Civil Engineers, Member 2000-Current; Quad Cities Section President and Board of DirectorsMember 2004-2008

National Society of Professional Engineers 2008-Current

Work History

Terracon, 1997 - Present

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- Moline, Illinois site with concerns related to industrial manufacturing activities, currently developed as a successful, regional civic center and sports arena (Mark of the Quad Cities and John Deere Commons, Moline, Illinois). Duties included managing groundwater montioring, on-site consultation with USEPA representatives, preparation of a summary document detailing substantial project activities and analytical results from multiple years of assessment, and preparation of documents required by Illinois' Site Remediation Program. This brownfield project was privately funded.

- Multiple sites in Dubuque, Iowa including public recreation, undeveloped, residential, commercial and industrial land uses. Phase I and II assessments were conducted on select properties to aid the City in planning its redevelopment efforts. The project was selected as a USEPA Region 7 Phoenix Award Finalist. Served as Phase I and II Coordinator. Duties included management of field crews and equipment, oversight of field procedures, and preparation of project documents including the project QAPP, Phase I and II Site Assessment Reports, and remedial cost estimates for redevelopment.

- Other brownfield projects in Metropolis, Illinois; Cedar Rapids, Clinton, Coralville, and Des Moines, Iowa; St. Joseph, Missouri; Evanston, Wyoming; and Wausau, Wisconsin.

COURSES/CONFERENCES

Systematic Planning Process and Quality Assurance Project Plans - USEPA, 1999

FIELDS/SADA Conference - USEPA, 2003

Brownfields All Pilots Forum - USEPA, 2003

Midwest Summit on the Sustainable Redevelopment of Brownfields - USEPA, 2005

Brownfields 2005 Conference, 2005

ASCE Zone III Leadership Conference, 2006

Midwest Community Development Institute, Corporate Sponsor and Presenter, 2007-2008

Four-State Professional Development Conference, Corporate Sponsor and Attendee, 2008

Illinois Tax Increment Association, Corporate Sponsor and Attendee, 2008

Missouri Conference at the Lake, Speaker, 2009

Missouri Waste Control Coalition Annual Conference, Speaker, 2010

PUBLICATIONS/PRESENTATIONS

Porter, Brian R., *Evolving Complexities of the Iowa Land Recycling Program: An Example in Application* (Quad City Society of Environmental Professionals, Microsoft® PowerPoint, 2002).

Porter, Brian R., *Terracon Limited Site Investigation Field Services Guide - Chapter 4: Investigative Strategies for Common Site Scenarios* (Internal Training Guide, Microsoft® Word, 2005).

PUBLICATIONS/PRESENTATIONS (continued)

Porter, Brian R., *Terracon Limited Site Investigation Field Services Guide - Chapter 12: Soil Sampling and Handling for VOC Analysis* (Internal Training Guide, Microsoft® Word, 2005).

Project Interview, KOKX Talk Radio, Participated in a live radio "Q&A" broadcast related to the Southeast Iowa Regional Planning Commission Brownfield Assessment Project. Other participants included the client, Iowa Department of Natural Resources, and the Mayor of Keokuk, Iowa.

Porter, Brian R., *Terracon Limited Site Investigation Field Services Guide - Chapter 4: Investigative Strategies for Common Site Scenarios* (Internal Training Presentation, Microsoft® PowerPoint, 2007).

Porter, Brian R., *Terracon Limited Site Investigation Field Services Guide - Chapter 12: Soil Sampling and Handling for VOC Analysis* (Internal Training Presentation, Microsoft® PowerPoint, 2007).

Porter, Brian R., *Brownfields: Population does not Measure Success, Small City Success in the Midwest* (Midwest Community Development Institute, Microsoft® PowerPoint, 2007).

Porter, Brian R. and Tapp, Brian, *Phased and Confused?: Rigorous standards during the Phase I assessment is [sic] necessary to complete complicated brownfield developments,* Heartland Real Estate Business, Feature Article, October 2007.

Porter, Brian R., *Environmental Engineering* (Exploring Engineering Careers, St. Ambrose University Outreach Event, Microsoft® PowerPoint, 2007).

Porter, Brian R., *Brownfield Remediation and Funding Options* (Midwest Community Development Institute, Microsoft® PowerPoint, 2008).

Porter, Brian R. and Burke, Patrick, *Breaking the Brownfields Cycle: Incorporating Sustainable Practices Into Municipal Operations and Environmental Site Assessments* (USEPA WebCast, Hazardous Waste Cleanup Information [CLU-IN], Microsoft® PowerPoint, 2008).

Porter, Brian R., *Cleanup in the Heart of America, No Mild-Mannered Approach to Brownfields in Metropolis,* Delivering Success (Terracon Client Newsletter), Winter 2009.

Porter, Brian R., *'City of Tomorrow' Banking on the Future,* Brownfield News, June 2009.

Porter, Brian R., *Remediation of a Dry Cleaning Chemical Facility* (Environmental Lecture Series, St. Louis University, Microsoft® PowerPoint, April 21, 2010).

Porter, Brian R., *Long-Term Groundwater Monitoring and Stability/Trend Evaluation (Plume Stability)* (Missouri Waste Control Coalition Annual Conference, Microsoft® PowerPoint, June 22, 2010).

MATT CATLIN, P.E.
PRINCIPAL / REGIONAL MANAGER

PROFESSIONAL EXPERIENCE

Mr. Catlin is the Regional Manager for Terracon's St. Louis and Bloomington operations. He also oversees environmental operations of Terracon's Bettendorf and Naperville/Chicago offices, and also serves as the Office Manager for the St. Louis office. Mr. Catlin is a member of Terracon's investigation and site remediation Practice Resource Group where he serves as a technical resource for the company for site investigations and risk based corrective action.

Mr. Catlin's experience includes:

- Management of more than 50 sites though regulatory programs including Illinois LUST, Illinois SRP, Missouri VCP and Missouri LPST. Particulary strong experience and expertise in the Illinois programs.
- Supervision of hundreds of instrusive site investigations in Illinois, Missouri, and Indiana.
- Supervision of more than a thousand Phase I Environmental Site Assessments of a wide variety of sites.
- Mr. Catlin has also supervised dozens of underground storage tank closures in Illinois, Indiana, and Missouri.

PROJECT EXPERIENCE

Manufacturing Site - Hannibal, Missouri
Explain your role on the project. Provide a very brief description of the project and the services you provided. See the example below.

20 Acre Former PCE Manufacturing facility, Metropolis, Illinois
Helped City obtain funding through Illinois EPA's Municipal Brownfield program by preparing grant application. Completed Site Investigation and Remedial Action Plan for site using State funding, managed grant. Helped City obtain USEPA Brownfield Cleanup Grant by preparing grant application. Designed and implemented insitu chemical oxidation, and insitu metals stabilzation strategies which resulted in attaining of site specific remedial objectives and NFR status for the site.

Agrichemical Site Remediation – 15 sites central/southern Illinois
Designed and performed investigations at numerous agrichemical fertilizer/pesticide distribution and storage centers in Illinois. Developed risk-based cleanup objectives for each site. Using a variety of Tier I, Tier II, and Tier 3 methods, total remediation costs were significantly reduced. Land farming under Illinois Department of agriculture signiicantly reduced the cost of managing more than 2,000 cubic yards of highly impacted soil. Sites participated in Illinois Site Remediation Program.

Illinois Municipal Brownfields Grant Site, DuQuoin, Illinois
Working with officials from the City of DuQUoin, applied for andreceived Brownfield grant to investigate abandoned contaminated site. Developed

Education
Masters of Environmental Engineering, 2000, Illinois Institute of Technology

Bachelor of Environmental Science, 1995, Monmouth College

Registrations
Professional Engineer (Illinois)

Certifications
OSHA 40-Hour
OSHA Confined Space
OSHA 8-Hour Supervisor

Work History
Terracon, Regional Manager, 2008-Present

Terracon, Office Manager, 2007-Present

Terracon, Environmental Services Manager, 2002-2008

Terracon, Phase II Section Manager, 2000-2002

Terracon, Project Manager, 1997-2000

Terri-Tec Environmental, Environmental Scientist, 1995-1997

Clean Harbors Environmental, Field Services Technician, 1995

Environmental Management Group,

Wastewater Treatment Plant technician, 1993-1995

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risk based cleanup objectives that resulted in the economical and successful redevelopment of the site and issuance of No Further Remediation Letter.

Pillsbury Plant – Belvidere, Illinois
Re-evaluated state-approved risk-based soil remediation objectives performed by another firm. State approved Terracon's recalculated objectives which were four times greater that previously proposed, significantly reducing the need for soil excavation and saving the client significant remedial expense. Supervised subsequent removal of 6,000 cubic yards of impacted soil.

Former Manufacturing Facility – Plainfield, Illinois
Served as the project manager for a former Ball Glass Container manufacturing facility redevelopment project in Plainfield, Illinois. Major phases of the project included a focused remediation of a former wastewater treatment lagoon and construction of an engineered barrier, temporary diversion of Norman Creek for an Army Corps of Engineers-regulated creek remediation, and a heavy metals
Risk assessment. The project included application of risk-based remediation objectives, and a Tier 3 statistical analysis of site-specific background arsenic concentrations. Two Focused No Further Remediation letters were issued by the IEPA for the lagoons and site subsurface arsenic concentrations.

Naperville Park District – Aquifer Pump Test – Naperville, Illinois
Designed, completed and evaluated a three-day pump test of a bedrock aquifer to evaluate the potential effect of the Park District's proposed 200,000 gallon per day irrigation demand on nearby residents. Presented findings to Park District Board at public meeting.

City of Chicago Brownfield Site
Served as the project manager for a brownfields project in Chicago, which included the removal of 13,000 cubic yards of impacted soil, the removal of over 110,000 gallons of UST storage capacity, the calculations of risk-based remediation objectives, and construction of engineered barriers. The Remedial Action Plan was approved by the IEPA on an expedited basis, and the property was developed for residential use. As part of this project, Terracon received the first City of Chicago Highway Authority Agreement where liability for off-site impact does not transfer with the property.

Agrichemical Site Remediation Leaf River, Illinois
Obtained the necessary permits and supervised the removal and subsequent land farming of approximately 5,000 cubic yards of agrichemical impacted soils resulting from an agrichemical facility fire in Leaf River, Illinos.

Agrichemical Facilities Permitting - Western Illinois
Completed the Illinois Department of Agriculture agchem facility
permitting for fifteen facilities, and designed and supervised the
construction of seven steel secondary containment structures, the largest of which provided over 400,000 gallons of secondary containment.

Agrichemical Spill Consulting - Illinois
Provided delineation services and the calculation of risk-based objectives for numerous spills for members of nationally known cooperative.

Dry Cleaners Remediation Site – Naperville, Illinois
Designed, installed and operated a soil vapor extraction system remediating a perchloroethylene release from a former drycleaning operation. Soil concentrations successfully reduced from free product levels to levels which could remain in place. Evaluated potential bedrock groundwater contamination and developed Tier 3 arguments leading IEPA to issue a No Further Remediation Letter.

Former Trap Range – Woodstock, Illinois
Developed a shot-count sampling procedure to delineate the shot-fall zone at a former Trap Range in Woodstock, Illinois.

Dry Cleaners Remediation Site – Prospect Heights, IL
Project Manager for site where hazardous waste levels of PCE were excavated for incineration. Residual contamination excavation was limited to meeting the risk-based modeling requirements toward a planned potable water well for an on-site grocery store.

Former Trap Range – Springfield, Illinois
Delineated 22 acre shotfall zone, and conducted successful soil stabilization bench test to render the soil non-hazardous for lead. Subsequently, a field scale one-acre test plot was implemented. The results of the field scale test are currently proposed to render the entire site non-hazardous and to allow for pathway exclusion.

Range Characterization – Indiana Dunes National Lakeshore (National Parks Service)
Characterized impacts to soil and groundwater at an active Federal range and at two inactive historical ranges where small arms were fired into sand dunes. Developed remedial cost estimates.

Metals Plating Facility Remediation – Chicago, Illinois
Project manager for remediation of a former metals plating facility, including stabilization of heavy metals impacted soil to levels below hazardous concentrations.

THOMAS D. CULIG
PROJECT MANAGER

PROFESSIONAL EXPERIENCE

Mr. Thomas D. Culig is a Project Manager in Terracon's Chicago, Illinois office. He has over 10 years of experience in the environmental consulting industry. Mr. Culig has successfully completed hundreds of Phase II Limited Site Investigations (LSIs) for property transactions and has obtained over 30 No Further Remediation (NFR) Letters from the Illinois EPA's Site Remediation (SRP) and Leaking Underground Storage Tank (LUST) Programs. Project management experience includes developing workplans and proposals, remediation and environmental closure cost estimates, and preparing techinical reports. Fieldwork experience includes environmental remediation oversight, UST removal oversight, goundwater sampling projects, and soil sampling projects. Project sites have covered agricultural land, commercial, and large industrial facilities.

PROJECT EXPERIENCE

Phase II Subsurface Investigations & Remediation

Mr. Culig has performed hundreds of LSIs in over six states. Typical LSIs have included detailed investigation design, site investigation, soil and groundwater sampling, client interface, and report preparation. Mr. Culig has also coordinated and managed over 100 underground storage tank (UST) removals in California and Illinois. Some notable Phase II projects Mr. Culig has performed include:

- Gasoline Retail Stations – Northern California
  Conducted subsurface investigations at over 50 gasoline retail stations throughout northern California for two large petroleum companies. Experience includes extensive soil and groundwater sampling in a variety of subsurface lithology, oversight of UST removals, client regulatory correspondance, and technical report writing.

- Chicago Department of Environment (DOE) Projects – Chicago, Illinois
  - Conducted a Phase II LSI on a vacant property that is being considered for use as a community garden.
  - Performed technical file review on a site and determined onsite impacts were due to adjacent manufacturing property. Obtained closure of LUST incident without performing the approved additional fieldwork.

- Proposed Metra Railroad Expansion – Bellwood, Illinois
  Conducted focused and supplemental site investigation soil boring work at large ink factory. Known RECs included potential USTs, onsite industrial operations, and adjacent railroad tracks. Site investigation activities included ground-penetrating radar, soil boring advancement and sampling, and surveying. Responsibilites included fieldwork scope development, technical meetings with numerous parties, development of preliminary environmental cleanup cost estimates for the site and adjacent industrial properties, and technical report writing.

Education
B.S., Geoscience, 1999, University of Iowa, Iowa City, Iowa

Certifications
40-Hour OSHA Hazardous Waste Operations (HAZWOPER) Training

8-Hour OSHA Confined Space (HAZWOPER) Training

Work History
Terracon, Chicago Office, Project Manager, 2011-present

Pioneer Engineering & Environmental Services, Inc., Project Manager, 2005-2010

TEC Accutite, Project Geologist, 2002-2004

Environmental Resolutions Inc., Staff Geologist, 2000-2002

Terracon

Illinois EPA Site Remediation Program (SRP) Experience

Mr. Culig has succesfully obtained NFR Letters for over 25 sites through the Illinois EPA's SRP. Site's ranged from small independent dry cleaners to large industrial properties being redeveloped for residential use. A list of notable projects in which Mr. Culig served as project manager and obtained a Focused or Comprehensive NFR letter include:

- Galewood Crossings – Chicago, Illinois
  Served as project manager for the redevelopment of a former 10-acre railroad yard and soy bean storage facilty into a single-family residential redevelopment on Chicago's west side. Known RECs included historical use of site as a railroad yard and potential USTs. Actvities included extensive soil and groundwater testing, test pits/trenching, UST removal, oversight of soil import/removal and engineered barrier placement. Responsibilites included project management, fieldwork scope development, and IEPA correspondance and report writing. Completed final remediation activities in stages to expedite real estate closings and resulting in the issuance of 12 Comprehensive NFR letters from the IEPA.

- Downtown Redevelopment – Chicago, Illinois
  Served as project manager for the redevelopment of a former auto repair facility being redeveloped as a 15-story mixed-use highrise in downtown Chicago. Actvities included extensive soil and groundwater testing, in-situ remediation of hazardous lead levels to meet special waste requirements, UST removals, oversight of soil removal, and engineered barrier placement documentation. Responsibilites included workplan development, development of proposed redevelopment costs associated with onsite environmental conditions, and IEPA correspondance and report writing.

- Community Wellness Center – Chicago, Illinois
  Served as project manager for the redevelopment of vacant land into a community wellness center that achieved LEED-Gold level certification. Actvities included soil and groundwater testing, ground-penetrating radar, test pits/trenching, UST removals, engineered barrier placement documentation. Responsibilites included project management, workplan development, technical meetings with client and developers, and IEPA correspondance and report writing.

- Former Manufacturing Facility – Melrose Park, Illinois
  Served as project manager for the environmental site closure activties for large manufacturing facility. Known RECs included onsite industrial operations, former USTs, documented chlorinated solvent contamination at hazrdous levels. Field actvities included extensive soil and groundwater testing, in-situ remediation of chlorinated solvents to below hazardous levels, engineered barrier placement documentation, assissted in design and oversight of soil vapor mitigation system over a 25,000 square foot area. Responsibilites included project management, fieldwork scope development, technical meetings with client, and IEPA correspondance and technical report writing.

- Former Industrial Area – Des Plaines, Illinois
  Served as project manager for the environmental site closure activties of a 10-acre industrial area being redeveloped for single-family homes. Known onsite RECs included former asphalt plant, numerous chemical plating and industrial facilities, former USTs, former onsite railroad spurs, confirmed free product in soil and numerous compounds above the Illinois EPA Tier 1 Soil and Groundwater Remediation Objectives. Field actvities included extensive soil and groundwater testing, hot-spot remediation, engineered barrier placement documentation. Responsibilites included project management; fieldwork scope development; technical meetings with client and contractors; review of technical reports; oversight and management of numerous consultants and contracors; and IEPA correspondance; and technical report writing.

ERIN C. GRADY
ENVIRONMENTAL PROJECT MANAGER

PROFESSIONAL EXPERIENCE

Ms. Grady is responsible for environmental operations, technical oversight on all environmental projects as well as the coordination and distribution of projects to personnel. Ms. Grady trains and supervises a professional staff of environmental specialists. Responsibilities include, preparing and editing technical reports, writing proposals, cost estimates, tracking all office projects, client interaction, and creating project strategies to improve profitability and productivity.

Ms. Grady is also responsible for performing and managing Phase I Environmental Site Assessment (ESA) reports and Phase II Limited Site Investigation (LSI) reports of residential, commercial, industrial and telecom properties. She performs soil borings, monitored wells and well installation; and soil and groundwater sampling.

In addition, Ms. Grady has performed asbestos surveys, asbestos abatement oversight, air monitoring and sampling, hazardous materials surveys, and lead surveys. Ms. Grady also has experience in basic hazardous materials management principles including hazardous material storage and hazard assessments and communication. Ms. Grady has prepared over 150 Phase I ESAs, over 30 Limited Site Investigations, over 30 Transaction Screens, over 100 NEPA reports, quarterly groundwater monitoring reports, Discharge Monitoring Reports (DMRs), over 40 asbestos reports, and over 20 lead reports.

PROJECT EXPERIENCE

Phase I ESA Reports, Nevada, Illinois, & Wisconsin

Project Environmental Scientist during the preparation of Phase I ESA reports. Duties include review of physical settings, historical records and regulatory agency records, performance of a field reconnaissance and inspection for the site and surrounding properties, and preparation of reports including documentation to support the analysis, conclusions, and recommendations. Recommends for sites have included Phase II soil and groundwater sampling, removal of dumped soil, post removal soil testing, proper abandonment of water well, an asbestos survey be conducted, and a lead-based paint survey be conducted.

Telecom Phase I, Nevada, Illinois, & Wisconsin

Project Environmental Scientist for cell phone tower projects requiring Phase I ESAs, Transaction Screens, and asbestos surveys. Responsible for tracking each project and directing technical and clerical staff in all phases of projects. Duties include drafting proposals, review of physical settings, historical records and regulatory agency records, performance of a field reconnaissance and inspection for the site and surrounding properties, collecting asbestos samples, interviewing project contact, and preparation of reports including documentation to support the analysis, conclusions, and recommendations. Delivered final reports on schedule and within budget.

Education

Bachelor of Science, Environmental Studies & Applications, Michigan State University,

Certifications

40-Hour Hazwoper Training; 2007, Refresher; 2010

AHERA: Asbestos Inspector, Contractor/Supervisor Training; 2008

Work History

Terracon Consulting, Inc., Chicago, Illinois, Environmental Project Manager, 2010-Present

Trileaf Corporation, Bensenville, Illinois, Assistant Project Manager, 2009-2010

Terracon Consulting, Inc., Las Vegas, Nevada, Senior Environmental Scientist, 2006-2009

Senator Patricia Birkholz Office, Lansing, Michigan, Summer 2006,

Senate Majority Policy Office, Lansing, Michigan, Summer 2006

Terracon

Phase II LSI Reports, Nevada, Illinois, & Wisconsin
Project Environmental Scientist during the preparation of Phase II Limited Site Investigations. Duties include reviewing historical records and reports, collection of soil samples, soil boring and groundwater sampling, and preparing a report of the lab analysis results, conclusions and recommendations.

NEPA REPORT EXPERIENE

US Cellular, T-Mobile, & Clearwire, Illinois, & Wisconsin
Project Environmental Scientist for NEPA reports on telecom properties. Duties include tribal consultation, submitting proposed project to Federal Communications Commission (FCC's) Tower Construction Notification System (TCNS), preparing and submitting to the State Historic Preservation Office (SHPO) compliance letters which include a historical, architectural, and archeological review, posting public notifications in newspapers, preparing and notifying Certified Local Government (CLG) letters, reviewing and preparing flood and wetland maps, preparing and submitting letters to the Department of National Resources (DNR) and Fish and Wildlife Services, and preparing report findings.

SOIL AND GROUNDWATER REMEDIATION PROJECT EXPERIENCE

Soil Sampling, Nevada, Illinois, & Wisconsin
Project Environmental Scientist for Phase II soil sampling. Duties and projects include soil sampling around dry cleaning machines and areas of visible perc waste staining on floor, soil borings, and hand augering, and collection of soil from dumped soil piles for laboratory analysis. Prepared report of findings.

Groundwater Sampling, Nevada, Illinois, & Wisconsin
Project Environmental Scientist for Phase II groundwater sampling. Duties and projects include installation of multiple monitoring wells on a former gasoline station, collection of groundwater samples free product recovery, compilation of quarterly groundwater monitoring data, and prepared report of findings.

ASBESTSOS PROJECT EXPERIENCE

Asbestos Inspector, Nevada
Project Environmental Scientist for approximately 18 asbestos surveys for Albertsons Grocery stores. Provided project management and report preparation, performing pre-renovation asbestos surveys, and bulk sampling. Prepared all reports including documentation and maps to support the analysis, conclusions, and recommendations. Delivered all 18 final reports on schedule and within budget. Project Environmental Scientist for pre-renovation asbestos, lead-based paint, and hazardous materials survey report. Provided management and report preparation, collected over 1,500 bulk samples and over 1,400 lead-based paint samples. Prepared report and maps to support the analysis, conclusions, and recommendations.

Asbestos Contractor/Supervisor, Nevada
Pre-demolition asbestos abatement for an apartment building. Environmental Project Manager for the air monitoring and sampling, and contractor oversight and data compilation.

RONALD J. HORAN
ENVIRONMENTAL SCIENTIST

PROFESSIONAL EXPERIENCE

Ronald J. Horan is an Environmental Scientist in Terracon's Naperville, Illinois office. Responsibilities include preparing and editing technical reports, project management, technical file review, site investigations, proposal and bid preparation, and client development. Mr. Horan has gained a practical knowledge of CERCLA, RCRA, NEPA, LUST/UST/AST regulations, and SRP regulations, as well as ASTM standards as they relate to Phase I/II ESAs and Vapor Encroachment.

PROJECT EXPERIENCE

Phase I Environmental Site Assessment - Nationwide

Performed over 300 ASTM E1528-06 Environmental Transaction Screens, ASTM E1527-05 Phase I ESAs, and other environmental due diligence reports for properties nationwide. Duties include site investigation, regulatory records review, physical setting review, interviews, historical information review, and any additional services requested. Properties have included vacant, agricultural, retail, residential, gas stations, dry cleaners, industrial, and warehouse properties. Reports have been prepared for, but not limited to, the following: O'Reilly Automotive, Inc., Ozinga Concrete, Bridgestone-Firestone, Microsoft Corporation, USDA, Cricket Communications, US Cellular, Chick-fil-A, JPMorgan Chase, Bank of America, Imperial Capital Bank, Chevrolet, CLMG Corp., CB Richard Ellis, Inland, Kroger, Ashley Furniture, Ryan Companies, Sears, Truckomat Corp., Love's Travel Centers, Nissan, Emeritus, Wal-Mart, Autozone, M&I Bank, Endres, Northern Trust, Wells Fargo, Habitat for Humanity, Baymont Inn, Lifetime Fitness, Aldi, and several municipalities.

HUD / IHDA – Throughout the State of Illinois

Performed several HUD/IHDA due diligence reports, which include the preparation of HUD-4128 Forms and IHDA Environmental/Historic Preservation Checklists. These reports include technical file review of wetlands, floodplains, Coastal Management Zones, mining data, historic preservation documents, noise sources, threatened and endangered species, aquifers, Environmental Justice, and physical setting information. Preperation of vapor encroachment screens and HUD Noise Assessment calculations (with regards to roads, airports, and trains) were completed where necessary. Mr. Horan has personally worked with IHDA staff (including attending IHDA QAP Workshop) to ensure that reports are consistent with their specific needs. Specific IHDA/HUD funded projects worked on in the past three years include: Evergreen Senior Residences (Chicago), Johnston Garden Apartments (South Beloit), G&A Senior Residences (Chicago), Oswego Senior Residences (Oswego), Hebron Townhouse Apartments (Zion), Mary Allen West Tower (Galesburg), Cypress Creek Senior Living (Bolingbrook), Thomas Place (Gurnee), Sterling Townhomes (Sterling), The Rusnack Lofts (Chicago), Moline Live-Work Lofts (Moline), Quincy Newcomb (Quincy), Peoria Senior Residences (Peoria), Victory Center of Chicago (Chicago), and Chicago Veterans Home (Chicago), and three unnamed parcels (Round Lake Beach, Joliet, and Springfield).

Education
Bachelor of Science in Geology and Environmental Geoscience, Northern Illinois University, 2007

Work History
Terracon – Environmental Scientist, July 2007 to present

Certifications / Seminars
ASTM Phase I Environmental Site Assessment Practices for Commercial Real Estate Seminar and Certification

ASTM Vapor Encroachment Screen Seminar and Certification

American Heart Association CPR, First Aid, and AED Training

EPA / AHERA Accredited Asbestos Building Inspector

OSHA 40-Hour HAZWOPER Training, including Confined Space Entry

EDR Due Diligence at Dawn Seminar (2010)

Security Clearance for FDIC

Licenses
Licensed Asbestos Building Inspector: State of Indiana, State of Illinois, State of Michigan

Affiliations
ASTM International

National Association of Environmental Professionals – Illinois Chapter

Association of Environmental and Engineering Geologists

Terracon

Phase II / LSI / SWPPP / CCDD – Throughout the Great Lakes Region

Performed soil and groundwater sampling in conjuction with a variety of site investigations. Soil sampling has included hand augering, composite soil sampling, collection of soil from dumped soil piles, and working with drill crews. Groundwater sampling has included pumping and manual baling from previously installed monitoring wells, measuring depths to groundwater, well surveys, and obtaining drinking water samples from open pipes. Additionally, has abandoned wells in accordance with state and local regulations. Technical reports and tables have been prepared to determine whether contamination exceeded state-mandated contamination levels. LSI work has also included GPR surveys, which consists of identifying subsurface features, including USTs, through the use of radar. Assisted in coordinating and performing SWPPP inspections in accordance with specific NPDES permits. Provided air monitoring at construction sites for worker safety concerns. Coordinated with CCDD facilities to obtain certifications of clean material removal.

NEPA (EA) – Throughout the Great Lakes Region

Prepared over 75 NEPA-related reports including: categorical exclusion letters, desktop reviews, new tower/collocation NEPA reports, and EAs. NEPA reports include indian tribe consultation using the FCC's TCNS, SHPO consultation, USFWS Section 7 consultation, historic district consultation, and Certified Local Government consultation, as well as technical file review of wetlands, floodplains, Coastal Management Zones, mining data, historic preservation documents, archaeological resource documents, noise sources, threatened and endangered species, aquifers, Environmental Justice, and physical setting information. Mr. Horan has a strong working relationship with the Illinois SHPO and various tribal entities to help expedite NEPA-related reports under his direction. NEPA-related work has been performed for Cricket Communications (during its Chicago buildout), Illinois Valley Cellular, the Veterans Administration (VA), FEMA, and as a portion of IHDA/HUD requirements.

Industrial Hygiene – Nationwide

Performed, managed, and wrote indoor air quality (IAQ) assessments, mold assessments, asbestos surveys, lead-based paint surveys, noise assessments, and radon surveys throughout the country. Responsibilities have included asbestos bulk sample collection, deployment of air quality equipment (including PID, Q-Trak, Multi-Rae, Interscan 4160 Meter, TSI Dust Track 8520, Ozone Aeroqual Series 500 Monitor, etc.), mold tape and air sampling, lead-paint XRF testing, completion of HUD Noise Assessment calculations, and radon kit deployment. Mr. Horan has Asbestos Building Inspector Licenses for the States of Illinois, Indiana, and Michigan and has received certification to use XRF testing equipment.

FDIC – Michigan, Illinois, and Wisconsin

Performed over 75 FDIC Checklists and FDIC Phase I ESAs for properties involved in bank closures. Properties primarily were occupied by residential and commercial entitities. Mr. Horan has obtained Security Clearance from the FDIC to perform FDIC-related environmental work. FDIC Phase I ESAs involve asbestos, radon, lead in drinking water, lead-based paint, threatened and endangered species, wetland, Coastal Barrier Improvement Act, floodplain, "Area of Scientific Significance", natural landmark, aquifer, historic property, and archaeological resource components.

Vapor Encroachment Screens – Chicagoland Area

Performed ASTM E2600-08 and E2600-10 vapor encroacment screens on properties in conjuction with several Phase I ESAs. Vapor encroachment screens involve regulatory file review to determine facilities located within ASTM E2600-10 agreed upon boundaries, and then determining whether the facilities pose a vapor threat to the property. Mr. Horan has attended the Vapor Encroachment Screen seminar provided by ASTM and is included on their list of providers who have obtained ASTM Vapor Encroachment Screen certification.

SARA M. LOCKE
FIELD ENVIRONMENTAL SCIENTIST

PROFESSIONAL EXPERIENCE
Ms. Locke is based in Terracon's Chicago, Illinois office. She is responsible for conducting to Phase I Environmental Site Assessments (ESAs) for residential, commercial and industrial properties. Her duties include preparing technical reports, data collection, client interaction, and site reconnaissance.

In addition, Ms. Locke has experience in groundwater sampling, surface water sampling, stream cross-sections and habitat surveys. She has assisted in the completion of several Phase I ESAs and has participated in multiple site visits.

PROJECT EXPERIENCE
Potential Ashley Furniture Store – Broadview, IL
Ms. Locke conducted a Phase I ESA on a shopping center that was a former industrial facility with former underground storage tanks (USTs) and aboveground storage tanks (ASTs).

O'Reilly Auto Parts – Imlay City, MI
Conducted a Phase I ESA on a vacant lot with adjacent leaking underground storage tank (LUST) facility.

MetLife Agricultural Investments (Auburn Bean & Grain Company Sites) – Hemlock, MI
Conducted a Phase I ESAs on six agricultural fertilizer and bean storage facilities, along with historical and regulatory research and the accompanying report.

Road Ranger, LLC – Belvidere, IL
Conducted a Phase I ESA on a former residential home and a restaurant with adjacent gasoline station and other nearby environmental concerns

Education
M.S., Water Resource Management, 2010, University of Wisconsin, Madison

B.A., English and Sociology, University of Wisconsin-Whitewater, Wisconsin

Work History
Terracon Consultants, Inc., Chicago, Illinois
Field Environmental Scientist
2010-present

University of Wisconsin, Madison, Wisconsin
Practicum Researcher
2008-2009

Kishwauketoe Nature Conservancy Williams Bay, Wisconsin,
Field Operations Assistant
Summer / Fall 2007

Terracon

J. DAVID MOON
SENIOR ENVIRONMENTAL PROJECT MANAGER

PROFESSIONAL EXPERIENCE

Mr. Moon is a Senior Environmental Project Manager for Terracon's Naperville and Rockford, Illinois offices. His responsibilities include managing/completing Phase I Environmental Site Assessments (ESAs), Wetland Delineations/Permitting, NEPA Environmental Assessments, Limited Site Investigations, Focused Site Investigations, Leaking Underground Storage Tank (LUST) and voluntary Site Remediation Program (SRP) projects. Mr. Moon is responsible for proposal preparation, project management, staff management, and coordination of investigative and remedial field work as well as the peer review of reports and proposals.

Mr. Moon recently returned to Terracon from JPMorgan Chase where he was an Environmental Risk Administrator (ERA). As an ERA, he reviewed environmental due diligence for loans that JPMChase took land as collateral, deals where environmental risk is likely present based on industry type/historical operations, foreclosures, and deals where JPMChase was taking an equity interest in a deal/property. Mr. Moon was responsible for understanding the loan structure, developing the appropriate level of due diligence, engaging consultants, summarizing the findings/conclusions for the banking/credit team. He assisted bankers and credit officers with developing risk mitigation strategies that included additional site assessment, remediation, reserve/escrow accounts, terms/conditions of loan agreements, and review of insurance policies prior to making a loan on contaminated property. Sites that were contaminated required his oversight during the remediation planning, execution, and regulatory oversight. He led conference calls with the bankers, credit officers, borrowers, construction management team, and government officials to devise environmental risk mitigation strategies and regulatory requirements. He was also responsible for the review of construction loan draw requests that included environmental investigation/remediation fees.

PROJECT EXPERIENCE

Health Resources and Services Administration

Prepared two Environmental Assessments (EAs) in accordance with the National Environmental Protection Act. The first EA was comlpeted on a 100 year old existing facility that was being renovated, modernized, and included expanded parking capacity. The second EA was for the construction of a new health clinic. Each EA was completed in a timely fashion and ending with a Finding of No Significant Impact determination.

Veterans Affairs Expansion

Completed Threatened and Endangered Species, Historic Preservation, Wetlands, Environmental Site Assessment, Environmental Justice evaluations for an EA prior to completing renovations/expansions to a Veteran Affairs (VA) Hospital in Joliet, Illinois.

Education

Bachelor of Environmental Science, 1996, Purdue University.

Certification

OSHA 40-Hour HAZWOPER

National Highway Institute NEPA and the Transportation Decision Making Process

USACE 38 Hour Wetland Delineation and Management Training

American Society for Testing and Materials Phase II Environmental Site Assessment Process, 2004

Illinois Licensed Asbestos Building Inspector

Indiana Licensed Asbestos Building Inspector

Work History

Terracon, Environmental Department Manager, 2009 to the Present

JPMorgan Chase, Environmental Risk Administrator, 2005 – 2009

Terracon, Project Manager, 1999-2005

Terracon, Project Scientist, 1997-1999

Soil Probe, Field Technician, 1996-1997.

Terracon

DuPage County Illinois Stormwater Management Permit
Prepared a wetland delineation, joint 404/401 permit application, and a Tab4 Stormwater Management Permit allication for a residential development in DuPage County Illinois. These permit applications included Wildlife Quality Assessment, US Fish and Wildlife Service endangered species evaluation, designed planting/seeding mitigation, and alternative analysis options.

Residential/Commerical Development
Reviewed multiple Phase I and Phase II reports, developed subsurface investigation scopes of work, designed vapor intrusion assessment for a college student housing/retail shopping center with seven historical and one active dry cleaner.

Ottawa, Illinois – Proposed Commercial Development
Conducted a wetland delineation of an 88 acre site and obtained 404/401 Permits to fill 0.43 acres of wetland at the site. Off-site mitigation was selected as the most beneficial form of mitigation to the environment and for the client.

Wetland Water Quality Certification – Hartford, Wisconsin
Conducted a wetland delineation and prepared a Wetland Water Quality Certification. This included meetings with the Wisconsin Department of Natural Resources, the developer, and interaction with the local government to design a site plan that met the involved parties desires through through a series of negoitations.

Telecommunincations Tower Buildout, Illinois-Indiana
Project manager for a cell tower buildout in the Illinois and Indiana reigion. Services included in this contract were Phase I ESAs, NEPA screens, Section 106 Submittals, Area of Potential Effect studies, Photosimulation Studies, and Wetland delineations.

Brownfield Redevelopment – New York, New York
Served as the ERA on a two city block brownfield site that included the review of Phase I, Phase II, Remedial Action Plans/Budgets. Led conference calls with the NY State Department of Environmental Conservation, NY City Department of Environemntal Protection, contractors, and the banking team.

Residential/Commerical Development
Reviewed multiple Phase I and Phase II reports, developed subsurface investigation scopes of work, designed vapor intrusion assessment for a college student housing/retail shopping center with seven historical and one active dry cleaner.

Managed Truck Transfer Facilities Phase I ESA Portfolio
Prepared the bid package, assembled the team to complete the ESAs in eight different states across the country, and provided guidance to staff completing the individual ESAs. The reports for these complex sites were delivered in a timely fashion to the client.

Nine City Block Site in Chicago, Illinois
Assisted in the preparation of proposed scope of work and cost estimate for this job. Assembled the team to complete the ESA which had several scope limitations and exceptions to the ASTM Standard E-1527 with the goal of providing a detailed screening tool of a large area to identify potential problems areas that will require additional investiation at a latter date.

Range Characterization – Indiana Dunes National Lakeshore
Characterized impacts to soil and groundwater at an active Federal small arms firing range and at two inactive historical small arms firing ranges. Prepared an engineering evaluation and cost analysis for one of the small arms firing ranges in accordance with CERCLA requirements.

Former Metal Plating Facility Remediation – Chicago, Illinois
Project Manager for the investigation and remediation of a former metals plating facility, including stabilization of heavy metals impacted soil to levels below hazardous concentrations.

Aldi Facilities in Chicagoland
Conducted Phase I/Phase II ESAs, Focused Site Investigations, Remedial Objectives Reports, Remedial Action Plans, UST removals, and remedial excavations at numerous sites in the Chicagoland area.

Kinder Morgan Liquid Terminals – Argo, Illinois
Project Manager for remediation of a Number 6 Oil release, numerous focused site investigations, and waste characterization projects.

Dry Cleaners Remediation Site – Naperville, Illinois
Assisted with the design, installation, and operation of a soil vapor extraction system remediating a perchloroethylene release from a former dry cleaning operation. The project was successful in reducing perchloroethylene concentrations from free product levels to concentrations that satisfied requirements outlined in the IAC 742 that will permit impacted soil to remain on-site beneath engineered barriers.

THOMAS R. TUCKER, JR.
ENVIRONMENTAL PROJECT SCIENTIST

PROFESSIONAL EXPERIENCE

Mr. Thomas R. Tucker is a Project Scientist in Illinois. He has over a year of experience in the environmental consulting industry. Mr. Tucker has successfully completed numerous Phase I Environmental Site Assessments for property transactions. Mr. Tucker's experience includes ASTM 1527-00 and 1527-05 Phase I ESAs, several national portfolio projects, asbestos inspections and wetland delineations. Coursework has included soil taxonomy, identification, and classification. Project sites have covered agricultural land, multi-tenant residential buildings, and commercial and industrial facilities located throughout Michigan, Indiana, Illinois, Maine, Connecticut, and New Hampshire.

PROJECT EXPERIENCE

Dearborn Wholesale Grocers - Chicago, Illinois

Conducted sub-surface site investigation soil boring work consisting of soil and groundwater sampling for a range of known contaminants. Responsibilites also screening of soil vapors, collection and preservation of soil samples, soil core logging, groundwater depth determination, soil boring layout, boring log development, hydrailic conductivity testing, and surveying. The project also involved subcontractor coordination and writing technical investigation reports that adhered to Illinois Environmental Protection Agency (IEPA) and the Chicago Department of Environment (CDOE) remediation standards.

Public Building Commission of Chicago – Chicago, IL

Conducted focused and supplemental site investigation soil boring work consisting of soil sampling for a range of known contaminants in accordance with the IEPA Site Remediation Program. Known RECs included underground storage tanks, PNA, and metal impacted soil. Site investigation activities also included ground-penetrating radar, test trenching, and asbestos containing material surveys. Responsibilities included soil core logging, soil boring layout, boring log development, modelling of observed impact, oversight of building demolition, oversight of soil removal, backfill clearance, technical review meetings, and regular status meetings. Site is part of on-going redevelopment work of the former manufacturing facility.

ConAgra Portfolio – Elmhurst, Illinois

Performed a Phase I ESA involving a site walkthrough, sourcing relevant environmental information from local authorities and investigation of apparent environmental concerns.

Comerica Portfolio – Southern Illinois

Performed the completion of several Phase I ESAs. Projects involved site walkthrough, sourcing relevant environmental information from local authorities and investigation of apparent environmental concerns.

Education

Bachelor of Science in Natural Resources and Environmental Sciences at the University of Illinois, 2006.

Registrations

EPA / AHERA Accredited Asbestos Building Inspector

Licensed Asbestos Building Inspector, State of Illinois Department of Public Health

Licensed Asbestos Building Inspector, State of Indiana, Indiana Department of Environmental Management

Certifications

Asbestos Building Inspector Occupational Training and Supply 2006

Refresher Class: Occupational Training and Supply, 2008

OSHA 40 Hr. HAZWOPER & 10 Hr. General Industry Safety & Health Occupational Training and Supply 2007

ASTM Phase I Environmental Site Assessment Practices for Commercial Relestate Seminar

Affiliations

ASTM International

Work History

Terracon, Illinois – Professional I, March 2006 to present

University of Illinois – Water and Soil Quailty Laboratory – Laboratory Assistant, April 2005 – March 2006

Terracon

Auto Wares Group - Chicago, Illinois
Responsible for the completion of Phase I ESA. Project involved a site walkthrough, sourcing relevant environmental information from local authorities and investigation of apparent environmental concerns.

Patriot Renewable Fuels - Annawan, Illinois
Performed an Army Core Of Engineers regulated wetland delineation. Project involved a field investigation, hydric soil determinations, and a site layout.

NSORO Cingular Portfolio - Nationwide, South Texas
Performed desktop NEPA reviews for cellular phone tower sites as part of Cingular's nationwide cellular tower acquisition program. Project involved tower registration seaches, coordination with State Historic Preservation Officers, Native American database searches, sourcing relevant environmental information from the National Wetland Inventory and floodplain maps to determine necessity of Environmental Assessments.

JP Morgan Chase, Road Ranger Portfolio - Indiana
Performed Phase I ESAs. Project involved a site investigation, Illinois Environmental Protection Agency (IEPA) file review, sourcing relevant environmental information from local authorities and investigation of apparent environmental concerns.

Waubonsee Community College - Aurora, Illinois
Assisted in the planning and performance of asbestos and hazardous materials inspections in over nine commercial and residential buildings. Final inspection report included locations of ACBM, hazardous materials, and response recommendations with cost estimates.

La Quinta Portfolio -Stamford and New Haven, Connecticut
Assisted in the planning and performance of a limited asbestos inspection in two multi-story Fairfield Inn hotels. Final inspection report included locations of ACBM and recommendations with cost estimates. Also performed Phase I environmental site assessment, limited radon survey, and coordinated a limited lead based paint survey of the properties.

MARK R. WILSON
PROJECT ENGINEER

PROFESSIONAL EXPERIENCE

Mr. Wilson is a Project Engineer in Terracon's Naperville, Illinois office. Mr. Wilson has 18 years of industry experience, including environmental remediation and compliance management and reporting for projects under RCRA, CAA, and CWA. He has been responsible for management of data collection, data evaluation, hazardous materials management, contractor oversight, and preparation of technical documents.

His responsibilities include remedial investigations, execution of Phase I and Phase II Environmental Site Assessments, remedial design and planning, remediation and preparation of LUST Closure reports.

PROJECT EXPERIENCE

Whitney Young Public Library Expansion- Chicago, Illinois

Mr. Wilson prepared a Site Investigation Report/Remedial Objectives Report/Remedial Action Plan which has been approved by IEPA for remediation of chlorinated solvents at the site. His responsibilities include remedial design and planning, cost estimations, preparation of bid specification, contractor management, correspondense with Illinois EPA and Illinois Office of Brownfields Assessments.

Former Select Cleaners

Mr. Wilson conducted site investigations and prepared a Site Investigation Reports/Remedial Objectives Report/ Remedial Action Plan which received IEPA approval in the Site Remediation Program. The site is a former dry cleaning facility with soil impacted by tetrachloroethylene. Mr. Wilson is providing oversight of in-situ remediation using chemical oxidation with a soil mixing procedure.

RCRA Corrective Action, Former Texaco Refinery – Lockport, Illinois

Provided technical oversight of regulatory compliance with RCRA, CAA and NPDES permits. The project involved remedial and corrective action activities at a 580 acre former refinery operating under a Part B RCRA Permit. Mr. Wilson conducted environmental investigations following Illinois TACO guidance. As Technical Services Manager, his responsibilities included supervising field technicians, overseeing and documenting assessment and corrective action activities, preparing environmental investigation and routine monitoring reports, managing, evaluating, and validating data, and providing contractor oversight. Mr. Wilson also trained and mentored employees on a behavior based Loss Prevention System, managed GPS survey data collection, and developed a construction and operating permit for a mobile concrete crushing unit.

City of Elgin Brownfields – Elgin, Illinois

Mr. Wilson conducted remedial investigations and prepared Site Investigation Reports, Remedial Objectives Reports, Remedial Action Plans, and Remedial Action Completion Reports for several brownfields sites as part of downtown redevelopment efforts for the City. His responsibilities included soil and groundwater investigations, remedial

Education

Master of Science, Environmental Engineering, 2003, Illinois Institute of Technology, Chicago, Illinois

Bachelor of Science, Environmental Biology, 1992, Eastern Illinois University, Charleston,Iillinois

Certifications

OSHA 40-Hour Hazardous Waste Operations and Emergency Response

American Heart Association, Heartsaver CPR, 2006

American Red Cross, Standard First Aid, 2006

Work History

Trihydro Corporation, Environmental Engineer, 2002-2006

TestAmerica, Incorporated, Project Manager, 2000-2002

Environmental Monitoring and Technologies, Inc., Groundwater Department Manager, 1992-2000

EA Science and Engineering, Field Technician, 1992

planning, remedial oversight, and preparation of reports for the Illinois voluntary Site Remediation Program.

CITGO Petroleum Corporation – Lemont, Illinois
Prepared a quality assurance plan and sampling and analysis plan for the groundwater monitoring program. His responsibilities included managing groundwater monitoring activities and BWON sampling activities, evaluating data, and preparing routine monitoring reports. Mr. Wilson also conducted analytical laboratory audits for CITGO.

Environmental Monitoring, Multiple Solid Waste Disposal Facilities – Illinois and Midwest
Management of groundwater monitoring and landfill gas migration monitoring at over 30 facilities. Responsibilities included managing field crews, coordinating with site engineers, managing laboratory analysis, evaluating data, and preparing reports. Also conducted point source air emissions monitoring at several landfill gas recovery plants to demonstrate compliance with federally enforceable state operating permits.

Waubonsee Community College – Downtown Aurora Campus
Mr. Wilson prepared the Site Investigation Report/Remedial Objectives Report/Remedial Action Plan for the redevelopment project. Mr. Wilson works with the IEPA Project Manager to advance the project through the Illinois Site Remediation Program. Mr. Wilson also prepared the bidding documents, plans, and specifications for the remediation work to be conducted at the site.

Wastewater and Stormwater Studies, Multiple Industrial Facilities – Chicago, Illinois
Conducted waste water treatment and effluent flow studies. As a technician, he installed flumes, weirs, and automated sampling equipment for effluent compliance monitoring. Also conducted industrial storm water run-off studies at several facilities to demonstrate compliance with storm water permits.

Iron Mountain Paper Mill – Michigan
Conducted bioassay study to assess the impact of papermill effluent on Walley Pike in the Minomonee River. Responsibilities included system construction, maintenance, and water quality data collection.

Chevron Former Indian Oil Refinery – Lawrenceville, Illinois
Conducted electromagnetic geophysical surveys to locate underground piping corridors. Responsibilities included data management and processing; generated site maps and identified targets for excavation in the field.

Appendix C

APPENDIX C
TABLE OF TERRACON STANDARD OPERATING PROCEDURES

REFERENCE NO.		TITLE	LAST REVISED OR REVIEWED
E.20		Standard Safe Operating Procedures for Hazardous Waste Operations	June 2010
E.30		Chain of Custody Documentation	June 2010
E.50		Sampling – Environmental Representativeness	June 2010
	E.1XX	Soil Sampling	
	E.100	Surface & Near Surface Soil Sampling – Grab	June 2010
	E.150/155	Soil Sampling – Volatiles By TerraCore™	August 2011
E.300		Sampling & Drilling Platforms	June 2010
E.400		Subsurface Sampling – Geoprobe© Platform	August 2011
	E.4XX	Sample Handling	
	E.468	Sample Handling – Soil (Level D)	June 2010
	E.470	Sample Handling – Groundwater (Non-Hazardous)	June 2010
	E.480	Surface Water Sampling	August 2011
E.5XX		Field Screening	
E.500		pH Field Screening – Soil	June 2010
E.530		pH Field Screening – Water	June 2010
E.540		Conductivity Field Screening – Water	June 2010
E.550		Field Surface Screening – Soil / Photoionization Detector	August 2011
E.552		Field Headspace Screening – Soil / Photoionization Detector	August 2011
E.554		Field Screening – Air / Photoionization Detector	August 2011
E.570		Temperature Field Screening	June 2010
E.580		Turbidity Field Screening	June 2010
	E.700	Well Construction – Temporary	June 2010
	E.800	Well Construction – Permanent	June 2010
E.9XX		Well Security	
E.900		Well Security – Type A (Simple Cap)	June 2010
E.905		Well Security – Type B (Locking Expansion)	June 2010
E.910		Well Security – Type B (Protective Casing)	June 2010
E.920		Well Security – Type C (Flush Mount)	June 2010
	E.1300	Well Development – Volumetric	June 2010
	E.1400	Well Development – Parametric	June 2010
	E.1500	Boring Abandonment – Commercial Sealant	June 2010
E.18XX		Physical Field Measurements	June 2010
E.1800		Field Measurement – Surface Layout	June 2010
E.1805		Field Measurement – Elevations	June 2010
E.1810		Field Measurement – Subsurface Soils	June 2010
E.1820		Field Measurement – Groundwater	June 2010
E.1830		Field Measurement - Free-Phase Product	June 2010
E.1840		Field Measurement – Hydraulic Conductivity Testing (Slug)	June 2010
	E.1900	Groundwater Sampling – Bailer	August 2011
	E.2000	Groundwater Sampling – Low Flow Pumping	August 2011
E.22XX		Site Housekeeping	
E.2210		General	June 2010
E.2220		Disposal of Spent Supplies	June 2010
E.2230		Handling and Storage of Drill Cuttings (Non-Hazardous)	June 2010
E.2235		Handling and Storage of Drill Cuttings (Hazardous)	June 2010
E.2240		Site Security Procedures	June 2010
	E.24XX	Cleaning & Decontamination	June 2010
	E.2405	Cleaning - General	June 2010
	E.2410	Cleaning - Manual Washing	June 2010
	E.2420	Cleaning - High-Pressure, Hot-water Washing	June 2010
E.3000		Bulk Sampling of Suspect Asbestos-Containing Material (ACM)	September 2011

E.4000		Sampling of Potential Lead-Based Paint	September 2011

STANDARD OPERATING PROCEDURE

E.100
SURFACE & NEAR-SURFACE SOIL SAMPLING - GRAB

LAST REVIEW OR REVISION: August 2011

OBJECTIVE AND APPLICATION

To provide standard procedure for collecting surface and near-surface soil samples appropriate to project conditions.

Surface soil/tailings/spoils samples are collected from the surface to a depth of approximately four (~4) inches. Near-surface soil/tailings/spoils samples are collected from the surface to a depth of approximately four to twelve (~4-12) inches from original surface. The original surface may be ground surface or the exposed horizontal or vertical surface of material excavated in mass from the subsurface.

Grab sampling is appropriate to conditions and projects where the end use of the sample is not overly sensitive to disturbance and handling in the course of collection. Grab sampling should not be applied where the sample is used for field or laboratory measurements of low levels of readily volatile organic compounds. Grab sampling is appropriate for general field screening and as a field guide to directing environmental excavation, if compatible with the physical properties of the chemical(s) to be measured. Grab sampling should not be used for samples intended to represent in-situ, undisturbed subsurface conditions.

Sufficient sample will be collected for the analysis that will be performed as prescribed by the project documents. Soil descriptions will be completed for each collected soil sample using the general terminology of the unified soil classification system (ASTM D2487). Descriptions shall be recorded in field books.

EQUIPMENT

- Rigid sampling equipment such as a trowel or shovel of inert material relative to the chemical(s) of concern and capable of reaching the depths prescribed for surface and near-surface soils.
- Disposable gloves.
- Chemical-resistant work gloves.
- Laboratory prepared sample containers.
- Roll of plastic sheeting.
- Plastic trash bag for collecting expended supplies.
- Field documentation forms or project logbook.

- Chain-of-Custody forms for samples intended for laboratory analysis.
- Marking pencils or indelible markers that will not leave residues which can cause interference with laboratory testing procedures.

PROCEDURES

Select the location. Identify it with a unique designation for the project. Diagram or otherwise describe the location on forms or in the logbook relative to a fixed benchmark that will allow the specific location to be re-visited in the future, if necessary. If appropriate, estimate the vertical elevation of the sample and record.

If the grab sample is constructed of multiple aliquots to represent averaging of conditions in soils/tailings/spoils, diagram or otherwise describe the area represented by the constructed sample. Diagram or otherwise accurately describe sub-sample locations on forms or in the logbook relative to a fixed benchmark that will allow the specific sub-sample locations to be re-visited in the future, if necessary. If appropriate, estimate the vertical elevation of the sub-samples and record.

At each location before collecting the soil sample, put on a clean pair of disposable chemical-resistant gloves.

Collect each sample by hand using procedures and equipment/tools specified by the project manager.

Place the sample directly into the laboratory prepared sample container(s) and complete the sample label and Chain-of-Custody as instructed by the project manager.

If intended for laboratory analysis, preserve the sample as required by project plans. Samples collected for analysis of volatiles should be collected first with minimal disturbance to the sample (refer to SOP E.150/155). The remainder of the soil sample should be mixed until homogenous and placed into the required sample containers.

ATTACHED REFERENCES

- **ASTM E1903-97** *Standard Guide for Environmental Site Assessments: Phase II Environmental Site Assessment Process*.

OTHER SUPPORTING DOCUMENTS

- **ASTM D2487-00** *Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System)*.

STANDARD OPERATING PROCEDURE

E.1300
WELL DEVELOPMENT – VOLUMETRIC

Last Review or Revision:June 2010

Objective

The objective of monitoring well development is to remove adulterated water in the well, sand pack, and surrounding saturated formations that formed during drilling and well installation. Development should be considered the final step in completing a groundwater monitoring well.

Initially, well development should purge any drilling fluids or other foreign materials from the monitoring well. Development should be performed as soon as practical after the well installation, but no sooner than 48 hours after the grouting is complete.

Equipment

Development should be accomplished with the use of a pump, bottom filling bailer, or nitrogen lift device. Measuring equipment should be consistent with that specified by the project Manager or Equipment Manager at mobilization and consistent with TSOP E.1820.

Protective safety equipment will as specified in the program of assessment and as directed by the Terracon Site Health & Safety Plan consistent with materials and concentrations specified therein.

Procedure

- Determine the geometry of the monitoring well (i.e., measure static water level, depth of well, determine volume of fluid lost while drilling.) See attached form.

- Determine the volume of one "standing volume" of water in the well, defined as the volume of standing water within the well casing plus the volume within the gravel pack (assuming 30% porosity).

- For monitoring wells where the boring was made without the use of drilling fluids, three (3x) times the standing water volume in the well should be removed.

- For those wells where the boring was advanced using drilling fluids, remove five (5x) times the standing volume plus three (3x) times the volume of water lost during drilling.

- If low permeability materials prevent the well from yielding adequate volumes of water, alternative development procedures will be implemented at the direction of the Project Manager (i.e. removal of fluids to dryness, allow well to recharge and then recheck quality of fluids).

- Complete the monitoring well development form if supplied by the Project Manager or record well development details in the field log book. Pertinent data will vary based on the parameters and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

- Store or discharge the development water consistent with direction of the Project Manager and as allowed by law. Reference E.2220.

Attached Supporting Documentation

- **Form 130** Log of Boring No., Monitoring Well Details, and Boring Elevations

- Terracon Volumetric Chart for Well Development

- Brainard Kilman Field Facts

Other References

a) ASTM D4448 Standard Guide For Sampling Groundwater Monitoring Wells
b) TSOP E.1900 Groundwater Sampling – Bailer
c) TSOP E.2000 Low Flow Pumping

STANDARD OPERATING PROCEDURE

E.1400
WELL DEVELOPMENT – PARAMETRIC

Last Review or Revision: June 2010

1. OBJECTIVE

The objective of monitoring well development is to remove adulterated water in the well, sand pack, and surrounding saturated formations which formed during drilling and well installation. Development should be considered the final step in completing a groundwater monitoring well.

Initially, well development should purge any drilling fluids or other foreign materials from the monitoring well. Development should be performed as soon as practical after the well installation, but no sooner than 48 hours after the grouting is complete.

Parametric stabilization is necessary when the groundwater may not be “clear” due to macroscopic particulate movement in the soil/fill matrix. This is not uncommon in old industrial fills containing flyash or carbon black. Parametric attempts to identify that the condition of physical parameters in formation water entering the well is reasonably comparable to water in the adjacent formation.

1. EQUIPMENT

Development should be accomplished with the use of a pump, bottom filling bailer, or nitrogen lift device. Measuring equipment should be consistent with that specified by the project Manager or Equipment Manager at mobilization and consistent with TSOP E.1820.

Protective safety equipment will be utilized as specified in the program of assessment and as directed by the Terracon Site Health & Safety Plan consistent with materials and concentrations specified therein.

2. PROCEDURE

- Determine the geometry of the monitoring well (i.e., measure static water level, depth of well, determine volume of fluid lost while drilling.) See attached form.

- Determine the volume of one "standing volume" of water in the well, defined as the volume of standing water within the well casing plus the volume within the gravel pack (assuming 30% porosity).

- Sample and record a baseline series of measurements for water in the well casing for;
  - Temperature; degrees Fahrenheit (F)
  - pH; standard units
  - Specific Conductance; micromhos (μmhos)
  - Turbidity; nephelometric turbidity units (NTUs)

- Remove three (3) additional standing volumes from the well, recording a measurement series at the end of each volume.

- If each of the parameters have demonstrated repeatability for three (3) consecutive readings the well will be considered developed comparable to formation water conditions;
  - Temperature; ± 1 degree F
  - pH; ± 0.3 standard units
  - Specific Conductance; ± 20 μmhos
  - Turbidity; ± 10 NTUs

- If parameters for each have not stabilized, continue to remove and measure non-stabilized parameters each additional standing volume, up to a total of ten (≤10x) the initial standing well volume. At this point the well will be considered reasonably comparable in condition to conditions of the surrounding formation water.

- For those wells where the boring was advanced using drilling fluids, remove the volume of water lost during drilling plus the one standing volume prior to taking the baseline measurement series.

- If low permeability materials prevent the well from yielding adequate volumes of water, alternative development procedures using TSOP E.2000 will be implemented at the request of the Project Manager.

- Complete the monitoring well development form.

- Store or discharge the development waters consistent with direction of the project Manager and as allowed by law. Reference E.2220.

4. ATTACHED SUPPORTING DOCUMENTATION

- Brainard Kilman Field Facts

5. OTHER REFERENCES

a) ASTM D4448 Standard Guide For Sampling Groundwater Monitoring Wells
b) TSOP E.1900 Groundwater Sampling – Bailer

c) TSOP E.2000 Low Flow Pumping
d) TSOP E.530 pH Field Screening – Water
e) TSOP E.540 Conductivity Field Screening – Water
f) TSOP E.570 Temperature
g) TSOP E.580 Turbidity Field Screening - Water

Terracon

STANDARD OPERATING PROCEDURE

E.150/155
SOIL SAMPLING
Volatiles / Terra Core™

Last Review or Revision: August 2011

Objective and Application:
To provide standard procedure for sample collection for analysis of volatile organic compounds. The methods will provide representative samples for laboratory analysis using EPA Standard Method SW-846 5035.

This application uses proprietary commercial equipment and materials which will be purchased from an authorized vendor.

Equipment:

- Commercial Terra Core™ Kits, each to include:
  - Disposable Terra Core™ Samplers
  - Tared 40-milliliter (ml) Volatile Organic Analysis (VOA) vials containing 5 mls of sodium bisulfate solution and integral stir bars
  - Tared 40-ml VOA vials containing methanol
  - Two ounce (or larger) dry weight jars with lids (unpreserved)
  - Zippered plastic bags
- Disposable chemical resistant gloves.
- Chain of Custody.
- Stable field platform to support test kit, screened from wind and elements.

Procedures:
Set up the working platform on a stable surface or in the field vehicle remote from fuel, exhaust or other contaminant sources. Cover the platform and secure with disposable plastic sheeting.

At each location prior to collecting the soil sample, put on a clean pair of disposable, chemical-resistant gloves. For each sample to be collected, soil must be deposited into 2 VOAs vials each containing 5 mls of sodium bisulfate (alternatively, a trisodium phosphate preservative is used in some states) solution and integral stir bars, 1 VOA vial containing methanol, and 1 unpreserved dry weight jar. With the plunger seated in

the handle, push the Terra Core™ into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 grams of soil.

Wipe all soil or debris from the outside of the Terra Core™ sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.

Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the tared 40ml VOA vial containing the appropriate preservative, and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial. When capping the 40ml VOA vial, be sure to remove any soil or debris from the threads of the vial. Perform this same operation for all required VOA vials per sample. Next, fill the unpreserved dry weight jar with representative soil to the top of the jar and cap the jar.

Label each sample container with a non-solvent-based permanent marker. Place in the zippered bags provided.

Pack well in the kit box provided by the manufacturer. Place the kit box in a large capacity Ziplock® freezer bag and seal. Avoid use of tape and a plastic bag as this method deals specifically with low-level volatile testing.

Place in the cooler filled with ice and pack suitable for overnight courier shipping.

Other Supporting Documents :

- **TerraCore™ Internet Website at http://www.ennovativetech.com/TC_sampler_kit.htm**
  En Novative Technologies, Inc.
  Phone:(920)465-3960
  Fax: (920)465-3963
  Toll Free: 888-411-0757
  Procedures by En Novative Technologies, Inc. 1999-2000, Modified: March 13, 2001

- **Alternative EPA Method 5035 Sampling as EnCore**

- **USEPA SW-846 Method 5035 For Organic Analytes Using Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples**
  Test methods for Evaluating Solid Waste – Volume I, EPA Document #SW-846, United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Section B, Chapter Four.

Terracon
STANDARD OPERATING PROCEDURE

E.1500
BORING ABANDONMENT – COMMERCIAL SEALANT

Last Review or Revision: June 2010

Objective and Application
To permanently close soil borings consistent with industry practice and close the soil boring to prevent its serving as a vertical conduit for movement of environmental impacts through soils. Soil borings of 2-inch diameter or greater are considered borings within the definition of this TSOP.

Equipment
Hand tools appropriate to the job.

Commercially-available sealant materials for well abandonment. Terracon preference for non-slurry fill will be chipped Benseal®, high yield Wyoming bentonite or equivalent.

Procedures
Backfill the soil boring with a mixture of soil cuttings and bentonite or other sealant material as required by state law (i.e. backfill with hydrated bentonite to near surface and cemtn the upper two feet). When specified by the project manager, attempt to place soil cuttings back in the borehole in the order that the soil was removed so that soil is returned to the approximate depth from which it originated.

Attached Supporting Documents

- Volume of Soil Boring, Annulus around 2” and 4” Casings, and Grout Mixtures
- Brainard Kilman Field Facts

Other Supporting Documents

- **ASTM D5299-99** *Standard Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities*.

VOLUME OF SOIL BORING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING					
				INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0.028	0.088	0.201	0.29	0.37	0.57
2	0.06	0.18	0.4	0.6	0.7	1.1
3	0.08	0.26	0.6	0.9	1.1	1.7
4	0.11	0.35	0.8	1.2	1.5	2.3
5	0.14	0.44	1.0	1.5	1.9	2.9
6	0.17	0.53	1.2	1.7	2.2	3.4
7	0.20	0.62	1.4	2.0	2.6	4.0
8	0.22	0.70	1.6	2.3	3.0	4.6
9	0.25	0.79	1.8	2.6	3.3	5.1
10	0.28	0.88	2.0	2.9	3.7	5.7
11	0.31	0.97	2.2	3.2	4.1	6.3
12	0.34	1.06	2.4	3.5	4.4	6.8
13	0.36	1.14	2.6	3.8	4.8	7.4
14	0.39	1.23	2.8	4.1	5.2	8.0
15	0.42	1.32	3.0	4.4	5.6	8.6
16	0.45	1.41	3.2	4.6	5.9	9.1
17	0.48	1.50	3.4	4.9	6.3	9.7
18	0.50	1.58	3.6	5.2	6.7	10.3
19	0.53	1.67	3.8	5.5	7.0	10.8
20	0.56	1.76	4.0	5.8	7.4	11.4
22	0.62	1.94	4.4	6.4	8.1	12.5
24	0.67	2.11	4.8	7.0	8.9	13.7
25	0.70	2.20	5.0	7.3	9.3	14.3
26	0.73	2.29	5.2	7.5	9.6	14.8
28	0.78	2.46	5.6	8.1	10.4	16.0
30	0.84	2.64	6.0	8.7	11.1	17.1
32	0.90	2.82	6.4	9.3	11.8	18.2
34	0.95	2.99	6.8	9.9	12.6	19.4
35	0.98	3.08	7.0	10.2	13.0	20.0
36	1.01	3.17	7.2	10.4	13.3	20.5
38	1.06	3.34	7.6	11.0	14.1	21.7
40	1.12	3.52	8.0	11.6	14.8	22.8
45	1.3	4.0	9.0	13.1	16.7	25.7
50	1.4	4.4	10.1	14.5	18.5	28.5
55	1.5	4.8	11.1	16.0	20.4	31.4
60	1.7	5.3	12.1	17.4	22.2	34.2
65	1.8	5.7	13.1	18.9	24.1	37.1
70	2.0	6.2	14.1	20.3	25.9	39.9
75	2.1	6.6	15.1	21.8	27.8	42.8
80	2.2	7.0	16.1	23.2	29.6	45.6
85	2.4	7.5	17.1	24.7	31.5	48.5
90	2.5	7.9	18.1	26.1	33.3	51.3
95	2.7	8.4	19.1	27.6	35.2	54.2
100	2.8	8.8	20.1	29.0	37.0	57.0
125	3.5	11.0	25.1	36.3	46.3	71.3
150	4.2	13.2	30.2	43.5	55.5	85.5
175	4.9	15.4	35.2	50.8	64.8	99.8
200	5.6	17.6	40.2	58.0	74.0	114.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING					
				INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0.17	0.66	1.5	2.14	2.78	4.29
2	0.3	1.3	3.0	4.3	5.6	8.6
3	0.5	2.0	4.5	6.4	8.3	12.9
4	0.7	2.6	6.0	8.6	11.1	17.2
5	0.9	3.3	7.5	10.7	13.9	21.5
6	1.0	4.0	9.0	12.8	16.7	25.7
7	1.2	4.6	10.5	15.0	19.5	30.0
8	1.4	5.3	12.0	17.1	22.2	34.3
9	1.5	5.9	13.5	19.3	25.0	38.6
10	1.7	6.6	15.0	21.4	27.8	42.9
11	1.9	7.3	16.5	23.5	30.6	47.2
12	2.0	7.9	18.0	25.7	33.4	51.5
13	2.2	8.6	19.5	27.8	36.1	55.8
14	2.4	9.2	21.0	30.0	38.9	60.1
15	2.6	9.9	22.5	32.1	41.7	64.4
16	2.7	10.6	24.0	34.2	44.5	68.6
17	2.9	11.2	25.5	36.4	47.3	72.9
18	3.1	11.9	27.0	38.5	50.0	77.2
19	3.2	12.5	28.5	40.7	52.8	81.5
20	3.4	13.2	30.0	42.8	55.6	85.8
22	3.7	14.5	33.0	47.1	61.2	94.4
24	4.1	15.8	36.0	51.4	66.7	103.0
25	4.3	16.5	37.5	53.5	69.5	107.3
26	4.4	17.2	39.0	55.6	72.3	111.5
28	4.8	18.5	42.0	59.9	77.8	120.1
30	5.1	19.8	45.0	64.2	83.4	128.7
32	5.4	21.1	48.0	68.5	89.0	137.3
34	5.8	22.4	51.0	72.8	94.5	145.9
35	6.0	23.1	52.5	74.9	97.3	150.2
36	6.1	23.8	54.0	77.0	100.1	154.4
38	6.5	25.1	57.0	81.3	105.6	163.0
40	6.8	26.4	60.0	85.6	111.2	171.6
45	7.7	29.7	67.5	96.3	125.1	193.1
50	8.5	33.0	75.0	107.0	139.0	214.5
55	9.4	36.3	82.5	117.7	152.9	236.0
60	10.2	39.6	90.0	128.4	166.8	257.4
65	11.1	42.9	97.5	139.1	180.7	278.9
70	11.9	46.2	105.0	149.8	194.6	300.3
75	12.8	49.5	112.5	160.5	208.5	321.8
80	13.6	52.8	120.0	171.2	222.4	343.2
85	14.5	56.1	127.5	181.9	236.3	364.7
90	15.3	59.4	135.0	192.6	250.2	386.1
95	16.2	62.7	142.5	203.3	264.1	407.6
100	17.0	66.0	150.0	214.0	278.0	429.0
125	21.3	82.5	187.5	267.5	347.5	536.3
150	25.5	99.0	225.0	321.0	417.0	643.5
175	29.8	115.5	262.5	374.5	486.5	750.8
200	34.0	132.0	300.0	428.0	556.0	858.0

VOLUME OF ANNULUS AROUND 2" CASING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0.065	0.178	0.26	0.34	0.54
2	0	0.13	0.4	0.5	0.7	1.1
3	0	0.20	0.5	0.8	1.0	1.6
4	0	0.26	0.7	1.0	1.4	2.2
5	0	0.33	0.9	1.3	1.7	2.7
6	0	0.39	1.1	1.6	2.0	3.2
7	0	0.46	1.2	1.8	2.4	3.8
8	0	0.52	1.4	2.1	2.7	4.3
9	0	0.59	1.6	2.3	3.1	4.9
10	0	0.65	1.8	2.6	3.4	5.4
11	0	0.72	2.0	2.9	3.7	5.9
12	0	0.78	2.1	3.1	4.1	6.5
13	0	0.85	2.3	3.4	4.4	7.0
14	0	0.91	2.5	3.6	4.8	7.6
15	0	0.98	2.7	3.9	5.1	8.1
16	0	1.04	2.8	4.2	5.4	8.6
17	0	1.11	3.0	4.4	5.8	9.2
18	0	1.17	3.2	4.7	6.1	9.7
19	0	1.24	3.4	4.9	6.5	10.3
20	0	1.30	3.6	5.2	6.8	10.8
22	0	1.43	3.9	5.7	7.5	11.9
24	0	1.56	4.3	6.2	8.2	13.0
25	0	1.63	4.5	6.5	8.5	13.5
26	0	1.69	4.6	6.8	8.8	14.0
28	0	1.82	5.0	7.3	9.5	15.1
30	0	1.95	5.3	7.8	10.2	16.2
32	0	2.08	5.7	8.3	10.9	17.3
34	0	2.21	6.1	8.8	11.6	18.4
35	0	2.28	6.2	9.1	11.9	18.9
36	0	2.34	6.4	9.4	12.2	19.4
38	0	2.47	6.8	9.9	12.9	20.5
40	0	2.60	7.1	10.4	13.6	21.6
45	0	2.9	8.0	11.7	15.3	24.3
50	0	3.3	8.9	13.0	17.0	27.0
55	0	3.6	9.8	14.3	18.7	29.7
60	0	3.9	10.7	15.6	20.4	32.4
65	0	4.2	11.6	16.9	22.1	35.1
70	0	4.6	12.5	18.2	23.8	37.8
75	0	4.9	13.4	19.5	25.5	40.5
80	0	5.2	14.2	20.8	27.2	43.2
85	0	5.5	15.1	22.1	28.9	45.9
90	0	5.9	16.0	23.4	30.6	48.6
95	0	6.2	16.9	24.7	32.3	51.3
100	0	6.5	17.8	26.0	34.0	54.0
125	0	8.1	22.3	32.5	42.5	67.5
150	0	9.8	26.7	39.0	51.0	81.0
175	0	11.4	31.2	45.5	59.5	94.5
200	0	13.0	35.6	52.0	68.0	108.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0.491	1.33	1.91	2.55	4.06
2	0	1.0	2.7	3.8	5.1	8.1
3	0	1.5	4.0	5.7	7.7	12.2
4	0	2.0	5.3	7.6	10.2	16.2
5	0	2.5	6.7	9.6	12.8	20.3
6	0	2.9	8.0	11.5	15.3	24.4
7	0	3.4	9.3	13.4	17.9	28.4
8	0	3.9	10.6	15.3	20.4	32.5
9	0	4.4	12.0	17.2	23.0	36.5
10	0	4.9	13.3	19.1	25.5	40.6
11	0	5.4	14.6	21.0	28.1	44.7
12	0	5.9	16.0	22.9	30.6	48.7
13	0	6.4	17.3	24.8	33.2	52.8
14	0	6.9	18.6	26.7	35.7	56.8
15	0	7.4	20.0	28.7	38.3	60.9
16	0	7.9	21.3	30.6	40.8	65.0
17	0	8.3	22.6	32.5	43.4	69.0
18	0	8.8	23.9	34.4	45.9	73.1
19	0	9.3	25.3	36.3	48.5	77.1
20	0	9.8	26.6	38.2	51.0	81.2
22	0	10.8	29.3	42.0	56.1	89.3
24	0	11.8	31.9	45.8	61.2	97.4
25	0	12.3	33.3	47.8	63.8	101.5
26	0	12.8	34.6	49.7	66.3	105.6
28	0	13.7	37.2	53.5	71.4	113.7
30	0	14.7	39.9	57.3	76.5	121.8
32	0	15.7	42.6	61.1	81.6	129.9
34	0	16.7	45.2	64.9	86.7	138.0
35	0	17.2	46.6	66.9	89.3	142.1
36	0	17.7	47.9	68.8	91.8	146.2
38	0	18.7	50.5	72.6	96.9	154.3
40	0	19.6	53.2	76.4	102.0	162.4
45	0	22.1	59.9	86.0	114.8	182.7
50	0	24.6	66.5	95.5	127.5	203.0
55	0	27.0	73.2	105.1	140.3	223.3
60	0	29.5	79.8	114.6	153.0	243.6
65	0	31.9	86.5	124.2	165.8	263.9
70	0	34.4	93.1	133.7	178.5	284.2
75	0	36.8	99.8	143.3	191.3	304.5
80	0	39.3	106.4	152.8	204.0	324.8
85	0	41.7	113.1	162.4	216.8	345.1
90	0	44.2	119.7	171.9	229.5	365.4
95	0	46.6	126.4	181.5	242.3	385.7
100	0	49.1	133.0	191.0	255.0	406.0
125	0	61.4	166.3	238.8	318.8	507.5
150	0	73.7	199.5	286.5	382.5	609.0
175	0	85.9	232.8	334.3	446.3	710.5
200	0	98.2	266.0	382.0	510.0	812.0

VOLUME OF ANNULUS AROUND 4" CASING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0	0.11	0.2	0.26	0.46
2	0	0	0.2	0.4	0.5	0.9
3	0	0	0.3	0.6	0.8	1.4
4	0	0	0.4	0.8	1.0	1.8
5	0	0	0.6	1.0	1.3	2.3
6	0	0	0.7	1.2	1.6	2.8
7	0	0	0.8	1.4	1.8	3.2
8	0	0	0.9	1.6	2.1	3.7
9	0	0	1.0	1.8	2.3	4.1
10	0	0	1.1	2.0	2.6	4.6
11	0	0	1.2	2.2	2.9	5.1
12	0	0	1.3	2.4	3.1	5.5
13	0	0	1.4	2.6	3.4	6.0
14	0	0	1.5	2.8	3.6	6.4
15	0	0	1.7	3.0	3.9	6.9
16	0	0	1.8	3.2	4.2	7.4
17	0	0	1.9	3.4	4.4	7.8
18	0	0	2.0	3.6	4.7	8.3
19	0	0	2.1	3.8	4.9	8.7
20	0	0	2.2	4.0	5.2	9.2
22	0	0	2.4	4.4	5.7	10.1
24	0	0	2.6	4.8	6.2	11.0
25	0	0	2.8	5.0	6.5	11.5
26	0	0	2.9	5.2	6.8	12.0
28	0	0	3.1	5.6	7.3	12.9
30	0	0	3.3	6.0	7.8	13.8
32	0	0	3.5	6.4	8.3	14.7
34	0	0	3.7	6.8	8.8	15.6
35	0	0	3.9	7.0	9.1	16.1
36	0	0	4.0	7.2	9.4	16.6
38	0	0	4.2	7.6	9.9	17.5
40	0	0	4.4	8.0	10.4	18.4
45	0	0	5.0	9.0	11.7	20.7
50	0	0	5.5	10.0	13.0	23.0
55	0	0	6.1	11.0	14.3	25.3
60	0	0	6.6	12.0	15.6	27.6
65	0	0	7.2	13.0	16.9	29.9
70	0	0	7.7	14.0	18.2	32.2
75	0	0	8.3	15.0	19.5	34.5
80	0	0	8.8	16.0	20.8	36.8
85	0	0	9.4	17.0	22.1	39.1
90	0	0	9.9	18.0	23.4	41.4
95	0	0	10.5	19.0	24.7	43.7
100	0	0	11.0	20.0	26.0	46.0
125	0	0	13.8	25.0	32.5	57.5
150	0	0	16.5	30.0	39.0	69.0
175	0	0	19.3	35.0	45.5	80.5
200	0	0	22.0	40.0	52.0	92.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0	0.84	1.48	1.95	3.46
2	0	0	1.7	3.0	3.9	6.9
3	0	0	2.5	4.4	5.9	10.4
4	0	0	3.4	5.9	7.8	13.8
5	0	0	4.2	7.4	9.8	17.3
6	0	0	5.0	8.9	11.7	20.8
7	0	0	5.9	10.4	13.7	24.2
8	0	0	6.7	11.8	15.6	27.7
9	0	0	7.6	13.3	17.6	31.1
10	0	0	8.4	14.8	19.5	34.6
11	0	0	9.2	16.3	21.5	38.1
12	0	0	10.1	17.8	23.4	41.5
13	0	0	10.9	19.2	25.4	45.0
14	0	0	11.8	20.7	27.3	48.4
15	0	0	12.6	22.2	29.3	51.9
16	0	0	13.4	23.7	31.2	55.4
17	0	0	14.3	25.2	33.2	58.8
18	0	0	15.1	26.6	35.1	62.3
19	0	0	16.0	28.1	37.1	65.7
20	0	0	16.8	29.6	39.0	69.2
22	0	0	18.5	32.6	42.9	76.1
24	0	0	20.2	35.5	46.8	83.0
25	0	0	21.0	37.0	48.8	86.5
26	0	0	21.8	38.5	50.7	90.0
28	0	0	23.5	41.4	54.6	96.9
30	0	0	25.2	44.4	58.5	103.8
32	0	0	26.9	47.4	62.4	110.7
34	0	0	28.6	50.3	66.3	117.6
35	0	0	29.4	51.8	68.3	121.1
36	0	0	30.2	53.3	70.2	124.6
38	0	0	31.9	56.2	74.1	131.5
40	0	0	33.6	59.2	78.0	138.4
45	0	0	37.8	66.6	87.8	155.7
50	0	0	42.0	74.0	97.5	173.0
55	0	0	46.2	81.4	107.3	190.3
60	0	0	50.4	88.8	117.0	207.6
65	0	0	54.6	96.2	126.8	224.9
70	0	0	58.8	103.6	136.5	242.2
75	0	0	63.0	111.0	146.3	259.5
80	0	0	67.2	118.4	156.0	276.8
85	0	0	71.4	125.8	165.8	294.1
90	0	0	75.6	133.2	175.5	311.4
95	0	0	79.8	140.6	185.3	328.7
100	0	0	84.0	148.0	195.0	346.0
125	0	0	105.0	185.0	243.8	432.5
150	0	0	126.0	222.0	292.5	519.0
175	0	0	147.0	259.0	341.3	605.5
200	0	0	168.0	296.0	390.0	692.0

GROUT MIXTURES

PORTLAND CEMENT GROUT

CUBIC FEET	CEMENT 94 lb. sack	BENTONITE lbs.	WATER gallons
1	0.6339	3.17	5.07
1.577	1.0	5.0	8.0
2	1.27	6.3	10.1
3	1.90	9.5	15.2
4	2.54	12.7	20.3
5	3.17	15.9	25.4
6	3.80	19.0	30.4
7	4.44	22.2	35.5
8	5.07	25.4	40.6
9	5.71	28.5	45.6
10	6.34	31.7	50.7
11	6.97	34.9	55.8
12	7.61	38.0	60.8
13	8.24	41.2	65.9
14	8.87	44.4	71.0
15	9.51	47.6	76.1
16	10.14	50.7	81.1
17	10.78	53.9	86.2
18	11.41	57.1	91.3
19	12.04	60.2	96.3
20	12.68	63.4	101.4
22	13.95	69.7	111.5
24	15.21	76.1	121.7
25	15.85	79.3	126.8
26	16.48	82.4	131.8
28	17.75	88.8	142.0
30	19.02	95.1	152.1
32	20.28	101.4	162.2
34	21.55	107.8	172.4
35	22.19	111.0	177.5
36	22.82	114.1	182.5
38	24.09	120.5	192.7
40	25.36	126.8	202.8
45	28.5	142.7	228.2
50	31.7	158.5	253.5
55	34.9	174.4	278.9
60	38.0	190.2	304.2
65	41.2	206.1	329.6
70	44.4	221.9	354.9
75	47.5	237.8	380.3
80	50.7	253.6	405.6
85	53.9	269.5	431.0
90	57.1	285.3	456.3
95	60.2	301.2	481.7
100	63.4	317.0	507.0
125	79.2	396.3	633.8
150	95.1	475.5	760.5
175	110.9	554.8	887.3
200	126.8	634.0	1014.0

BENTONITE GROUT

CUBIC FEET	BENTONITE 50 lb sack	WATER gallons
1	0.444	6.22
2	0.9	12.4
2.25	1	14
3	1.3	18.7
4	1.8	24.9
5	2.2	31.1
6	2.7	37.3
7	3.1	43.5
8	3.6	49.8
9	4.0	56.0
10	4.4	62.2
11	4.9	68.4
12	5.3	74.6
13	5.8	80.9
14	6.2	87.1
15	6.7	93.3
16	7.1	99.5
17	7.5	105.7
18	8.0	112.0
19	8.4	118.2
20	8.9	124.4
22	9.8	136.8
24	10.7	149.3
25	11.1	155.5
26	11.5	161.7
28	12.4	174.2
30	13.3	186.6
32	14.2	199.0
34	15.1	211.5
35	15.5	217.7
36	16.0	223.9
38	16.9	236.4
40	17.8	248.8
45	20.0	279.9
50	22.2	311.0
55	24.4	342.1
60	26.6	373.2
65	28.9	404.3
70	31.1	435.4
75	33.3	466.5
80	35.5	497.6
85	37.7	528.7
90	40.0	559.8
95	42.2	590.9
100	44.4	622.0
125	55.5	777.5
150	66.6	933.0
175	77.7	1088.5
200	88.8	1244.0

Terracon

Standard Operating Procedures

E.1800
PHYSICAL FIELD MEASUREMENTS – SURFACE LAYOUT

Last Review or Revision: June 2010

Objective

To provide standard procedures for the gathering of surface layout data to be used in the development of site diagrams.

Layouts obtained using this procedure will be approximate. Distances from available reference features are generally measured using a tape, and angles are estimated. The location of site features will be accurate only to the degree implied by these methods. Typically, Terracon field technicians will strive for a lateral layout precision of ±5 feet. If more precise locations are desired, use TSOP E.1808.

Equipment

- Clipboard,
- Graph Paper,
- Measuring Tape,
- Measuring Wheel,
- Optical Rangefinder (optional), and
- Scale with a straight edge.

Procedures

Measure distances from available reference features using a tape or measuring wheel and estimate angles.

Alternate Methods

1. Baseline and offsets
   Designate a baseline for the X- and Y-dimensions. Measure perpendicular dimensions to site features from the X- and Y-dimension baselines.

2. Triangulation
   Locate two (2) stationary points on the site that are significantly separated (e.g., two (2) corners of an on-site building, etc.). Measure distances to site features from each stationary point. Angles need not be measured or estimated if distances from each stationary point are recorded.

3. Distance and direction
   Designate a stationary point on the site. Measure distance and angle to site features from the stationary point.

4. Rangefinder
   An optical rangefinder is an optical device for measuring distances. When looking through the rangefinder at the desired target surface, two images are presented. Using an adjustment knob on the rangefinder, the user can bring the two images together so that they overlap. A scale can then be read which indicates the distance to the target surface. In this manner, distances to relevant features can be estimated without traversing a site. It should be noted that optical rangefinders must be calibrated periodically.

Terracon

STANDARD OPERATING PROCEDURE

E.1805
FIELD MEASUREMENT – SURFACE ELEVATIONS

Last Review or Revision: June 2010

Objective

The purpose of this procedure is to establish proper and accurate methods to be used when surveying elevations on environmental sites.

Background

On many environmental sites, the horizontal spacing of monitoring wells may be less than 100 feet. The hydraulic gradient between these wells may be less than 0.1 feet, which necessitates an accurate survey of the top of casing elevations to 0.01 feet. Inaccurate elevations in some cases could result in an inaccurate determination of groundwater flow direction.

Procedures

a) In performing an elevation survey of a site, the first task is to choose a benchmark (BM). The BM should be of a permanent nature, that will exist over time and will not change in elevation. When using a fire hydrant as a BM, do not use the operating plug or adjustment nut. The elevation on the operating plug can change after the fire hydrant is used. Poured-in-place concrete, sign foundations and hydrants will make a good BM. Attempt to identify a BM with a known elevation to USGS or another datum.

b) When setting up the level, the instrument person should first ensure that the tripod legs are stabilized and then the instrument can be leveled using the leveling screws. Instruments which are leveled using a bar level need to be turned at right angles and re-leveled until the instrument indicates level in all directions without adjustment. Instruments with a bull's eye level ordinarily need to be leveled once, but the instrument should be turned 360° to ensure that it is accurately leveled.

c) When using the level, the instrument person should be able to accurately read the rod to 0.01 feet. This entails limiting the distance of the shot so that this accuracy can be maintained. Windy conditions will affect accuracy, especially on shots made where the rod is extended beyond 15 feet in the air. If the instrument person has difficulty reading the rod due to the wind blowing and bending the rod, or because the rod person has difficulty holding the rod in a stable position, the instrument person has the responsibility of resetting to ensure an accurate shot.

d) Finally, the instrument person should perform a closed loop, utilizing balanced backsight and foresight distances. This requires shooting back to the point of beginning, usually the BM. Upon completion of the loop, the instrument person is responsible for immediately calculating the closure error. On a small site with no turning points, this consists of resetting the instrument, thus creating a turning point and a loop. In these situations, the BM elevation should close with less than 0.01

foot difference. In situations where there are several turning points, the closure error should not be greater than 0.01 foot per 100 linear feet of surveyed distance.

Attached Supporting Documents

- Terracon Survey Notes Form

Terracon

STANDARD OPERATING PROCEDURE

E.1810
FIELD MEASUREMENT – SUBSURFACE SOILS

Last Review or Revision: June 2010

Objective

To obtain accurate and precise depth measurements from boreholes.

Equipment

- Monitoring equipment specified by the project manager.
- Decontamination equipment (Alconox detergent, buckets, brushes and water).
- Proper forms as specified by project manager.
- Measuring tape.
- Tool Box.
- Site Map.

Procedures

- Meet with project manager to discuss project;
- Measure the depth to bottom of borehole by lowering the measuring tape down into the borehole until the tape becomes slack. Raise and lower tape several times to ensure that the bottom of the hole has been accurately defined.
- Observe the measurement on the tape at the reference point to the nearest 0.1 foot.
- Record the borehole depth measurements on the appropriate field form. Write legibly and check the form for accuracy and completeness prior to departing the borehole location.
- If the tape becomes dirty from contacting the side of the borehole, perform proper decontamination procedures before the next measurement.

Terracon

STANDARD OPERATING PROCEDURE

E.1820
PHYSICAL FIELD MEASUREMENT - GROUNDWATER

Last Review or Revision: June 2010

1. OBJECTIVE

To obtain accurate and precise groundwater measurements from monitoring points including monitoring wells, piezometers, observation wells and pumping wells. The groundwater levels may represent static water levels, non-static levels undergoing recovery, or pumping water levels based on site-specific conditions.

2. EQUIPMENT

- Monitoring equipment specified by the project manager;
- Decontamination equipment (Alconox detergent, buckets, brushes and water);
- Proper forms as specified by project manager - Take copy of most recent data form to use as a field check for representative values. Make sure you have referenced benchmark elevation for each well.
- Measuring devices: electronic water level indicator or interface probe;
- Steel Tape (100' minimum) marked off in units as described above;
- Fuel and water indicating paste;
- Tool Box; and
- Site Map.

3. PROCEDURES

- Meet with project manager to discuss project;
- Select the measuring device based on site specific conditions and regulatory requirements. Note: some state regulatory agencies may not allow the use of plumbers chalk or water detection paste. Familiarize yourself with the measurement equipment. Check equipment conditions prior to mobilization, and consider possibility of field break down. Be prepared to use back up equipment.

- Maintain a similar reference point which should be the top of the riser pipe for the monitoring well itself DO NOT USE THE TOP OF THE PROTECTIVE CASING DUE TO POSSIBLE SEASONAL FROST HEAVE OF THE OUTER CASING(S).

- Use the marked point on the riser pipe or the highest point on the riser pipe as the specific reference point unless the riser pipe terminus is level. In this case, use the side of the riser pipe terminus which is closest to the lock clasp on the protective casing.

- Measure the depth to water level by lowering the measuring device down into the well until the water level is encountered. With the electronic devices, an audible or visual signal will be broadcast. This method may be of limited use in steel cased wells.

- Once contact with the groundwater has been established, observe the measurement on the tape at the reference point to the nearest 0.01 foot. Record this measurement to the nearest 0.01 foot.

- If collecting a pumping water level from a well which contains a pump, take care to ensure that the tape/probe is not in contact with the pump intake.

- Collection of water levels from a potable well will require stringent decontamination procedures for organic, inorganic and microbiological concerns. Consult with the project manager for specific procedures.

- Record the water level and well depth measurements on the water level data sheet. Write legibly and check the form for accuracy and completeness prior to departing the well location.

- Perform proper decontamination procedures between each well.

4. ATTACHED SUPPORTING REFERENCES

Form 164 10.89 - Groundwater Elevation Data

5. OTHER REFERENCES

- Equipment manuals specific to unit prescribed by the Equipment Manager or project Manager.
- “Interface Probe User Guide & Manual”, ORS Environmental Equipment, 1991.
- ASTM D4750-87 (Reapproved 1993) “Standard Test method for Determining Subsurface Liquid levels in a Borehole or Monitoring Well (Observation Well)”.

Terracon
STANDARD OPERATING PROCEDURE

E.1830
PHYSICAL FIELD MEASUREMENT – FREE-PHASE PRODUCT

Last Revision or Review: June 2010

1. OBJECTIVE

To accurately and precisely determine the thickness of product floating on the groundwater (assuming it is a density of less than 1.0 – light non-aqueous phase liquid - LNAPL) or at the bottom of the well (assuming density of greater than 1.0 – dense non-aqueous phase liquid - DNAPL).

2. EQUIPMENT

- Monitoring equipment specified by the project manager.
- Decontamination equipment - (scrub buckets with distilled water, Alconox, brushes,clean paper towels and ispopropyl alcohol or other solvent, as required.)
- Proper forms and indelible pen.
- Measuring device;

    a) Interface Probe.
    b) Water Level Indicator.

- Fuel indicating paste;
- Toolbox; and
- Site Map.

3. PROCEDURE FOR USING INTERFACE PROBE - LNAPL

- Electronic devices such as the interface probe will signal contact with product and groundwater. These instruments should be used based on manufacturer's recommendations.
- Perform battery test and check decon condition of device prior to leaving the office.
- Turn on device.

- Carefully lower the sensor probe out of protective housing and into the well. (Avoid sliding the tape over protective pipe casing edge or monitoring well casing as it may scrape or kink conductor edge measuring tape.)

- As probe encounters product (in excess of 1 mm thick) a tone will sound. Measure fluid depth relative to top of monitoring well casing as discussed in E.1820. Report reading to nearest 0.01 feet. Remove probe and re-check reading three times.

- Lower probe through product layer very slowly and stop when water interface is encountered. Use previous records to prejudge this distance if possible. Water is encountered when a second tone type is generated. Record after 2 readings, recheck water level within 0.01 foot.

- Retrieve tape slowly, carefully cleaning the tape and probe as it is withdrawn from the well. (Never store a contaminated probe or tape in the protective housing or carrying case.)

4. PROCEDURE FOR USING INTERFACE PROBE - DNAPL

* Electronic devices such as the interface probe will signal contact with product and groundwater. These instruments should be used based on manufacturer's recommendations.

* Perform battery test and check decon condition of device prior to leaving the office.

* Turn on device.

* Carefully lower the sensor probe out of protective housing and into the well. (Avoid sliding the tape over protective pipe casing edge or monitoring well casing as it may scrape or kink conductor edge measuring tape.)

* As probe encounters water, a tone will sound. Measure fluid depth relative to top of monitoring well casing as discussed in E.1820. Report reading to nearest 0.01 feet. Remove probe and re-check reading three times.

* Lower probe through water layer very slowly and stop when water/product interface is encountered. Use previous records to prejudge this distance if possible. Product is encountered when a second tone type is generated. Record after 2 readings, recheck product level within 0.01 foot.

* Retrieve tape slowly, carefully cleaning the tape and probe as it is withdrawn from the well. (Never store a contaminated probe or tape in the protective housing or carrying case.)

5. PROCEDURE FOR USING WATER LEVEL INDICATOR

* Obtain a water level using the proper measuring device.

* Select the measuring device based on site specific conditions and regulatory requirements. NOTE: some state regulatory agencies may not allow the use of water/fuel detection paste.

* Maintain a similar reference point which should be the top of the riser pipe for the monitoring well itself. DO NOT USE THE TOP OF THE PROTECTIVE CASING. Use the highest point on the riser pipe as the specific reference point unless the riser pipe terminus is level. In this case, use the side of the riser pipe terminus which is closest to the lock clasp on the protective casing.

* Place fuel indicating paste on the water level indicator.

* Measure the product thickness by carefully lowering the measuring device until it encounters groundwater based on the previously measured depth. Retrieve the device and measure the height of petroleum product above the water level based on the color change on the paste.

* Repeat measurements until three consistent measurements are obtained.

* Based on the method employed, it may be necessary to subtract the height of water from the total petroleum wetted height.

* Minimize submergence of the indicator below the water level to prevent mixing of petroleum and water.

* Record product thickness measurements and visual observations such as color on the water level data sheet. Complete record entry on data sheet. Re-check entries and proceed to next well after properly decontaminating all equipment used down hole.

ATTACHED SUPPORTING REFERENCES

Form 163 (8/89) - Floating Product and Groundwater Elevation Data

OTHER REFERENCES

- Equipment manuals specific to unit prescribed by the Equipment Manager or project Manager.
- "Interface Probe User Guide & Manual", ORS Environmental Equipment, 1991.
- ASTM D4750-87 (Reapproved 1993) "Standard Test method for Determining Subsurface Liquid levels in a Borehole or Monitoring Well (Observation Well)".

Terracon
STANDARD OPERATING PROCEDURE

E.1840
FIELD MEASUREMENT – HYDRAULIC CONDUCTIVITY TESTING (SLUG)

Last Review or Revision: June 2010

Objective

To obtain a representative average horizontal hydraulic conductivity value for the aquifer material around the screened interval.

Equipment

Well lock keys.
Data logger and transducer, field printer with power source.
Water level indictor/interface probe.
Time recording device capable of recording in seconds.
Bailer pump or slug device.
Decontamination equipment.
Water supply or collection containers.
Funnel for slug in testing (not recommended).
Solid slug for slug in/slug out testing.

Procedures

a) Slug-out test.

- Meet with the Project Manager (PM) to identify which monitoring wells are to be tested, and to identify geology and anticipated conditions.

- After measuring the total depth of the well, place the transducer in the well at least one foot off the well bottom.

- For projects involving more than one well, establish well testing order by moving from least contaminated to most contaminated.

- Select measuring equipment and time interval for test. If aquifer consists of silt, sand, or gravel, the data logger and transducer should be used to record the rapid changes in water levels. A timer and a water level indictor can be used in low permeability aquifers. If a slug test is conducted in an aquifer with a grain size less than the grain size of the gravel pack in the well, the slug test should be at least 45 minutes in duration. This will allow the effects of gravel pack drainage to be minimized and the actual water level change in the aquifer to be measured.

- Measure static water level and total depth of monitoring well to be tested.

- When using the data logger and transducer, start recording elapsed time before removing the volume of water.

- Remove a volume of water from the well using a bailer or pump. Be sure not to lower the water level in the well below the transducer.

- Record water level changes with time as the water level increases toward the static level.

- Initially, water levels should be obtained frequently (minimum every 15 seconds for first five minutes; shorter intervals may be required for more conductive formations).

- Stop recording water level data with time when the water level is within 10% of the static level.

- Perform calculations using methods determined by the PM, if so desired.

b) Slug-in test

- Slug-in tests should only be performed in wells where the static water level is above the screened interval or well conditions will not allow a slug-out test to be performed. When a slug-in test has to be performed, enough water should be added to the well so that the changes in water level during the test occur within the riser pipe and not the screened interval.

- Meet with the PM to identify which monitoring wells are to be tested, identify geology and anticipated conditions.

- For projects involving more than one well, establish well testing order by moving from least contaminated to most contaminated.

- Select measuring equipment. If the aquifer consists of silt, sand, or gravel, the data logger and transducer should be used to record the rapid changes in water levels. A timer and water level indicator can be used in low permeability aquifers.

- Measure static water level and total depth of monitoring well to be tested. After measuring the total depth of the well, place the transducer in the well at least one foot off the bottom.

- When using the data logger and transducer, start recording elapsed time before adding water.

- Add water to the well. Be sure to add enough water so that the water level in the well rises above the screened interval.

- Record water level changes with time as the water level decreases toward the static level.

- Initially, water levels should be obtained frequently (minimum: every 15 seconds for first five minutes; shorter intervals may be required for more permeable formations).

- Stop recording water level data with time when the water level is within 10% of the static level.

- Complete field hydraulic conductivity test form as test progresses.

- Perform calculations using methods determined by the PM, if required.

ATTACHED SUPPORTING REFERENCES

- In-Situ, Inc., Operator's Manual, Hermit® 1000C
- BRSLUG: A Bouwer-Rice Slug Test Program for Windows, 1995.
- Terracon Field Form: Slug Test Form

OTHER REFERENCES

ASTM D4044-96 "Standard Test Method for (Field Procedure) for Instantaneous Change in Head (Slug) Tests for Determining Hydraulic Properties of Aquifiers".

Terracon

STANDARD OPERATING PROCEDURE

E.1900
GROUNDWATER SAMPLING - BAILER

Last Review or Revision: August 2011

Objective

To collect a representative groundwater sample from the sampling point for chemical analysis. This includes the documentation of sampling methods, sampling supplies, and protocol to reduce potential for alteration and or cross-contamination during the sampling event.

Equipment

- Monitoring equipment specified by project manager (e.g. pH meter, turbidity meter, conductivity meter, etc).
- Water level indicator;
- Decontamination equipment;
- Proper forms, labels and indelible ink pen;
- Sample containers and packing material, tape, and labels;
- Filtration device and filters (and fixing agents as appropriate);
- Cooler with ice pack and packing media;
- Bucket (calibrated in gallons or liters)
- Disposable bailers
- Keys for locking cap on well;
- Rope - steel, nylon, teflon, or polypropylene;
- Deionized (DI) water;
- Knife; and
- Site map.

Procedures

a) Preparation

- Meet with Project Manager;
- Obtain the bottles, forms, and equipment necessary to complete the sampling event;
- Calibrate all field equipment;
- Establish sampling well sequence (generally least impacted to most impacted).

b) Field Activities

- Open the manway and remove the well cap for each well. Allow at least 10 minutes for the groundwater level to stabilize in each well to atmospheric conditions.
- Using the electronic water level indicator, collect and record the water level from each well (and well depth, if requested by P.M.). The water level measurement should be taken from a marking/notch on the well PVC riser if present, if no notch is present the measurement should be taken from the side of the riser facing north for uniformity of data. The electronic water level indicator must be decontaminated after use at each well.

1) Contamination minimization

- Use plastic sheet if necessary
- Use proper bailing techniques (hand over hand) to prevent rope from touching the ground

2) Purging

- Use a bailer (dedicated).
- Purge each well of 3 well volumes or until the well goes dry. Field measurements as requested by the Project Manager (i.e. pH, temperature, and specific conductance) will be measured during the well purging. Field equipment must be decontaminated after use at each well.
- Disposal of water - containerize if necessary or collect in a bucket and dispose of on site away from the well to minimize potential vertical migration along the annular well space.
- May need to vary the purging rate to minimize drawdown and subsequent cascading of water if dealing with very volatile compounds (i.e., vinyl chloride).

c) Sample Collection

1) Preservation

- For volatiles, most laboratories provide pre-preserved vials, so field preservation is not required.

- The laboratory may provide an unpreserved container along with ampules of preservative. The ampules should be broken, releasing the preservative into the container, prior to collecting a sample. Gloves should be worn during this step to prevent exposure to the preservatives, and care should be taken to avoid spillage of the preservative.

2) Filtration

- In some states and under certain circumstances, water samples for metals analysis can or may be filtered in the field. Typically, this is accomplished using one-time, disposable 45 micron filters. Water is poured directly from the sample collection device into the filter, which is positioned over the laboratory-supplied container. After use, the filters should be disposed of consistent with the project's other waste streams.

- If a reusable filter apparatus (e.g., "barrel filter" or equivalent) is used, the non-expendable portions of the equipment should be cleaned in accordance with E2410 between each sample. If pre-filters are used, they should be positioned before the 45 micron filters. All filter media should be used only once and replaced between samples.

3) Collection

- Refer to SOP E.1400 for stabilization of water quality parameters prior to sample collection.

- Minimize disturbances which may aerate the sample (i.e., lower bailer slowly into water, pour slowly into sample container, etc.).

- Pour water from the top of the bailer or insert the bottom emptying device for sample transfer.

- Transfer the groundwater sample directly to the laboratory prepared sample container or the filter cup.

- Samples collected for VOC's should always be collected from a recently filled bailer full of water as soon as it is brought to the surface.

- Collect samples for VOC's by forming a positive meniscus on the sample vial and capping immediately.

- VOC samples must be free of air bubbles (i.e. turn VOA upside down to check for air bubbles). If air bubble(s) persist within the vial, note in the field log and on the chain-of-custody to notify the laboratory that the sample is unpreserved.

- Do not over-fill sample containers which contain a preservative.

- Place samples in cooler with ice or blue-ice to maintain a temperature between 2°C and 6°C.

4) Data Documentation

- Record all pertinent sampling information on the sampling container label, sampling information form, chain-of-custody, and shipping form.

- Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, sampling methodology, parameters to be analyzed, stabilization data, and general observations.

- Make all entries in the chain-of-custody form prior to leaving the site. Ensure that the chain-of-custody protocol required for the project is maintained.

- If samples must be shipped, the chain-of-custody form must be enclosed with the samples and the container sealed with Terracon security labels. Obtain a post office receipt, bill of lading or similar document from the shipper to be included as part of the chain-of-custody documentation. Return one copy of the chain-of-custody documentation to the project manager.

Attached Supporting Documents

- **ASTM D4448-85a** *Standard Guide for Sampling Monitoring Wells.*

Terracon

STANDARD OPERATING PROCEDURE

E.20
STANDARD SAFE OPERATING PROCEDURES FOR HAZARDOUS WASTE OPERATIONS

Last Review or Revision: June 2010

I. PURPOSE

This document defines standard safe operating procedures for use on project sites where intrusive activities will be preformed and where soil/groundwater contaminants are known or are reasonably expected to exist. These procedures will be incorporated in the project safety and health plan which is mandatory for intrusive activities conducted at all such project sites.

II. SAFETY AND HEALTH RESPONSIBILITY

A. The Project Manager is ultimately responsible for ensuring that work on environmental investigation/remediation projects are performed in accordance with provisions outlined in the project safety and health plan. A Site Safety and Health Officer (SSO) will be designated at each project site to monitor compliance with this safety-related practices during field activities. The SSO will ensure that a copy of the safety and health plan is available on site for the duration of project activities.

B. The Corporate Safety and Health Manager will be consulted for each intrusive environmental investigation/remediation project. The Corporate Safety and Health Manager will develop the site safety and health plan, and will be available to consult with Project Manager/SSO in the event of questions, concerns or changed site conditions. The Corporate Safety and Health Manager will specify air monitoring and personal protective equipment requirements for the project, and will assist in obtaining specialized equipment required for the project.

C. If hazardous conditions develop during the course of project activity, the SSO in conjunction with the Terracon Corporate Safety and Health Manager, will coordinate actions required to safeguard site personnel and members of the general public. Additional safety measures will be verbally communicated to all project personnel, recorded in writing and appended to the site safety and health plan.

D. Terracon and subcontractor task leaders will be responsible for:

- Presenting the contents of the site safety and health plan to all subordinate site personnel.

- Monitoring compliance with applicable provisions of the safety and health plan.

- Periodically inspecting heavy equipment and other machinery and maintaining such equipment in compliance with applicable federal, state or local safety regulations.

- Enforcement of corrective actions.

III. MEDICAL SURVEILLANCE

A. All Terracon personnel participating in intrusive projects with known or reasonably anticipated contaminants must be enrolled in the Terracon Medical Surveillance Program. Each project participant will be certified by a licensed physician as fit for respirator and semi-permeable/impermeable protective equipment use. Medical clearance will be current to within one year of the project start date.

B. Certificates of medical examination for all project personnel will be maintained by the Corporate Safety and Health Manager and/or by the SSO in the project command center or support vehicle.

C. At the discretion of the Terracon consulting physician, an "exit" physical examination will be conducted at the completion of project activities or upon termination of a project participant. Follow-up medical examinations will also be provided in the event of job site injury or unprotected exposure to contaminants in excess of eight-hour time weighted average permissible exposure limits.

IV. TRAINING REQUIREMENTS

A. Terracon personnel participating in hazardous waste operations will have completed 40 hour Hazardous Waste Operations Training and at least three days supervised field activity. A current 8-hour annual refresher training certificate will be required for all personnel. Training certificates for all project personnel will be maintained by the Corporate Safety and Health Manager.

B. The SSO will conduct a pre-project safety and health briefing for all project participants. The personnel responsible for project safety and health will be addressed, as will site history, scope of work, site control measures, emergency procedures and site communications. The briefing will address site contaminants, air monitoring protocols, action levels for upgrade/downgrade of personal protective equipment and level of personal protective equipment to be employed for each project task.

C. Project participants will sign the Acknowledgment of Instruction form contained in each safety and health plan following the initial site briefing.

V. CHEMICAL HAZARDS

A. The Project Manager is responsible for obtaining available information regarding site contaminants, including analytical information obtained from samples previously collected at the project site, and for forwarding the information to the

Corporate Safety and Health Manager for research and hazard analysis. A site specific safety and health plan will be developed notifying personnel of the potential chemical contaminants, their health hazards, routes of entry, warning properties and symptoms of exposure.

B. Evaluation of chemical contaminants/concentrations will be used in the development of the air monitoring and personal protective equipment requirements contained in the site safety and health plan.

VI. PHYSICAL HAZARDS

A. Drilling Projects

- All personnel working around drill rigs will be familiarized with emergency shut-down procedures and the position of "kill" switches.
- No loose fitting clothing, jewelry or unsecured long hair is permitted near the rig.
- Keep hands and feet away from all moving parts while drilling is in progress. Shovel auger cuttings with long handled shovel. *DO NOT* use hands or feet.
- Daily inspection of all ropes, cables and moving parts is mandatory.
- A first aid kit and fire extinguisher will be immediately available at all times.
- All drill crews shall consist of at least two persons.
- No drilling is permitted during impending electrical storms, tornadoes or when rain creates a hazardous work environment.
- A minimum horizontal and vertical clearance distance of **10 feet** must be maintained between the drill rig and overhead power lines; use spotters to help rig operator position the vehicle when near overhead power lines.

B. Excavation Project Sites

- Wherever possible, soil samples will be collected from backhoe buckets. Personnel will enter excavations only as a final option and only in accordance with the regulatory requirements outlined above.
- AT NO TIME WILL TERRACON PERSONNEL ENTER EXCAVATIONS TO COLLECT SOIL SAMPLES UNTIL A PROPER MEANS OF EGRESS/EXIT IS PROVIDED AND THE EXCAVATION HAS BEEN INSPECTED BY A COMPETENT PERSON AND APPROPRIATELY SHORED OR SLOPED IN ACCORDANCE WITH THE OSHA EXCAVATIONS STANDARD (29 CFR 1926, SUBPART P).
- Personnel will remain at least 3 feet from the sides of excavations during sample collection and excavation observation.

- Personnel will remain outside the swing radius of backhoe buckets during excavation, and will stand behind the backhoe or within line-of-sight contact with the backhoe operator at all times.

VII. ACCIDENT PREVENTION

- The Site Safety Officer will hold daily safety briefings at the beginning of each day of site activity.

- Traffic control measures will be arranged for all projects conducted within or immediately adjacent to active roadways. Signage, warning and/or channelizing devices will conform to the Manual on Uniform Traffic Control Devices. Flagging operations will be conducted only by personnel who have received training in proper traffic flagging procedures. The preferred method of traffic control will be to contract these services to a reputable traffic control service knowledgable in local traffic control regulations.

- Safety orange work vests will be worn by personnel working within 10 feet of any active roadway.

- The Site Safety Officer will ensure that unauthorized personnel do not enter the work zone. Authorized visitors will be briefed on site contaminants, personal protective equipment requirements and the decontamination provisions of the site safety and health plan.

- The Site Safety Officer will continually inspect the work area for infractions of safety and health requirements as contained in the site specific safety and health plan.
- The Site Safety Officer will investigate and immediately report all accidents to the Corporate Safety and Health Manager.

- Site activities will be conducted only during daylight hours unless adequate portable lighting is mobilized to the project site.

- The "buddy system" will be observed at all times during intrusive site investigations. A minimum of two people will work together and remain within eye sight or not greater than 100 ft. apart.

- Teamwork and the use of mechanical lifting devices will be employed where practical to ease lifting tasks and reduce the potential for muculoskeletal injury.

VIII. SITE CONTROL

A. An Exclusion Zone, Contaminant Reduction Zone and a Support Zone will be established on hazardous waste operations sites requiring Level C or Level Bpersonal protective equipment. Defined access and egress points will be established and personnel will enter only through those points.

B. As permitted by site topography, the area within a 50 foot radius of a drill rig and 100 foot radius of UST removal excavations will be considered the Exclusion Zone. Only those personnel designated by the Project Manager/SSO are allowed to enter the Exclusion Zone. Where practical, or where their use will prevent public injury, temporary signs or barricade fencing will be established to define the

Exclusion Zone. ABSOLUTELY NO SMOKING WILL BE PERMITTED WITHIN THE EXCLUSION OR CONTAMINANT REDUCTION ZONES ON ANY PETROLEUM CONTAMINATED SITE.

C. If unauthorized personnel attempt to enter the exclusion zone, the SSO will verbally inform the individual(s) to leave the project site. If unauthorized individuals refuse to leave the Exclusion Zone or are considered in danger or pose danger to project personnel, the SSO will cease project activities (i.e., shut down drill rigs, excavation equipment, etc.) and notify the client representative or the local police of the situation.

IX. AIR MONITORING

A. Air monitoring protocols will be designed to prevent personnel exposure to airborne contaminants in excess of established permissible exposure limits. The results of field air monitoring will be used to determine the continued adequacy of initial personal protective equipment.

B. Task Leader(s) will be knowledgeable in the operation and troubleshooting of air monitoring equipment. A manual on the operation of each air monitoring instrument and an appropriate calibration kit will be mobilized to the project site with the instrument. Air monitoring instruments will be calibrated under field conditions each day prior to use. Task Leaders are instructed to consult the operator's manual for appropriate calibration gas and calibration techniques.

X. PERSONAL PROTECTIVE EQUIPMENT

Personal protective equipment requirements for each hazardous waste operations site and task will be specified in the site safety and health plan. Personal protective equipment selection will be based upon the site contaminants and tasks to be performed. Personal protective equipment ensembles will be selected in general accordance with standard EPA levels of protection as follows:

A. **Level A** - To be selected when the greatest level of skin, respiratory, and eye protection is required. Level A personal protective equipment ensembles will consist of:

1. Positive pressure, full face-piece self-contained breathing apparatus (SCBA), or positive pressure supplied air respirator with escape SCBA, approved NIOSH.
2. Totally-encapsulating chemical-protective suit.
3. Coveralls.(optional, as appropriate)
4. Long underwear.(optional, as appropriate)
5. Gloves, outer, chemical-resistant.
6. Gloves, inner, chemical-resistant.
7. Boots, chemical-resistant, steel toe and shank.
8. Hard hat (under suit).(1)
9. Disposable protective suit, gloves and boots

B. **Level B** - To be selected when the highest level of respiratory protection is necessary but a lesser level of skin protection is required. Level B personal protective equipment ensembles will consist of:

1. Positive pressure, full-facepiece self-contained breathing apparatus (SCBA), or positive pressure supplied air respirator with escape SCBA (NIOSH approved).
2. Hooded chemical-resistant clothing (overalls and long-sleeved jacket; coveralls; one or two-piece chemical-splash suit; disposable chemical-resistant overalls).
3. Coveralls.(optional, as appropriate)
4. Gloves, outer, chemical-resistant.
5. Gloves, inner, chemical-resistant.
6. Boots, outer, chemical-resistant steel toe and shank.
7. Hard hat.

C. **Level C** - To be specified when the identity and approximate concentration of airborne contaminants is known and the criteria for using air purifying respirators are met. Level C personal protective equipment ensembles shall consist of the following:

1. Full-face (typically) air purifying respirators (NIOSH approved).
2. Chemical-resistant clothing (coveralls; two-piece chemical-splash suit; disposable chemical- resistant overalls, as appropriate).
3. Gloves, outer, chemical-resistant.
4. Gloves, inner, chemical-resistant.
5. Boots (outer), chemical-resistant steel toe and shank.
6. Disposable boot-covers, outer, chemical-resistant (optional)
7. Hard hat.(1)

D. **Level D** - A work uniform affording minimal protection. Level D or modified Level D personal protective equipment will be used when contaminant concentrations are low and not readily absorbable through the skin and where atmospheric monitoring indicates no need for respiratory protection. The following constitute Level D equipment; it may be used as appropriate:

1. Coveralls.
2. Chemical-resistant gloves.(style selected based on contaminants)
3. Boots/shoes, chemical-resistant steel toe and shank.
4. Boots, outer, chemical-resistant (disposable).
5. Safety glasses or chemical splash goggles (where appropriate).
6. Hard hat.

XI. DECONTAMINATION

A. Equipment decontamination is necessary on all contaminated project sites. Personnel decontamination for projects below personal protective Level C will consist of washing off safety footwear, proper cleaning or disposal of outer and inner gloves and thorough washing of face, arms and hands. A full body shower will be required as soon as possible upon leaving the project site.

B. For projects involving personal protective equipment at Level C or above, a decontamination station will be established on the interface of the Exclusion Zone. A Contaminant Reduction Zone will be established and will extend 10 feet beyond the decontamination station.

- Two Wash Tubs
- Scrub Brush
- Plastic Bags
- Water and Alconox Detergent

The wash tub on the exclusion zone side of the site will contain a solution of water and Alconox detergent; the second wash tub will contain clean rinse water. Personnel decontamination will consist primarily of detergent washing and rinsing of reusable exterior protective gear. Coveralls will be removed by turning the clothing inside out.

Personnel may not leave the contaminant reduction zone without proceeding through the decontamination sequence described below.

- Wash work gloves, boots and polylaminated protective coveralls,
- Rinse work gloves, boots and coveralls,
- Remove tape at wrists and ankles,
- Remove protective coveralls,
- Remove respirator
- Dispose of spent cartridges; wash and rinse respirator
- Remove outer gloves
- Remove inner gloves

Expendable personal protective equipment will be placed in plastic trash bags, sealed and disposed of per client agreement. Decontamination solutions will be containerized or disposed of as arranged by Project Manager.

C. Decontamination of equipment will be performed to limit the migration of contaminants off-site. All equipment will be cleaned prior to site entry to remove grease, oil and encrusted soil. Decontamination of large equipment will consist of physically removing gross contamination with shovels, brushes etc. followed by detergent and water high pressure wash with a clean water rinse. The Project Manager is responsible for determining if decontamination solutions must be containerized. If so, a decontamination sump or polyethylene sheeting and fluid containers will be mobilized and established in the decontamination area. Decontamination of hand samplers and similar small equipment will be performed at a designated location within the Contaminant Reduction Zone. Decontamination of such equipment will consist of detergent solution wash and clean water rinse. Specialized decontamination solutions (acids, solvents, biocidal, etc.) may be specified in site specific site safety and health plans.

XII. SITE COMMUNICATIONS

Communication between personnel on most hazardous waste project sites will be via verbal communication or hand signals. Visual contact between members of task teams should be possible throughout the course of project activities. Contact with the SSO will be through direct verbal communication. The following hand signals will be used whenever verbal communication is limited:

Signal	Meaning
Thumbs Up	OK, all is well
Grab throat with both hands	Can't breathe
Shake head, thumbs down	NO, negative
Point right (when facing equipment operator)	Move/steer left
Point left when facing equipment operator)	Move/steer right
Grab partner's wrist	Leave area immediately

XIII. EMERGENCY RESPONSE PROCEDURES

A. The Project Manager is responsible for obtaining and recording emergency telephone contacts in the appropriate section of the site safety and health plan prior to site mobilization: A mobile telephone will typically be available on hazardous waste operations project sites.

B. In the case of personal injury, appropriately trained personnel will be requested to provide first aid and emergency rescue. For minor injuries, such as cuts, burns, exhaustion, heat cramps, insect stings, etc., the affected employee will be removed to an uncontaminated area. The SSO or other designated employee trained in first aid procedures will administer appropriate first aid. If the injury requires additional medical attention, the injured employee will be cleaned and transported to the nearest hospital or emergency medical facility.

C. For more serious injuries the SSO or designee will summon an ambulance to the project site. No attempt will be made by Terracon personnel to move the victim, without the aid and/or instructions of qualified medical personnel.

D. If the victim cannot be safely moved without a stretcher or other specialized equipment, the victim will be removed at the earliest possible moment by appropriately attired Terracon personnel with the direction and/or assistance of qualified medical response personnel. The injured employee will be immediately decontaminated and transported to the nearest medical facility. A crew member designated by the SSO will inform the ambulance crew of contaminants of concern and provide assistance with additional decontamination if required.

XIV. EVACUATION AND SHUTDOWN PROCEDURES

A. On project sites posing a significant risk of chemical or physical hazard exposure, the site safety and health plan will instruct the SSO to establish and notify site personnel of emergency "rally" points. In the event of a site emergency, personnel will immediately exit the site and assemble at the designated rally point. Evacuation routes will be dependent on site topography and wind conditions. The

routes will be selected and presented by the SSO daily prior to site activity.

B. If emergency evacuation becomes necessary, the SSO will sound the emergency alarm (e.g. support vehicle horn or compressed air horn). Personnel will safely shutdown all electrical and mechanical equipment and quickly proceed to closest designated rally point. The SSO will then account for each crew member on site.

C. In the event that a Terracon employee does not report to the designated rally point within 5 minutes of the evacuation alarm, the SSO will perform an immediate assessment of site conditions. If site conditions do not pose an immediate hazard to life or health, the SSO will initiate search and rescue efforts utilizing two crew members attired in appropriate personal protective equipment.

Terracon
STANDARD OPERATING PROCEDURE

E.2000
GROUNDWATER SAMPLING – LOW FLOW GROUNDWATER SAMPLING

Last Revision or Review: August 2011

Objective

To collect a representative groundwater sample from the sampling point for chemical analysis. This procedure should be used when attempting to minimize stress to the aquifer due to monitoring well sampling. This procedure includes the documentation of sampling methods, sampling supplies and protocol to reduce potential for alteration and or cross-contamination during the sampling event.

Equipment

- Groundwater Elevation Data form;
- laboratory Chain-of-Custody form;
- laboratory sample labels;
- field logbook;
- indelible ink pen;
- pH, temperature, and specific conductance meter;
- turbidity meter;
- sample containers and packing material;
- cooler with ice pack and packing media;
- well purging equipment – disposable bailers and string;
- sampling device – low-flow peristaltic pump;
- mobile, secure shed to protect equipment during sampling (insulated during colder weather);
- keys for locking cap on well and secure shed;
- deionized (DI) water; and
- site map.

Procedures

Preparation - Meet with Project Manager;

- obtain the sample containers, forms, and equipment necessary to complete the sampling event;

- calibrate all field equipment i.e., temperature, pH, specific conductance meter and turbidity meter;
- document equipment calibration in field logbook; and
- establish sampling sequence.

Monitoring Well Purging

- document all field activities in field logbook and field forms
- water levels
  - Open the manway and remove the well cap for each well. Allow at least 10 minutes for the groundwater level to stabilize in each well to atmospheric conditions.
  - Using the electronic water level indicator, collect and record the water level from each well (and well depth, if requested by P.M.). The water level measurement should be taken from a marking/notch on the well PVC riser if present, if no notch is present the measurement should be taken from the side of the riser facing north for uniformity of data. The electronic water level indicator must be decontaminated after use at each well.
- purge monitoring wells
  - using bailers, remove three (3) well casing volumes of water from each well (or until well goes dry)
  - collect purge water in a bucket and dispose of on site away from the well to minimize potential vertical migration along the annular well space
- return to site after stabilization period of two (2) days
- contamination minimization
  - use plastic sheet as an apron to isolate the wellhead by splitting sufficiently to pass the well protector and slide over wellhead onto ground.

Sampling

- Preservation
  - For volatiles, most laboratories provide pre-preserved vials, so field preservation is not required.
  - The laboratory may provide an unpreserved container along with ampules of preservative. The ampules should be broken, releasing the preservative into the container, prior to collecting a sample. Gloves should be worn during this step to prevent exposure to the preservatives, and care should be taken to avoid spillage of the preservative.
- Filtration
  - In some states and under certain circumstances, water samples for metals analysis can or may be filtered in the field. Typically, this is accomplished using one-time, disposable 45 micron filters. Water is poured directly from the sample collection device into the filter, which is positioned over the laboratory-supplied container. After

use, the filters should be disposed of consistent with the project's other waste streams.

- If a reusable filter apparatus (e.g., "barrel filter" or equivalent) is used, the non-expendable portions of the equipment should be cleaned in accordance with E2410 between each sample. If pre-filters are used, they should be positioned before the 45 micron filters. All filter media should be used only once and replaced between samples.

- use a low-flow sampling pump
- determine proper depth of purging device, approximately half the distance of the water column or approximate middle of the well screen
- attach new section of pump tubing to pump
- lower pump suction tubing slowly (to minimize disturbance) into well to midpoint of sampling zone
- start pump at its lowest setting and slowly increase speed until discharge occurs.
- check water level and adjust pump speed to maintain drawdown at less than four (4) inches and pump no faster than 0.1 L/min
- stabilization parameters (refer to E.1400)
  - record turbidity, temperature, pH, and specific conductance at regular intervals

- transfer the groundwater sample directly to the laboratory prepared sample containers
  - fill sample containers by allowing pump discharge to flow gently down the side of the container with minimal disturbance
  - do not over-fill sample containers which contain a preservative, certain containers will come pre-preserved from the laboratory
  - place samples in cooler with ice

Data Documentation

- record all pertinent sampling information on the sampling container label, sampling information form, chain-of-custody, and shipping form.
- pertinent data will vary based on the parameter and the form; however, the following data must be recorded - time, date, job number, project name, sampling location, samplers name, sampling methodology, parameters to be analyzed, stabilization data, and general observations.
- make appropriate entries in the chain-of-custody form at time of sample collection - ensure that the chain-of-custody protocol required for the project is maintained

ATTACHED SUPPORTING REFERENCES

Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells, EPA Region 1, July 30, 1996, Revision 2.

Terracon

STANDARD OPERATING PROCEDURE

E.2210
GENERAL SITE HOUSEKEEPING

Last Review or Revision: June 2010

Objective

To create and maintain an orderly working area that reduces the likelihood of injury and potential for sample contamination due to messy site conditions.

Equipment

- Equipment specified by project manager.
- Trash bags.
- Disposable chemical-resistant gloves.

Procedures

After using disposable equipment, dispose of the materials in general trash bags. Segregate materials if needed as specified by the project manager. Use disposable gloves when handling waste material to reduce the chance for cross-contamination and/or exposure to contaminants. Bags, paper, packaging, and other materials should not be allowed to remain unattended on the site. Dispose of full trash bags using an on-site dumpster, at the Terracon dumpster after returning to the office, in a designated sealed drum on-site, or as specified by the project manager. After decontamination of non-disposable equipment, store in an orderly fashion on the remedial vehicle or trailer to facilitate quick location and reduce the likelihood of injury. Dispose of auger cuttings and cleaning fluids as specified by the Project Manager.

Terracon

STANDARD OPERATING PROCEDURE

E.2220
DISPOSAL OF SPENT SUPPLIES

Last Review or Revision: June 2010

Objective
To provide for proper disposal of sampling equipment, personal protective equipment (PPE), etc. in accordance with applicable regulations.

Equipment

- Equipment specified by project manager.
- Trash bags.
- Disposable chemical-resistant gloves.

Procedures
Collect sampling equipment, spent PPE, cleaning fluids, etc. as specified by the Project Manager. This may include segregating the material and sealing in 55-gallon drums, placing securely on the site, or disposal to a nearby dumpster (non-hazardous). If material is to be containerized for transportation or storage on-site, clearly mark all containers as to their materials, taking care to use proper signage. Transportation of materials off-site may be by Terracon or a hazardous waste hauler. If a hazardous waste hauler is used, collect all documentation provided (i.e., scale tickets, waste manifests, etc.).

Terracon

STANDARD OPERATING PROCEDURE

E.2230
HANDLING AND STORAGE OF DRILL CUTTINGS (NON-HAZARDOUS)

Last Review or Revision: June 2010

Objective

To dispose of drill cuttings generated by drilling and well installation activities. This procedure is applicable to sites where the cuttings generated can be reasonably be assumed to be non-hazardous in nature. For handling and storage of drill cuttings that can be reasonably be assumed to be hazardous in nature, reference TSOP E.2235.

Equipment

- Plastic Sheeting.
- Shovel(s).
- Wheelbarrow.
- USDOT-approved 55-gallon drums
- Disposable chemical-resistant gloves.
- Material as specified by Project Manager.

Procedures

Spread the plastic sheeting over the ground in an area designated by the project manager for drill cutting storage. Using the shovel(s) and wheelbarrow, transport the generated cuttings to the stockpile area, if required. Use disposable gloves to reduce the likelihood of sample cross-contamination and/or exposure to site contaminants. Segregate the stockpiles of soil on the plastic sheeting if instructed by the project manager. After stockpiling soil, cover the soil piles with additional plastic sheeting. Use rocks or other available moderately heavy material to prevent the plastic sheeting from blowing off the pile.

As an alternate procedure, the Project Manager may prefer to containerize the auger cuttings in fifty-five (55) gallon drums for transportation off-site or disposal at a later date. Also as an alternate, the Project Manager may specify that auger cuttings be returned to the borehole (if allowed by state law) as much as possible, and then spread over the ground in the area near the boring.

Terracon
STANDARD OPERATING PROCEDURE

E.2240
SITE SECURITY PROCEDURES

Last Review or Revision: June 2010

Objective
To establish procedures for the security of the subject site and remediation systems during hours when employees are not present.

Equipment

a) Keyed-a-like locks.

b) Site key file box.

c) Site diagrams.

d) Operation and Maintenance procedures.

e) Materials specified by the Project Manager.

Procedures

a) Site Security

The Project Manager will determine the specific security requirements for each site. Coordination with the client may be necessary to provide additional keys if needed. Keyed-a-like locks should be the same throughout the entire system. A benchstock of commonly used locks is maintained for the field crews to replace broken or damaged locks. If open excavations or sensitive materials are located on-site, the Project Manager may specify the use of access control, such as fencing or barricading.

b) Key Control

Keys for each system will be maintained in the central key control box in the Terracon Office. Additional keys may be maintained by field crews and the project manager for routine access. The site keys will be contained on a tab and clearly marked with the site name, job number and location.

c) Site Monitoring

The field crew conducting field services is responsible for the security of the site. A final review must be conducted daily prior to leaving the site to check and document site security. The field crew will complete the "Daily Job Report" to indicate that the site is secure or that exceptions were encountered at the site. The project manager will then be able to schedule the necessary corrective action.

d) Site Documentation

Documentation must be maintained at each remediation site to assist field crews in routine operation, maintenance, and emergency activities. The following documents are to be maintained on site:

1. Site diagram of all wells associated with the site;
2. Schematic of the system including free product and water lines;
3. Electrical diagram;
4. Site Safety Plan;
5. Maintenance checklist and schedule;
6. Current settings and operational limits of the system (switch settings, and flow rates);
7. Summary of system operational characteristics (flow rates, inventory levels,
8. Operational and Maintenance manuals for all system components;

All components of the system are to be labeled for identification. This includes pump controls, flow lines, product lines, level probes, and valves.

e) Signs

Each site should have a sign identifying the site and indicating critical information concerning the operation of the system in the event unusual operations are encountered. This could include the client's name and phone number, or Terracon's name and phone number.

f) Vehicles

Secure all vehicles left on-site during non-working hours. Lock all doors and close all windows. Store the keys in the central key control box.

Following is an example of a Daily Job Report. Either this form or another similar form will be utilized to document daily on-site activities.

Job No _____

Test Date _____

DAILY JOB REPORT

Time on Job _____

Job Name _____

Time in Lab _____

Job Location_____

Time in Office _____

Contractor _____

Travel Time _____

TOTAL CHARGEABLE HOURS _____

Client _____

MILEAGE _____

Description of technical and/or engineering field operations including field test data*, locations (show direction of north), elevations, and other information:

_____

_____

_____

_____

_____

_____

_____

_____

TERRACON states that the above tests and/or field engineering services have been performed and the results are reported herein. This report, however, does not relieve the contractor of the responsibility to comply with the plans and specifications.

*Indicate when the field test data is estimated, pending final laboratory test results.

Attested to: _____

Position: _____

Company: _____

TERRACON
By: _____

FORM 113 - 6/85

Terracon

STANDARD OPERATING PROCEDURE

E.2405
CLEANING – GENERAL

LAST REVIEW OR REVISION: June 2010

OBJECTIVE AND APPLICATION

To prepare the equipment for field activities in a manner that minimizes the potential for obtaining biased or erroneous data due to contaminant transfer. Cleaning is performed as a quality assurance measure and a safety precaution. It minimizes cross-contamination between samples and also helps to maintain a clean working environment. This procedure provides general guidelines and should be used in conjunction with more specific procedures applicable to the cleaning method used.

EQUIPMENT

- As determined by the project manager
- Expendable supplies:
  - Disposable chemical-resistant gloves
  - Garbage bags
  - Aluminum foil or plastic
  - Laboratory glassware detergent such as Alconox or Trisodium Phosphate (TSP)
  - Containers for collection of waste liquids, if necessary
  - Dilute acid, methanol, ethanol, isopropyl alcohol or other cleaning fluid
- Source of potable water without chemicals that would interfere or be identified in chemical analysis of samples. The project manager may require laboratory testing of cleaning water as a background for evaluating chemical analyses.

PROCEDURES

Cleaning procedures will vary considerably based on the equipment, type of contaminant, type of sample and detection levels. Initial cleaning should take place at the site prior to demobilizing. This will minimize the spread of contamination. The extent of on-site cleaning will vary based on specific conditions; however, an attempt should be made to decontaminate as thoroughly as possible on site. The more care one applies on keeping the equipment clean, the less energy will be required on cleaning.

All field equipment must be prepared at the laboratory/office prior to use. This will include additional cleaning, inspection, and maintenance.

Equipment such as hand trowels, bailers, mixing bowls, hand augers, etc., should be cleaned and wrapped in aluminum foil (with shiny side out) or plastic, as appropriate, prior to mobilization.

Sampling and monitoring equipment is normally cleaned by washing and rinsing with liquids such as a soap or detergent solution, potable tap water, deionized water (DI), isopropyl alcohol, methanol, or a dilute acid.

The extent and type of contaminant will determine the degree of cleaning. If the level of contamination cannot be readily determined, cleaning should be based on the assumption that the equipment is highly contaminated.

Waste products produced by the cleaning procedures such as waste liquids, solids, gloves, used Chem-wipe® cleaning pads, etc., should be collected, stored in USDOT-approved 55-gallon drums on-site and disposed based on the nature of the contaminant. Specific details for the handling of these wastes should be addressed by the project manager.

Terracon

STANDARD OPERATING PROCEDURE

E.2410
CLEANING – MANUAL WASHING

LAST REVIEW OR REVISION: June 2010

OBJECTIVE AND APPLICATION

To prepare the equipment for field activities in a manner that minimizes the potential for obtaining biased or erroneous data due to contaminant transfer between sampling locations. Cleaning is performed as a quality assurance measure and a safety precaution. It minimizes cross-contaminants between samples and also helps to maintain a clean working environment.

EQUIPMENT

- As determined by the project manager
- Expendable supplies:
  - Disposable chemical-resistant gloves
  - Chem-wipe® cleaning pads
  - Garbage bags
  - Laboratory glassware detergent such as Alconox or Trisodium Phosphate (TSP)
  - Containers for collection of waste liquids, if necessary
  - Dilute acid, methanol, isopropyl alcohol, ethanol or other cleaning fluid
- Wash rack facility
- Cleaning containers with brushes (plastic, steel or stainless steel buckets)
- Aluminum foil or plastic
- Source of potable water without chemicals that would interfere or be identified in chemical analysis of samples. The project manager may require laboratory testing of cleaning water as a background for evaluating chemical analyses.

PROCEDURES

Cleaning procedures will vary considerably based on the equipment, type of contaminant, type of sample and detection levels. Initial cleaning should take place at the site prior to demobilizing.

This will minimize the spread of contamination. The extent of on-site cleaning will vary based on specific conditions; however, an attempt should be made to decontaminate as thoroughly as possible on site. The more care one applies on keeping the equipment clean, the less energy will be required on cleaning.

All field equipment must be prepared at the laboratory/office prior to use. This will include additional cleaning, inspection, and maintenance.

Equipment such as hand trowels, bailers, mixing bowls, hand augers, etc., should be cleaned and wrapped in aluminum foil (with shiny side out) or plastic, as appropriate, prior to mobilization.

Sampling and monitoring equipment is normally cleaned by washing and rinsing with liquids such as a soap or detergent solution, potable tap water, deionized water (DI), methanol, isopropyl alcohol or a dilute acid.

The extent and type of contaminant will determine the degree of cleaning. If the level of contamination cannot be readily determined, cleaning should be based on the assumption that the equipment is highly contaminated.

Listed below is a cleaning procedure which may be employed for field equipment such as a water level indicator at a monitoring well which contains dissolved petroleum hydrocarbons. If different or more elaborate procedures are required, they should be specified by the project manager during the project initiation meeting.

- Remove gross contamination from the equipment using a Chem-wipe® cleaning pad or brush.
- Wash with a soap or detergent solution
- Rinse with D.I. water
- Rinse with methanol or isopropyl alcohol (if method requires) and repeat rinse with D.I. water
- Repeat the entire procedure or any part of the procedure as necessary.

Waste products produced by the cleaning procedures such as waste liquids, solids, gloves, used Chem-wipe® cleaning pads, etc., should be collected and disposed of based on the nature of the contaminant. Specific details for the handling of these wastes should be addressed by the project manager.

Terracon

STANDARD OPERATING PROCEDURE

E.2420
CLEANING – HIGH-PRESSURE, HOT-WATER WASHING

Last Review or Revision: June 2010

Objective

To prepare the equipment for field activities in a manner which minimizes the potential for obtaining biased or erroneous data due to contaminant transfer. Decontamination is performed as a quality assurance measure and a safety precaution. It minimizes cross-contaminants between samples and also helps to maintain a clean working environment.

Equipment

- As determined by the project manager
- High pressure hot/cold water washing device or steam cleaner
- Expendable supplies:
- Disposable chemical-resistant gloves
- Chem-wipes
- Garbage bags
- Detergent (Alconox or TSP)
- Containers for collections of waste liquids, if necessary D.I. water
- Dilute acid, methanol, ethanol or other cleaning fluid
- Wash rack or other approved decontamination fluid collection system
- Cleaning containers with brushes (plastic, steel or stainless steel buckets)
- Aluminum foil or plastic, as appropriate
- Safety monitoring devices as specified in the safety plan.

Procedures

Decontamination procedures will vary considerably based on the equipment, type of contaminant, type of sample and detection levels. Initial decontamination should take place at the site prior to demobilizing. This will minimize the spread of contamination. The extent of on-site decontamination will vary based on specific conditions; however, an attempt should be

made to decontaminate as thoroughly as possible on site. The more care one applies on keeping the equipment clean, the less energy will be required on decontamination.

All field equipment must be prepared at the laboratory/office prior to use. This will include additional decontamination, inspection, and maintenance.

Equipment such as hand trowels, bailers, mixing bowls, hand augers, etc., should be cleaned and wrapped in aluminum foil (with shiny side out) or plastic, as appropriate, prior to mobilization.

Decontamination of larger objects, such as the working end of the drill rig or the downhole tools is accomplished using a high pressure wash.

Sampling and monitoring equipment is normally cleaned by washing and rinsing with liquids such a soap or detergent solutions, tap water, D.I. water, methanol, or a dilute acid.

The extent and type of contaminant will determine the degree of decontamination. If the level of contamination cannot be readily determined, cleaning should be based on the assumption that the equipment is highly contaminated.

Listed below is a decontamination procedure which may be employed for field equipment such as a water level indicator at a monitoring well which contains dissolved petroleum hydrocarbons. If different or more elaborate procedures are required, they should be specified by the project manager during the project initiation meeting.

- Remove gross contamination from the equipment using a chem-wipe or brush.
- Wash with a soap or detergent solution
- Rinse with D.I. water
- Rinse with methanol or other alternate cleaning agent (if method requires)
- Rinse with D.I. water
- Repeat the entire procedure or any part of the procedure as necessary.

Waste products produced by the decontamination procedures such as waste liquids, solids, gloves, chem-wipes, etc., should be collected, stored in USDOT-approved 55-gallon drums on the site, and disposed based on the nature of the contaminant. Specific details for the handling of these wastes should be addressed by the project manager.

Terracon

STANDARD OPERATING PROCEDURE

E.30

CHAIN OF CUSTODY DOCUMENTATION

Last Revision: June 2010

OBJECTIVE AND APPLICATION

This document defines standard operating procedures for documenting sample collection using proper chain-of-custody techniques. The purpose of proper chain-of-custody techniques is to provide accountability for and documentation of sample integrity from the time samples are collected until sample disposal.

This procedure is intended to document sample possession during each stage of a sample's life cycle, that is, during collection, shipment, storage, and the process of analysis.

EQUIPMENT

- Terracon chain-of-custody record(s) or laboratory-specific chain-of-custody forms (typically supplied with sample containers),
- If samples are being shipped via courier, custody seals for coolers,
- Indelible ink marker, and
- Zip top bag.

PROCEDURE

Sample containers will be labeled in advance of sampling with the sample date, location (well identifier), sampler's initials, and project name. Written sample custody procedures will be followed whenever samples are collected, transferred, stored, analyzed, or destroyed, in order to trace possession and handling of a sample from collection to disposal. Accountability for a sample begins when the sample is collected. Each sample will be accounted for with the use of sample labels, chain-of-custody forms, a record of sample collection, and field data notebooks.

The following chain-of-custody procedures will be implemented by the field staff:

- Entries in the field notebook and chain-of-custody form will be made in ink. Documentation of each sample must be completed at the time of sampling.
- The chain-of-custody should include at a minimum:
  - Project name and/or number
  - Name and contact information for the sampler collector

  - Collector's signature
  - Sample designation
  - Date sampled
  - Time sampled
  - Sample media
  - Number and size of containers for each sample
  - Types of sample preservatives used
  - Analyses requested
- The original chain-of-custody must accompany the samples at all times after collection, until receipt at the analytical laboratory. A copy of the chain-of-custody form will be kept by the field staff until filing at the office.
- The original chain-of-custody form should be sealed in a Ziplock bag if shipping samples on ice via courier. The sealed Ziplock bag will protect the document from moisture that may be present due to sample preservation. The chain-of-custody should be the last item packed in a sample cooler, so that it is easily accessible if the cooler is misplaced by the courier or shipped to an incorrect address.
- If shipping samples, a chain-of-custody specific to the contents of each cooler will be packaged with the respective samples. Chain-of-custody forms should not be shipped in separate containers than the samples they document. At least one custody seal should be completed by the collector and applied to each cooler sent to the laboratory. The custody seal should be affixed to the cooler in such a manner as to ensure breakage of the seal upon opening of the cooler (e.g., across the cooler lid opening).
- When the possession of samples is transferred, the individuals relinquishing and receiving the samples will sign, date, and note the time on the chain-of-custody form.
- If samples are shipped, strict chain-of-custody is violated. However, at the discretion of the project manager the procedures can still be followed.

ATTACHED REFERENCES

Terracon Form COC-7/92 *Chain-of-Custody Record*, revised 4/93

Quality Environmental Containers *Custody Seal*

OTHER SUPPORTING DOCUMENTS

ASTM D4840-99 *Standard Guide for Sampling Chain-of-Custody Procedures*

Terracon

STANDARD OPERATING PROCEDURE

E.300
SAMPLING & DRILLING PLATFORMS

Last Review or Revision: June 2010

Objective and Application

To provide standard procedure for the mechanical and working support of soil sampling collection by providing equipment platforms for the advancement of borings using traditional drilling equipment and techniques of the current industry. This procedure is applicable to drilling and sampling unconsolidated soils and highly weathered bedrock.

Equipment

The platform to carry the self-powered drill may be either truck-mounted or adapted to an all-terrain vehicle capable of leveling the platform for perpendicular alignment of the drill unit. The drill rig to be used for the field exploration employs a hydraulic head for drilling and sampling.

Drilling equipment may be solid stem augers, commercially manufactured, ranging in flight diameter from 6 to 8 inches. Commercially manufactured friction safety clips will be used to connect drill sections.

Drilling equipment may be three and one-quarter (3.25)-inch or four and one-quarter (4.25) nominal inside diameter hollow stem augers. Nominal outside diameter of flights will be 8 to 10 inches, depending on manufacturer's construction. Commercially manufactured connecting bolts will be used to connect drill sections.

Supporting tools and equipment capable of implementing standard sampling methods for soil and weathered bedrock.

Training

The drill operator and drill helper will have entered and be current in the following;

- Industry drilling experience no less than 3 years for operator, 1 year for helper.
- Enrolled in Terracon medical monitoring program for environmental operations
- Completed 40-Hour OSHA 1910.120 HAZWOPER training, with respiratory protection.
- Current certificate of annual 8-Hour OSHA 1910.120 HAZWOPER Refresher Training
- Knowledge and experience in drilling both vadose and saturated soils.
- Knowledge and experience in the operation and maintenance of the specified drill platform.

Procedures

The proper platform will be selected using as guidance ASTM D4700. Consistent with Section 1.7 of the standard, the guidance is not intended to replace education or experience and will be used in conjunction with professional judgment.

The drill platform will be as specified by the designated Drilling Coordinator or project manager. The field conditions as known and as recognized from historical working experience for a specific project will be reviewed. The appropriate drill platform will be assigned.

The Project Manager or Task Manager assigned will coordinate with Drilling to provide during mobilization the appropriate equipment and tools to support the specified sampling procedures presented for the field work.

Supporting Documents

- **ASTM D4700** *Standard Guide for Soil Sampling from the Vadose Zone*.

Other References

1. *A Well Drilling Methods Primer - An Overview of the Various Major Well Construction Methods*, Water Well Journal, November 1981.

2. Central Mine Equipment Manuals:CME550/CME750, specific to limits, use and supporting equipment of the drilling platform, most current.

3. *Drilling At Hazardous Waste Sites*, Drill Bits - Newsletter of the National Drilling Foundation, Columbia, SC, December 1984.

4. *Drilling* Safety Guide, National Drilling Federation, Diamond Core Drill Manufacturers Association, and National Drilling Contractors Association, 1989.

5. *Sling* Safety, Bulletin 3072, U.S. Department of Labor, Occupational Safety and Health Administration, 1986.

6. *TERRACON Employee Job Descriptions*, Terranet, Terracon, Inc. Human Resources, 2000.

Terracon

STANDARD OPERATING PROCEDURE

E.3000
BULK SAMPLING OF SUSPECT ASBESTOS-CONTAINING MATERIAL (ACM)

Last Review or Revision: September 2011

I. PURPOSE

The purpose of this standard operating procedure (SOP) is to provide information on the hazards of asbestos and procedures to follow to sample suspect materials for laboratory analysis. The following guidelines contained in this document apply to Terracon personnel who engage in bulk sampling of suspect ACM and are designed to provide standardization with respect to sample collection. This procedure should ensure that potential ACM samples are collected in a manner which allows for accurate analysis of the material and that sampling personnel are protected against potential asbestos fiber releases through controlled sampling techniques or appropriate personal protective equipment.

The objective of bulk sampling building materials and components suspected to contain asbestos is to characterize the items that contain asbestos in quantities greater than 1% or other content limit as specified by local or state guidelines. By characterizing the locations and quantities of ACM, exposure hazards can be greatly reduced.

II. BACKGROUND AND REFERENCE

Asbestos has been a common component used in building materials because of its strength enhancing and fire resisting properties. However, asbestos has been recognized as a human carcinogen and respiratory hazard. Due to its health hazards, building inspections and asbestos bulk sampling are required for schools as well as public or commercial properties prior to building renovation or demolition activities. Therefore, identifying, locating and quantifying materials containing asbestos is essential in the effort to prevent worker exposure to asbestos and prevent environmental contamination.

As a consequence of inhalation of asbestos fibers, a body of federal and state regulations has been developed. Federal regulations pertaining to asbestos are included in the Asbestos Hazard Emergency Response Act (AHERA) US EPA 40 CFR 763, Subparts E, F; National Emissions Standards for Hazardous Air Pollutants (NESHAP) (EPA 40 CFR 61); OSHA Asbestos Standards (29 CFR 1910.1001 and 29 CFR 1926.1101), and Asbestos School Hazard Abatement Reauthorization Act(ASHARA). Many states and local authorities have additional requirements including state-specific licensing and certification.

Terracon will comply with applicable federal, state and local regulations when conducting asbestos-related services.

III. EQUIPMENT

The minimum equipment necessary to conduct bulk sampling of suspect materials, in addition to the personal protective equipment outlined below in the Health and Safety Section, is listed below.

- Utility Knife, Chisel, Hammer, Screwdriver, Coring Tool
- Duct Tape
- Sample Containers (preferably ziplock-style clear plastic bags)
- Sample Labels and Indelible Marker
- Spray Atomizer containing Detergent Amended Water, Paper Towels/Wet Wipes
- Spray Adhesive
- Roof Patch Kit (if necessary)
- Measuring Wheel
- Camera
- Flashlight
- Field ACM Sample Log

IV. CERTIFICATION

Individuals conducting asbestos sampling must have the certifications listed below. Copies of these certifications and licenses should be taken to the site during the sampling event.

- United States Environmental Protection Agency Building Inspector training (and refresher training, if applicable)
- Asbestos Inspector State-license for the state of the project location (where necessary)

In addition, Terracon requires company-based training courses and hands-on experience of employees prior to commencing asbestos-related field services. Each employee must also receive respirator training, be medically monitored and successfully pass a fit-test utilizing issued respirator(s).

V. HEALTH AND SAFETY

Asbestos has been recognized to cause asbestosis, cancer of the lungs and digestive tract and mesothelioma. Asbestosis is a lung disorder characterized by a diffuse interstitial (between cell) fibrosis. The onset of asbestosis probably depends upon the asbestos dust

concentration, the morphology of the fiber and length of exposure. Cigarette smoking is strongly implicated as a co-carcinogenic among asbestos workers.

Under the OSHA asbestos standards, the employer has an obligation to protect employees against exposure to asbestos fibers in excess of 0.1 fibers per cubic centimeter of air (0.1f/cc). Personnel engaged in asbestos-related activities (including building inspections) must be trained, medically cleared and fit-tested for respiratory protection. Therefore, enrollment in a medical surveillance program in compliance with the OSHA asbestos and respiratory protection standards is mandatory. Terracon employees are not permitted to engage in asbestos-related activities unless they are enrolled in the Terracon medical surveillance program and have been medically cleared for respirator use by a physician.

The following safety and health protocols apply to Terracon personnel who engage in asbestos-related services. The guidelines contained in this document are based upon potential health hazards from exposure to asbestos fibers and physical hazards which may be encountered on survey project sites. Field activities will be performed in accordance with the procedures outlined in this document and applicable federal/state health and safety regulations.

Terracon personnel will use professional judgment during sample collection to prevent exposure to other building occupants. If unauthorized personnel attempt to enter a sampling area which could reasonably pose a fiber release hazard, the inspector will curtail bulk asbestos sample collection activity and request that the individual(s) leave the work area. If unauthorized personnel refuse to leave the work area, immediately contact the Project Safety Officer and/or a client representative. Sample collection activities should recommence only after unauthorized personnel have left the work area.

In the event that minor amounts of suspect ACM such as thermal system insulation (TSI), sprayed-on or troweled-on surfacing materials, ceiling texture, etc. are released during the course of sampling, sampling team members will immediately evacuate the area and don Level C personal protective equipment. The area of potential ACM release will then be approached and suspect materials will be thoroughly wetted with amended water, slowly and deliberately swept to a centralized pile, re-wetted, and containerized in 6-mil asbestos disposal bags. Affected surfaces will then be re-wetted and swabbed with clean cloths or paper towels. Used wipes will be disposed of as asbestos-containing waste.

In the event that large quantities of potential ACM are released during sample collection activities, personnel will immediately evacuate the area and notify the Project Safety Officer and the client representative. The Project Safety Officer will request that the area be sealed until a properly attired response team can be mobilized to the area with a high efficiency particulate air filter (HEPA) vacuum and other equipment necessitated by site conditions.

If suspect materials are in deteriorated condition and fiber release appears likely, or if sampling must be conducted overhead and/or above drop ceilings, personnel will upgrade to Level C personal protective equipment as itemized above. Level C personal protective equipment should be donned before moving drop ceiling panels, attic access panels, etc. where friable fireproofing or thermal system insulation are known to be present.

The indicated personal protective equipment shall be mobilized to asbestos sampling project sites on each day of sample collection and utilized, if necessary:

- Tyvek (standard) protective coveralls
- Half face or full face air purifying respirator equipped with HEPA (P-100) cartridges
- Impermeable gloves (nitrile or latex).
- Tyvek boot covers or washable outer footwear

Additional Health and Safety protocols such as those established by the owner/operator of the project site and Terracon's company policy regarding ladder safety, confined space entry and electrical hazards shall be followed.

VI. SAMPLING HAZARDS

a. Elevated Surfaces

Asbestos building inspections may include roofing materials and ceiling spaces containing suspect ACM. Appropriate ladders or other suitable devices (e.g., man lifts) will be used for gaining access to elevated sampling locations. Ladders will be inspected prior to use. Spreaders will be fully extended on all step ladders and firmly positioned prior to use. Where footing is uncertain, a sample team member will hold or otherwise secure ladders while in use by another sample team member. Personnel must always face ladders during both ascent and descent. Extension ladders will not be positioned more than one-quarter of their working length from buildings, walls, etc. (4:1 pitch). Sample team personnel will not walk on steeply pitched roof surfaces and will not walk on low pitched roofing surfaces while wet. Remain on designated roof walkways wherever present. Terracon personnel will visually inspect roofs prior to beginning sample collection activities and will avoid all areas which appear to be structurally unsound.

b. Confined Space Entry

Terracon asbestos inspectors will not enter any pit, shaft, tunnel, etc. which has limited means of egress, the potential for an oxygen deficient or toxic atmosphere or which was not designed for human occupancy without first developing a written safety plan which includes a confined space entry permit and procedures. Readily accessible spaces such as pipe tunnels in which personnel may stand can be entered to a distance where continuous visual and verbal communication can be maintained with another sample team member.

Adequate portable lighting must be utilized during sample collection in tunnels and similar spaces. No Terracon or sample team member may attempt to walk through a pipe tunnel, etc. beyond the sight of a stand-by team member unless written confined space entry procedures have been prepared for the project.

c. Electrical Contact Hazards

Personnel will remain cognizant of the location and condition of electrical wiring during the collection of bulk asbestos samples. A visual assessment of each work space will be made prior to sample collection and electrical contact hazards will be evaluated. Unguarded junction boxes, exposed wiring, knife switches, etc. will be avoided during the collection of bulk ACM samples, and coring tools will not be used in near proximity to electrical switches or receptacles.

VII. PROCEDURES FOR BULK SAMPLING OF SUSPECT ACM

The primary purpose of this section is to identify the methods and techniques of controlled sampling, sampling site control and use of appropriate personal protective equipment to protect Terracon personnel and members of the general public from exposure to asbestos fibers during sampling activities. Adherence to these procedures should enhance personnel safety during sample collection activities and aid in the suitability of samples for analysis. Field activities will be performed in accordance with the procedures outlined in this document and applicable federal/state health and safety regulations.

Protocols for inspection and bulk sampling are defined in AHERA regulations. These are applicable for any type of survey; for example, a school, an area prior to renovation, a building prior to demolition and inspections undertaken to rebut the OSHA presumption that certain materials contain asbestos.

An accredited Building Inspector must perform the inspection. A summary of AHERA sampling protocols is as follows:

1. Visually inspect the building interior and/or exterior and identify locations of suspect ACM. Identify homogenous areas of friable and non-friable suspect ACM. Document locations, condition, classification and estimated quantities of each suspect material. It is recommended to depict locations of materials on a building diagram and take photographs of sampled materials.

2. Touch each suspect ACM to determine its friability.

3. Collect representative samples of suspect ACM. Terracon recommends a minimum of three (3) samples of each material be collected from each homogeneous area.

However, specific materials may require additional samples such as surfacing material and insulation as described below. Judgment should be used on the need for and quantity of additional sample collection.

Surfacing materials: Collect, in a statistically random manner, at least three bulk samples from each homogenous area of 1,000 sq. ft. or less, at least five bulk samples from each homogenous area that is greater than 1,000 sq. ft. but less than or equal to 5,000 sq. ft., and at least seven bulk samples of each homogenous area larger than 5,000 sq. ft.

Thermal System Insulation (TSI): Collect, in a randomly distributed manner, at least three bulk samples from each homogeneous area of TSI; collect at least one bulk sample from each homogeneous area of patched TSI if the patched section is less than six linear or square feet; collect bulk samples from each insulated mechanical system where cement or plaster is used on tees, elbows, etc. in a manner sufficient to assess whether the material is ACM. After sampling collection, the inspector shall prevent the potential release of fibers by encapsulating the material, using a filler, caulk, duct tape, re-wettable cloth lagging or other applicable material.

Wet methods will be employed in the collection of suspect ACM samples. Water amended with a minimum of 10% commercially available window cleaning solution or other suitable surfactant should be used to moisten materials prior to sampling. Bulk asbestos samples will not be collected over the heads of, or in near proximity to, non-project personnel. Respiratory protection is not required when sampling non-friable suspect materials or materials below the breathing zone which are adequately wetted with amended water.

4. Bulk ACM samples should be immediately placed in sample containers and sealed while the materials are wet. If collecting samples of friable ACM or normally non-friable materials which are in deteriorated condition, precautions must be taken to prevent the release of fibers to the work area. Precautions include aggressively wetting the surface or potentially isolating the material (e.g. glove bagging) prior to disturbance.

5. Reusable sampling equipment will be gently rinsed with amended water. Dry the equipment with paper towels to be disposed of as contaminated materials.

6. The following decontamination sequence should be used following sample collection activities requiring Level C personal protective equipment:

   - Remove coveralls slowly turning the outside inward.
   - Place in heavy mil asbestos disposal bag.
   - Remove gloves and shoe covers (if utilized)
   - Remove respirator and carefully dispose of respirator cartridges in asbestos disposal bag.
   - Securely seal protective clothing and any potentially contaminated disposable sampling equipment in heavy mil asbestos disposal bags.

- Do not smoke or eat with soiled hands.
- Wash hands, face and forearms thoroughly before eating, drinking smoking or using toilet facilities.
- Shower thoroughly as soon as possible upon leaving the project site.

7. Bulk sample analysis for asbestos content is performed by polarized light microscopy (PLM). The analytical testing procedure is based on U.S. Environmental Protection Agency (USEPA) methods and National Voluntary Laboratory Accreditation Program (NVLAP) requirements. Terracon will use NVLAP accredited and appropriately licensed laboratories for analysis of asbestos bulk samples. Samples will be controlled with the analytical laboratory through chain of custody documentation.

STANDARD OPERATING PROCEDURE

E.400
SUBSURFACE SAMPLING - GEOPROBE® PLATFORM

Last Revision or Review: August 2011

1. OBJECTIVE

Geoprobe® equipment is a proprietary system capable of performing both sampling and onsite analysis functions. The proprietary name Geoprobe® has become synonymous with pushprobe technology, where the samplers or monitoring technology are advanced to depth by hydraulic push. The push may be assisted by inducing a vibration to the sampler.

Generally, sampling is accomplished using hydraulically pushed probes and analysis is accomplished using a gas chromatograph (GC). This equipment was originally developed to screen for the subsurface presence of volatile organic compounds (VOCs) in unsaturated zone soil gas. It may also be used to obtain soil and ground water samples and to screen such samples for VOCs or semivolatile organic compounds (SVOCs). However, the analytes of primary interest in Geoprobe® work are generally VOCs.

Geoprobe® equipment offers a relatively high degree of mobility for sampling and the ability to produce screening level analytical results while still mobilized to a site. The cost-effectiveness of this approach is reduced when soil or ground water samples are of interest, the analytes involved are SVOCs, or a higher degree of analytical quality is necessary.

The purpose of this document is to provide a standard field procedure for general application. It may be supplemented by the generation of written site-specific sampling and analysis plans (SAPs) prepared prior to field work. Project Managers are generally responsible for providing Geoprobe® operators with a SAP sufficiently in advance of proposed site work to allow for proper mobilization (including procurement of any necessary sampling equipment, sample containers, analytical standards, reagents, personal protective equipment, or other necessary equipment) and are required to consult with Geoprobe® operators in the development of such SAPs.

A site-specific SOP should be developed for on-site soil gas sampling and analysis, if needed, that includes details for the parameters of interest, GC configuration/setup, calibration, etc.

2. SAFETY AND HEALTH

All Terracon field work is carried out under the provisions of a safety and health plan. Most Geoprobe® work is covered by generic safety and health plans pertinent to intrusive work where petroleum hydrocarbons are anticipated or where there is a potential to encounter low concentrations of either petroleum hydrocarbons or chlorinated compounds. For any project involving greater potential hazard (e.g., work inside spaces with restricted ventilation and the potential to encounter high concentrations of volatile compounds, work involving the potential to encounter high concentrations of chlorinated compounds, or solvent extraction prior to analysis),

the Project Manager will ensure that a site-specific safety and health plan that includes proper safety procedures for the Project-specific hazards is obtained from the Corporate Safety and Health Manager prior to mobilization.

3. EQUIPMENT

Geoprobe® equipment may vary from unit to unit, but should carry the equivalent of the following equipment mounted on a mobile one ton van or truck:

1. Geoprobe® components -

a. GW-40 hydraulically powered probe.
b. 1 inch nominal diameter 2 to 4 feet length probe rods.
c. Soil piston samplers and acetate liners.

2. GC components[1] -

a. Shimadzu GC-14A (or equivalent) laboratory quality GC.
b. Shimadzu (or equivalent) electron capture detector (ECD).
c. Shimadzu (or equivalent) flame ionization detector (FID).
d. Photoionization detector (PID).
e. 30 m x 0.53 mm ID Supelco 3 mm Vocol (or equivalent) capillary column.
f. Shimadzu (or equivalent) gas flow controller.
g. Compressed air (service to FID).
h. Compressed hydrogen (service to FID).
i. Ultrapure compressed nitrogen or helium (carrier gas).
j. Regulators for compressed gas bottles.
k. Various size glass syringes (1 uL to 5 mL).
l. Appropriate analytical standards.
m. 40 mL nominal volatile organic analysis (VOA) vials.

3. Data system components -

a. APEX CSI (or equivalent) dual channel A to D converter.
b. Laptop computer.
c. APEX data system program (Version 2.1) (or equivalent).

4. Miscellaneous equipment -

a. Scale.
b. Oven.
c. Fire extinguisher.

[1] *Current equipment is listed. Equipment may change as appropriate for the analytes of concern and to reflect new technological developments. For example, the PID is currently out of service and is expected to be replaced in the future. Therefore, specific information regarding it is not listed. The standard column currently in use is listed. However, other columns may be more appropriate for the analyte(s) of concern in a specific project.*

d. Various tools.
e. Clean distilled and/or deionized water (hereinafter referred to as distilled) water.

4. METHODS

A. Sample Collection and Holding

The Geoprobe® van, the working end of the hydraulic system (i.e., those portions of the system in contact with probe rods), all used probe rods, and all used sample contacting equipment will be cleaned prior to coming onsite for the first time in a project. Hydraulic lines will also be checked and tightened, if necessary, to ensure that no leaks are occurring. Clean probe rods will be used for each probe and used probe rods will be kept segregated from clean probe rods from the time they have been used until they have been cleaned. The working end of the hydraulic system will also be checked between probes and cleaned as necessary to reduce potential cross-contamination (see Section 4.D below regarding cleaning procedures). Soil collection will be in accordance with Terracon Standard Operating Procedure E.100, Surface and Near-Surface Soil Sampling–Grab and E150/155, Soil Sampling Volatiles/Terra Core™,

1. Soil Gas Samples (Onsite Analysis)

a. Soil gas samples provide semiquantitative information concerning concentrations of VOCs in soil gas at the time of sampling. These may be influenced by the proximity of sources of VOC contamination (e.g., vertical contamination in the unsaturated zone soil profile or contaminated ground water) as well as a variety of other factors (e.g., soil and VOC characteristics and ambient temperature).

b. Soil gas samples will be taken at a depth of at least 6 feet below ground level (BGL). When attempting to characterize contaminated ground water plumes, soil gas samples will be taken within 3 feet of ground water.

c. Soil gas probes will be placed into the ground in a manner that will maintain a seal between the probe and the surrounding soil.

d. The suitability of each sampling location will be determined by testing the air permeability of the soil from which the sample is to be taken. An amount of air equal to five to 10 times the internal volume of the probe will be extracted prior to sampling. If this volume cannot be drawn within 10 minutes, soil gas cannot be used to test for VOC contamination at that location.

e. The sample will be collected for analysis without opening the system to outside air.

f. The sampling stream will be completely free of elastomers and each component of the sampling stream shall be new and clean or have been cleaned prior to each use. The cleanliness of the sampling stream shall be verified by running analytical blanks as specified in this document.

g. Soil gas samples will be analyzed immediately after the sample has been obtained.

2. Soil Samples (On/Offsite Analysis)

a. Currently accepted standard procedures for sample collection, sample preparation, and analysis of VOC contaminated soil samples necessarily involve substantial losses. Therefore, results for such samples should only be considered semiquantitative. Additionally, quantitative comparison of results should take into account whether they are based on dry or wet sample weight. Generally, commercial analytical laboratories report on a wet weight basis. However, results may be reported on a dry weight basis under certain circumstances.

b. Soil samples will be obtained using any standard Geoprobe® sampler. Typically these consist of various size piston samplers. Preference is given to the use of large bore samplers with removable acetate liners. These are capable of recovering cores 24 to 48 inches long and 1-1/8 inches in diameter. Standard thin-walled samplers (Shelby tubes) may also be utilized.

c. A variety of containers are appropriate for the collection and storage of soil samples. These include the acetate liners noted above. When such liners are used, the ends must be sealed during storage prior to analysis and aliquots to be analyzed should be obtained from as near the center of the sample as possible. Soil samples to be sent offsite for analysis should be collected in clean, 4 ounce glass containers with Teflon-lined lids. Soil should be placed in these containers rapidly, with as little matrix disturbance as possible, and in a manner that minimizes headspace. The container should be tightly sealed immediately after the sample is placed in it. All soil samples should be preserved by cooling to 4 ^{o}C if they are not analyzed immediately after collection. Geoprobe® analysis of soil samples for VOCs must be completed within 24 hours of sample collection.

d. Soil samplers will be cleaned prior to initial use at a site. They will also be cleaned prior to each subsequent use. The cleanliness of soil samplers shall be verified by running analytical blanks as specified in this document.

3. Ground Water Samples (On/Offsite Analysis)

a. Ground water samples obtained using Geoprobe® equipment can be expected to contain substantial concentrations of sediments. Therefore, they should be considered equivalent in character to borehole water samples.

b. Ground water samples will normally be collected by vacuum extraction using the same probe rods and tubing utilized for soil gas sample collection. The Geoprobe® may also be used to install small diameter slotted well points or push screen point samplers. Additionally, ground water samples may be obtained through probe rods or from slotted well points using stainless steel mini-bailers.

c. Ground water samples will be placed in clean, glass 40 mL VOA vials with open caps and Teflon septums. The sample shall be placed into these vials rapidly, with as little

turbulence as possible, and in a manner that eliminates headspace. The container should be tightly sealed immediately after the sample is placed in it. All ground water samples should be preserved by cooling to 4 ^{o}C if they are not analyzed immediately after collection. Geoprobe® analysis of ground water samples for VOCs must be completed within 24 hours of sample collection.

d. The sampling stream will be completely free of elastomers and each component of the sampling stream shall be new and clean or have been cleaned in accordance with the procedures specified in this document prior to each use. When reusable equipment is used (e.g., stainless steel mini-bailers), it will be cleaned prior to initial use and each subsequent use. The cleanliness of the sampling stream shall be verified by running analytical blanks as specified in this document.

Probe holes will generally be abandoned by backfilling with bentonite pellets immediately after rods and samplers have been withdrawn. In the event more restrictive project, local, or state plugging requirements exist, they will be identified prior to initiation of field work and complied with.

B. Sample Preparation and Analysis (Onsite Analysis)

Manufacturer instructions will be complied with in the operation of the GC and ancillary equipment for the analysis of environmental samples. Information from the scientific literature will also be relied on for guidance. Selection of columns, GC operating parameters, and detectors will be consistent with and appropriate for the types of analytes of concern.

1. Soil Gas Samples

Soil gas samples are obtained with a glass syringe and run by direct injection into the GC. No other sample preparation is required. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards. Soil gas samples must be run at the time they are obtained. They may not be held for subsequent analysis.

2. Soil and Groundwater Samples

a. VOCs

1) VOCs must be extracted from the soil or ground water matrix for analysis by GC. This may be accomplished by purge and trap or headspace procedures. The normal Geoprobe® procedure will be heated headspace. Research indicates that results by either approach can be expected to correlate well in the case of water samples that do not contain substantial concentrations of sediments. Data pertaining to the effect of sediments is lacking; however, purge and trap would be expected to be a more effective extraction procedure than headspace in the case of VOCs that are more likely to be adsorbed (i.e., having higher log octanol/water partition coefficients). Differences in the efficiency of extraction procedures for specific VOCs should be considered when comparing Geoprobe® and analytical laboratory results for split samples.

2) Soil Heated Headspace Analysis

a) Weigh a clean, dry, 40 mL VOA vial (with the top off). Record this weight to the nearest 0.1 g. The approximate mean weight of such vials is 24.1 g.

b) Place approximately 5 g of the soil sample in the vial and obtain their combined weight. Record this weight to the nearest 0.1 g and proceed to the next step (the combined weight minus the empty vial's weight is the weight of the sample). In the case of heavily contaminated samples, the mass of sample used can be reduced to keep the response within the range of the calibration curve and bracketed by standards.

c) Using a graduated cylinder, add 20 mL of clean, distilled water into the vial with the sample and cover it snugly by screwing on the vial's open top cap and Teflon seal.

d) Shake the sample to attempt to break the soil up.

e) Heat the sample in an oven at 60 ^{o}C for 15 minutes to facilitate volatilization of VOCs from the sample into the vial headspace.

f) Using a clean glass syringe, obtain a headspace gas sample through the Teflon seal for direct injection into the GC. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards.

3) Ground Water Heated Headspace Analysis

a) Using a clean graduated cylinder, place 20 mL[2] of the ground water sample into a clean 40 mL VOA vial and cover it snugly by screwing on the vial's open top cap and Teflon seal.

b) Heat the sample in an oven at 60 ^{o}C for 15 minutes to facilitate volatilization of VOCs from the sample into the vial headspace.

c) Using a clean glass syringe, obtain a headspace gas sample through the Teflon seal for direct injection into the GC. The volume of the injection can be varied to keep the response within the range of the calibration curve and bracketed by standards.

b. Various chemical extraction methods are available which may be used for SVOCs. Selection of an appropriate method will be made on a case-by-case basis. Whenever such a method is used, quality assurance (QA) measures will be utilized to evaluate its effectiveness (e.g., submittal of duplicate samples for laboratory confirmation or analysis of soil

[2] *The intention is to fill the vial to a volume exactly the same as that used for standards and approximately half full. Since the actual volume of these vials is approximately 44 mL, this will make the volumetric concentration of headspace gas slightly less than that of the ground water sample, if all VOCs can be driven from the aqueous to the gaseous phase. However, aqueous phase samples and standards having the same contaminant concentrations should produce equal gas phase concentrations.*

media certified performance evaluation samples) and appropriate provisions will be incorporated within the project safety and health plan.

3. Temperature Programs

The temperature program utilized must be appropriate for the analyte(s) of concern. Standard programs are as follows:

a. Chlorinated Solvents

1) Injector 225 ^{o}C
2) Initial column[3] 35 ^{o}C for 2 minutes
3) Column ramp rate 10 ^{o}C/minute
4) Final column 100 ^{o}C for 2 minutes
5) ECD and PID 250 ^{o}C

b. Petroleum Hydrocarbons

1) Injector 225 ^{o}C
2) Initial column 60 ^{o}C for 2 minutes
3) Column ramp rate 10 ^{o}C/minute
4) Final column
    1) BTEX/Gas TPH 150 ^{o}C for 2 minutes
    2) Diesel TPH 200 ^{o}C
5) FID and PID 250 ^{o}C

4. Compressed Gas Flow Rates

The standard column nitrogen gas flow rate is 10 mL/minute. The standard FID gas flow rates are 20 mL/minute for air and 4 mL/minute for hydrogen.

C. Analytical Quality Control and Assurance

Quality control (QC) and assurance (QA) terms have been variously defined. In this procedure they are defined as follows: QC consists of those activities performed for the purpose of controlling analytical quality; and QA consists of those activities performed for the purpose of providing assurance that analytical quality is in fact being achieved. By these definitions, QC activities include training of personnel, utilization of standard procedures, maintaining clean conditions through the use of new or properly cleaned equipment and reagents, maintenance of equipment, calibration activities, and documentation. QA activities include analysis of blanks, analysis of known concentrations or spikes (including surrogates), analysis of replicate samples, analysis of evaluation samples, and audits. QA activities provide evidence that the analytical process is under control and capable of producing suitably unbiased, accurate, and precise results.

[3] *When ambient air temperature exceeds 75 ^{o}F the initial column temperature will be 45 ^{o}C.*

1. Quality Control

A primary QC requirement is that GC operators be properly trained and knowledgeable. However, operator training will not be otherwise addressed in this document. The purpose of this procedure is to help ensure that standard methods are available and implemented. Equipment cleaning procedures are specified in Section 4.D of this document. In general, maintenance of all equipment will be performed as specified in manufacturer instructions. Documentation requirements are specified in Section 5 below. Minimum calibration requirements are as follows:

a. Standards for calibration will be prepared from pure standard materials or purchased as certified solutions. Standards will be prepared in methanol. Standards will be stored with minimal headspace, at 4 ^{o}C, and protected from light. All standards must be replaced after six months, or sooner if comparison with check standards or other QA measures indicate a problem.

b. Initial calibration. When new equipment has been placed in operation, an initial calibration curve will be generated. A minimum of three standard levels will be used for initial instrument calibration when either an FID or PID is in use. When an ECD is used, a minimum of five standard levels will be used.

c. Continuing calibration. The calibration curve will be verified each working day by the injection of one or more calibration standards prior to analysis of any samples. If the response for analytes is within the range of 80 to 120 percent of that predicted, sample analysis may continue with the same calibration curve. If recovery is outside of that range, a new calibration curve will be prepared for the analyte involved by running two additional standards. Calibration standards will be run at a frequency of at least one for every 10 samples. When an ECD is in use, this frequency will be increased to one for every five samples. When a new calibration curve is required, results for all samples which have been run since the last satisfactory continuing calibration will be appropriately qualified to indicate that circumstance.

d. The type of calibration standard will be appropriate to the type of sample being analyzed. Gas phase standards will be used when analyzing soil gas and aqueous phase standards will be used to produce headspace gas for injection when analyzing soil and ground water samples. The working range will generally be defined by initial calibration standards of the following concentrations -

1) Gas phase: 15, 30, and 60 ug/L for all detectors. These concentrations will be achieved by varying injection volumes from a single concentration standard.

2) Aqueous phase: 5, 25, and 100 ug/L for FID and PID and 1, 5, 10, 25, and 50 ug/L for ECD. These concentrations will be achieved by a combination of varying the mass of standard injected into distilled water and/or injection volumes.

e. The calibration curve will be linear and pass through the origin. Additionally, the calculated correlation coefficient for it shall be 0.95 or greater.

f. Other combinations of calibration standards may be used if information on site conditions indicates they would produce equivalent or better results.

Additional QC requirements are as follows:

a. Glass syringes may be used on one sample per day. After use, they must be cleaned prior to reuse. Cleaning is accomplished by washing as necessary, rinsing with hexane and methanol, and baking at a temperature of at least 60 ^{o}C for at least 15 minutes.

b. The GC sample injector port septum will be replaced regularly, depending on use, to prevent possible gas leakage.

c. When pulling gas or aqueous phase samples from probes, tubing will be connected to the vacuum pump via an adaptor which does not come into contact with the sample stream. The adaptor will be replaced periodically as necessary to ensure cleanliness and a good fit. At the end of each working day, adaptors which have been used will be cleaned with a detergent solution, rinsed with control water, and baked at a temperature of at least 60 ^{o}C for at least 15 minutes prior to being reused.

2. Quality Assurance

a. Method Blanks

Method blanks will be analyzed for the purpose of evaluating process bias. The following method blanks will be performed:

1) A method blank will be run at the beginning and, generally, near the end of each day of operation. However, the second method blank will not be run if cleaning of sample contacting equipment has not been performed or if results for a site are predominantly low or below detection limits. The method blank will include final rinse water from cleaning of probe rods and/or sample contacting equipment and aliquots of any reagents used in sample preparation. Distilled or "control water" will be substituted for final rinse water if the latter is unavailable.

2) If analytes are detected in a method blank, the source of the contamination will be identified and measures instituted to eliminate it. Results for any project samples which have already been run since the last clean method blank will also be evaluated to determine the impact of this circumstance. If the analytes involved were detected in them, they will be rerun. Otherwise, they will not be rerun.

b. Known Concentrations or Spikes

Continuing calibration standards will be utilized as known concentrations for the purpose of evaluating process accuracy and bias. Surrogates and matrix spikes will not normally be analyzed. If surrogates or matrix spikes are analyzed, the acceptance range is 80 to 120 percent recovery. Results within this range indicate an acceptable level of accuracy. If results are outside of this range, data must be appropriately qualified to indicate that circumstance and corrective action taken to regain process control.

c. Replicates

For every 10 samples (or at least once per day), a replicate shall be run for the purpose of evaluating process precision. Replicates shall be carefully prepared to minimize sampling variation as a source of error. All available site information shall be utilized to ensure that detectable concentrations of analytes are present in replicates. If detectable concentrations of analytes are not present in samples, a replicate of at least one continuing calibration standard per day shall be run. Results of 25 percent or less relative percent difference (RPD) indicate an acceptable level of precision. The RPD for comparison with this criterion is calculated as the absolute difference between replicate results divided by their mean. If results exceed this RPD, data must be appropriately qualified to indicate that circumstance and corrective action taken to improve process control.

d. Performance Evaluation Samples

The mobile laboratory QA Officer will ensure that at least one certified performance evaluation sample (water media) per quarter shall be run on a single blind basis. The nature of such samples shall take the general sample load into consideration. When target compounds are predominantly volatile petroleum hydrocarbons, most performance evaluation samples will consist of the compounds benzene, toluene, ethylbenzene, and xylenes (BTEX). At least once a year, a performance evaluation sample (water media) for selected volatile halocarbons will also be run.

e. QA Audits

QA audits will be performed by the mobile laboratory QA Officer on an on-call and project-specific basis. The file on at least one completed mobile laboratory project involving onsite GC analysis will be randomly selected for audit each quarter. At or about the same time, the mobile laboratory QA Officer will visually inspect the mobile laboratoryand all onboard associated equipment.

The mobile laboratory QA Officer will prepare a report following each calendar year for submittal to Terracon. It will include the following information for that period:

1. Performance evaluation sample results.
2. Routine project file audit results.
3. On-call project-specific audit results.
4. Mobile laboratory van visual inspection results.

D. Equipment Cleaning

Cleaning of field equipment will be performed in accordance with ASTM Standard D 5088-90. At a minimum, this means that probe rods and sample contacting equipment will be washed with a detergent solution and rinsed with "control water". Control water is defined as water having a known chemistry. Water from any public water supply operating in compliance with the Safe Drinking Water Act should meet this requirement. It will generally not be necessary to document the quality of control water unless there are unresolved method blank detections. Trihalomethanes are the volatile contaminants most likely to be encountered in control water.

Where more rigorous cleaning procedures are necessary, they will be specified in the site-specific SAP.

5. DOCUMENTATION

A separate project file (alpha identifier) will be maintained for Geoprobe® projects. This file will include the following minimum documents:

1. That portion of the project SAP covering Geoprobe® work.
2. The site-specific safety and health plan for all work requiring one.
3. A short narrative project report to include -
    a. A summary of field work performed.
    b. A summary of field methods actually used if there were any substantial deviations from or changes to the SAP.
    c. Reasons for any changes to the SAP.
    d. A site diagram with sufficient detail or information to approximately identify the location of all probes performed and/or samples obtained.
    e. The identity of onsite Terracon project personnel.
    f. The identity of onsite client project personnel.
    g. The identity of onsite regulatory or other significant personnel.
4. Results for each sample run (including calibration and QA samples). Results include the chromatogram obtained and output values determined for analytes (i.e., retention time, area, peak height, and concentration). The source of calibration should be identified for all samples.
5. The initial calibration curve, results of continuing calibration standards, and any subsequent calibration curves.
6. Results for all QA samples including calculated recovery and relative percent difference (RPD) values.
7. Other relevant project data.

Terracon

STANDARD OPERATING PROCEDURE

E.4000
SAMPLING OF POTENTIAL LEAD-BASED PAINT

Last Review or Revision: September 2011

I. PURPOSE

The purpose of this standard operating procedure (SOP) is to provide information on the hazards of lead-based paint (LBP), as well as to describe procedures for identification and proper sampling techniques of LBP. The following procedures for sampling potential LBP using a direct reading instrument and paint chip collection are designed to provide standardization with respect to location and number of samples collected and method of labeling sample locations. In addition, this procedure should ensure that potential LBP samples are collected in a manner which allows for accurate analysis of the material. Finally, this procedure will help to ensure that sampling personnel are protected against potential lead dust releases through controlled sampling techniques or appropriate use of personal protective equipment.

The objective of sampling coated (i.e., painted, pigmented or stained) surfaces for LBP (LBP) is to characterize materials and components that contain lead in surface coatings and compare them to established limits such as the United States Environmental Protection Agency (USEPA) or Housing Urban Development (HUD) standards and guidelines such as at quantities greater than 1.0 milligram per square centimeter (mg/cm^2) or 0.5% by weight or other regulated quantity as specified by state or local authority. By characterizing the locations and quantities of LBPs, exposure hazards can be greatly reduced.

II. BACKGROUND AND REFERENCE

Lead is a toxic heavy metal which may cause blood, kidney and nervous system disorders if inhaled or ingested. Metallic lead and lead contained in dusts are not readily absorbed through the skin. However, skin contact with potentially contaminated site materials should be avoided.

The USEPA and HUD have established an action level for LBP of 1.0 milligram per square centimeter (mg/cm^2) or 0.5% by weight. Coatings with quantities equal to or greater than these values are considered LBP. The Occupational Safety and Health Administration (OSHA) does not establish a LBP quantity but regulates the amount of lead that can become airborne and either inhaled or ingested by setting limits for air concentrations of

0.05 milligram per cubic meter (mg/m^3) of air over a work shift and blood values of 50 micrograms per deciliter of blood.

Lead was a common ingredient in paint until 1978 when the Consumer Product Safety Commission (CPSC) banned the sale of LBP for use in residences in quantities greater than 0.06% by weight. However, some industrial paints still contain lead today and are used in several applications. Lead can be introduced into the air by sanding or abrading surfaces containing LBP, and inhalation or ingestion of the dust is possible. Ingestion of lead can occur when children consume deteriorated paint chips, children place exposed toys or body parts into their mouths or when industrial workers exposed to lead-containing dust eat or smoke without washing their hands.

The lead content of paint can be determined with direct-reading instrumentation or by analysis of a bulk paint chip sample. An X-ray fluorescence (XRF) type analyzer is recommended to obtain direct readouts of lead content in coated surfaces. The XRF analyzes for lead by atomic absorption spectroscopy (AAS). Results are in milligrams of lead per square centimeter. Paint chip sample analysis is performed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) in accordance with EPA SW-846 Method 6010B. Results are typically reported as lead percent per paint chip weight.

When sampling for LBP in buildings classified under the United States Department of Housing and Urban Development (HUD) or other child occupied-facilities (i.e., publicly and privately owned-housing, public buildings, daycares, etc.), procedures stated in the most current revision of the Department of Housing and Urban Development *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards Housing*, June 1995 revision will be used.

Lead is also regulated by the OSHA and the USEPA. Occupational exposure to lead occurring in the course of construction work, including maintenance activities, painting, renovation and demolition, is subject to OSHA standard (29 CFR 1926.62), Lead Exposure in Construction. Construction work covered by 29 CFR 1926.62 includes any repair or renovation activities or other activities that disturb in-place, lead-containing materials. Employers must assure that no employee will be exposed to lead at concentrations greater than 0.05 mg/m^3 averaged over an eight-hour period without adequate protection.

The Resource Conservation and Recovery Act (RCRA) provides the USEPA with the authority to regulate the waste status of demolition or renovation debris, including lead-containing materials. Specific notification and testing requirements must be addressed prior to transporting, treating, storing or disposing of hazardous wastes. Lead containing wastes are considered hazardous waste under RCRA if Toxicity Characteristic Leachate Procedure (TCLP) results exceed 5 milligrams per liter (mg/L). USEPA exempts from most RCRA requirements those generators whose combined hazardous waste generation is less than 100 kilograms (kg) per month.

III. EQUIPMENT

LBP analysis can be conducted by using a direct reading XRF analyzer or by collecting paint chip samples. Equipment necessary to conduct both methods is listed below.

For direct reading sampling:

- X-ray fluorescence (XRF) analyzer and accessories
- XRF result field log (optional if downloading software is used)

For paint chip sampling:

- Heating tool and extension cord
- Tape measure or template
- Chisel
- Chipping hammer or scraper (lead paint samples from metal structures)
- Face shield or chipping goggles
- Sample containers (preferably sturdy, clear plastic vials)
- Sample labels
- Laboratory chain of custody for paint chip sample analysis

IV. CERTIFICATION

Individuals conducting LBP inspection services should have the certifications listed below. Copies of these certifications and licenses should be taken to the site.

- USEPA lead inspector and/or risk assessor certification
- As applicable, local- or State-licensed lead inspector and/or risk assessor (required for HUD projects)
- Manufacturer training certification for the XRF analyzer

V. HEALTH AND SAFETY

The OSHA personal exposure limit (PEL) for lead is 0.05 mg/m^3 and the action level is 0.03 mg/m^3. The primary route of exposure of lead is through inhalation of contaminated dusts or by accidental ingestion; however, collection of small sample volumes required for analysis is not expected to generate significant dust. Although if painted surfaces are being disturbed and dust is generated in the vicinity, personnel will take protective measures as indicated below. Project activities may be conducted in Level D personal protective equipment modified as specified below.

- Lead sampling activities will be performed in Level D personal protective equipment to include standard work uniform, safety footwear and hard hat if overhead hazards are present.
- Protective goggles or a full face shield will be worn during chipping hammer operations.
- Protective gloves should also be worn during lead chip sampling to prevent abrasion and contact with site materials. Half face disposable dust/fume/mist respirators (3M 9920 "surgical style" masks) or half-face air purifying respirators equipped with HEPA filter cartridges will be worn if dusty conditions develop on-site.

Due to the potential of accidental ingestion when working in areas with lead dust, do not smoke or eat with soiled hands. Wash thoroughly before eating, drinking or smoking. Shower thoroughly as soon as possible upon leaving the site.

The XRF analyzer contains a radioactive source and should be transported and used according to the manufacturer's instructions. Personnel utilizing the equipment shall have the proper training and certifications required for use of the equipment.

VI. PROCEDURES FOR XRF ANALYSIS

1. Identify areas with coated or prepared surfaces. This includes building materials, components and fixtures finished with a coating such as paint, stain and varnish. Wallpaper can mask prepared surfaces and should be included in the survey. Some ceramic tiles have a lead-containing glaze and should also be assessed, particularly where required by state or local regulatory agencies.

2. Select appropriate materials and locations to be sampled. When sampling painted interior surfaces, representative samples must be obtained per client instruction or in general compliance with the most current revision of the HUD *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards Housing*, June 1995 revision.

3. Calibrate the XRF unit according to the manufacturer's instructions before and after the survey from a known source provided by the manufacturer. One set of three nominal-time XRF calibration check readings will be taken before the inspection begins and a second after inspection work has been completed for the day, or at least every 4 hours, whichever occurs first. XRF calibration check readings will be taken on Standard Reference Material (SRM) paint film nearest 1.0 mg/cm² within the National Institute of Standards and Technology (NIST) SRM used. Calibration checks should be taken through the SRM paint film with the film positioned at least one foot away from any potential source of lead. The NIST SRM film should be attached to a solid wood board or other nonmetal rigid substrate such as drywall, or attached directly to the XRF probe. If readings are outside the acceptable calibration check range (0.7 to 1.3 mg/cm^2), follow the manufacturer's instructions to bring the instruments into control before XRF testing proceeds. Note: The XRF monitors its own

internal spectrum and makes self-adjustments as necessary. Thus, an operator does not need to perform any system calibrations in the field.

4. Conduct XRF sampling on selected surfaces. Document descriptions of each surface sampled on the XRF result field log or using the manufacture's software. Record results on the XRF result field log if software is not used.

If results yield 1.0 mg/cm^2 (inconclusive), then a paint chip sample should be collected using the applicable procedures indicated in the following section.

If results indicate the presence of lead in quantities greater than 1.0 mg/cm^2, it is recommended that a photograph depicting the material and location be taken.

VII. PROCEDURES FOR PAINT CHIP SAMPLING

1. Identify areas with prepared surfaces. This includes building materials, components and fixtures finished with a coating such as paint, stain and varnish. Wallpaper can mask prepared surfaces and these areas should be included in the survey.

2. Select appropriate materials and locations to be sampled. When sampling painted interior surfaces, representative samples must be obtained per client instruction or in general compliance with the most current revision of the HUD *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards Housing*, June 1995 revision.

3. A heating tool (using a low temperature (below 1100 degrees F)) and chisel is recommended to collect representative samples of painted surfaces, but a knife, chipping hammer or paint scraper may also be used. Minimum force should be used to prevent the generation of dusts and particles. Wear protective goggles, abrasion resistant gloves and/or particulate respirator as appropriate to the task.

Check with the analytical laboratory to determine minimum sample size required. A two square inch (2 in^2) sample is recommended for each sample. The sample size should be documented on the sample log. Some laboratories conducting TCLP analysis may request up to ten grams (10 g) per sample. LBP samples must be removed down to the bare substrate to ensure each layer of paint has been collected. Use a brush or mini-vacuum to clean up residual material and place it in the sample container.

4. Assign a sample number to each sample collected. Affix a label or mark the sample container indelibly with a sample identification number. Seal the sample container securely. Document descriptions and locations of each surface sampled on a field log. It is recommended that a photograph depicting the material and location be taken.

5. Send results and chain or custody to a laboratory accredited by the AIHA Laboratory Accreditation Programs, LLC under the Environmental Lead Laboratory Accreditation Program (ELLAP) for analysis.

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STANDARD OPERATING PROCEDURE

E.468
SAMPLE HANDLING - SOIL (LEVEL D)

Last Review or Revision: August 2011

1. OBJECTIVE

   To obtain a representative soil or sediment sample for chemical analysis. This includes the documentation of sampling methods, and protocols used for sample collection, processing, handling and shipment.

2. EQUIPMENT

   - Monitoring equipment (HNU, OVA, OVM, TGI, TIP, FID color metric detector tubes) as specified by Project Manager;
   - Sampling Device (split barrel sampler, hand auger, hand trowel, shovel, posthole digger, tube sampler, or other appropriate sampling device);
   - Decontamination Equipment;
   - Laboratory prepared sample containers;
   - Forms including "Soil Sampling Information Sheet", chain-of-custody, etc;
   - Indelible ink pen;
   - Stainless steel bowl;
   - Plastic sheet;
   - Site map;
   - Measuring wheel;
   - Engineers tape marked in units of feet, tenths of a foot (0.1 ft.), and hundredths of a foot (0.01 ft.);
   - Tool box;
   - Disposable chemical-resistant gloves; and
   - Chem-wipes.

3. PROCEDURES.

a) Surficial soil/sampling

- Determine sample location (set grid, if necessary)
- Determine the proper sampling device based on soil type, depth, sample type, etc.
- Collect each sample at the specified depth consistently for each sample.

b) Direct Sampling

- Transfer sample directly from the sampling device to the sample container. Samples collected for analysis of volatiles should be collected first with minimal disturbance to the sample (refer to SOP E.150/155). The remainder of the soil sample should be mixed into a homogenous mixture and placed into the required sample containers.
- If evaluating for organic vapors, transfer half of sample to glass mason jar or plastic bag (zip top) for field testing. The sample should be split so as to obtain a sample for screening that is representative of the sample for testing. This can be accomplished by slicing the sample (if cohesive) lengthwise or by using other mechanical means. Care should be taken so as not to over-agitate the sample, especially if volatile organic compound testing is required.
- Document visual and physical characteristics

c) Composite sampling (non-volatile only)

- Decreases analytical cost but also decreases ability to detect low level contamination
- Transfer equal volume/weight of sample from each location/depth to a stainless steel mixing bowl
- Use a hand trowel or spoon to mix the soil sample
- If the sample size is very large, composite on a large sheet of clean plastic or stainless steel cookie sheet pan, or mix equal volumes from numerous composite samples.
- If soils are cohesive, break up clumps.
- Spread soil uniformly on plastic sheet or in bottom of stainless steel bowl or stainless steel tray and divide into quarters.

- Obtain equal quantity of soil from each sample for transfer to sample container (without mixing or break up).

d) Decontamination

- Decontamination procedures should be specified by the project manager.
- Decontamination procedures for UST sites includes an Alconox® detergent scrub followed by a clean water rinse.
- Decontamination fluids are to be replaced between sample locations (each boring) to reduce the potential for cross contamination.

e) Sample preservation - store in cooler with ice.

f) Sample documentation

- Complete the "Soil Sampling Information Sheet" and chain-of-custody form. Date to be recorded includes sampling location, methodology, depth, visual and physical characteristics, time and date.

4. ATTACHED SUPPORTING DOCUMENTATION

a) ASTM D4220 *Practice For Preserving and Transporting Soil Samples*

5. OTHER REFERENCES

a) Laboratory- or program-specific requirements for handling, preservation, and transport of samples for chemical analyses.

EXAMPLE SOIL SAMPLING INFORMATION SHEET

PROJECT NAME _____ PROJECT NO. _____

PROJECT LOCATION _____

SAMPLE POINT _____ DATE _____ TIME _____
SAMPLE POINT DESCRIPTION _____
SAMPLE METHOD _____
SAMPLE INTERVAL _____
SAMPLE DESCRIPTION _____
SAMPLE APPEARANCE _____
ORGANIC VAPOR READING _____
SAMPLING PROBLEMS _____
CLEANING PERFORMED IN FIELD _____
COMMENTS _____

SAMPLE POINT _____ DATE _____ TIME _____
SAMPLE POINT DESCRIPTION _____
SAMPLE METHOD _____
SAMPLE INTERVAL _____
SAMPLE DESCRIPTION _____
SAMPLE APPEARANCE _____
ORGANIC VAPOR READING _____
SAMPLING PROBLEMS _____
CLEANING PERFORMED IN FIELD _____
COMMENTS _____

FORM COMPLETED BY: _____ DATE _____

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STANDARD OPERATING PROCEDURE

E.470
SAMPLE HANDLING – GROUNDWATER (LEVEL D)

Last Review or Revision: August 2011

OBJECTIVE

To collect a representative groundwater sample from the sampling point for chemical analysis. This includes the documentation of sampling methods, sampling supplies, and protocol to reduce potential for alteration and or cross-contamination during the sampling event.

EQUIPMENT

- Monitoring equipment specified by project manager;
- Electronic water level indicator, phase level indicator, etc.;
- Decontamination equipment;
- Proper forms, labels and indelible ink pen;
- pH, temperature, and specific conductance meter;
- Sample containers and packing material, tape, and labels;
- Filtration device and filters (and fixing agents as appropriate);
- Cooler with ice pack and packing media;
- Bucket (calibrated in gallons or liters)
- Sampling device
- Bailers, pumps, etc.
- Keys for locking cap on well;
- Rope - steel, nylon, teflon, or polypropylene;
- Deionized (DI) water;
- Chemical-resistant gloves
- Knife; and

- Site map.

PROCEDURES

a) Preparation

- Meet with Project Manager;
- Obtain the bottles, forms, and equipment necessary to complete the sampling event;
- Calibrate all field equipment i.e., pH and conductivity meters;
- Establish sampling well sequence (generally least impacted to most impacted).

b) Field Activities

- Open the manway and remove the well cap for each well. Allow at least 10 minutes for the groundwater level to stabilize in each well to atmospheric conditions.
- Using the electronic water level indicator, collect and record the water level from each well (and well depth, if requested by P.M.). The water level measurement should be taken from a marking/notch on the well PVC riser if present, if no notch is present the measurement should be taken from the side of the riser facing north for uniformity of data. The electronic water level indicator must be decontaminated after use at each well.
- Purge each well of 3 well volumes or until the well goes dry. Field measurements as requested by the Project Manager (i.e. pH, temperature, and specific conductance) will be measured during well purging. . Field equipment must be decontaminated after use at each well.

c) Contamination minimization

- Use plastic sheet if necessary
- Use proper bailing techniques (hand over hand) to prevent rope from touching the ground or low flow sampling techniques with dedicated disposable tubing

d) Sample Collection

1) Preservation

- Use containers with proper preservative if necessary.

- Routine preservatives are listed in the attached documentation (Check with Project Manager and/or lab).

2) Filtration

- Metals only - (Do not filter samples for VOC analyses).
- Field filter samples collected for dissolved metals analyses immediately after collecting the sample (if required and/or allowed by local or state regulations).
- Filter the sample prior to adding preservative or transferring to sample container containing a preservative(if required and/or allowed by local or state regulations).
- Discard the filter, tubing and transfer container.

3) Collection

- Refer to SOP E.1400 for stabilization of water quality parameters prior to sample collection.
- Minimize disturbances that may aerate the sample (i.e., lower bailer slowly into water, pour slowly into sample container, use low flow sampling procedures, etc.). Refer to SOP E.2000 if low flow sampling procedures will be utilized.
- Pour water from the top of the bailer or insert the bottom emptying device for sample transfer.
- Transfer the groundwater sample directly to the laboratory prepared sample container or the filter cup.
- Samples collected for VOCs should always be collected from a recently filled bailer full of water as soon as it is brought to the surface.

- Collect samples for VOCs by forming a positive meniscus on the sample vial and capping immediately.
- VOC samples must be free of air bubbles. If air bubble(s) persist within the vial, note in the field log and on the chain-of-custody to notify the laboratory that the sample is unpreserved.
- Do not over-fill sample containers which contain a preservative.
- Place samples in cooler with ice or blue-ice.

4) Filtration Procedures

- Set up filtering apparatus according to the manufacturer's directions.
- Use a 0.45 micron membrane filter (may need a pre-filter to prevent clogging if the sample is turbid).
- Flush a minimum of 250 ml of D.I. water (or larger volume if recommended by manufacturer specifications) through the filtering apparatus and filter prior to filtering the sample.
- Pump the sample through the filter and discard the initial 100-200 ml (if you have sufficient volume).
- Collect and transfer the remaining sample volume to the sample container.

5) Data Documentation

- Record all pertinent sampling information on the sampling container label, sampling information form, chain-of-custody, and shipping form.
- Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, sampling methodology, parameters to be analyzed, stabilization data, and general observations.
- Make all entries in the chain-of-custody form prior to leaving the site. Ensure that the chain-of-custody protocol required for the project is maintained.
- If samples must be shipped, the chain-of-custody form must be enclosed with the samples and the container sealed with Terracon security labels. Obtain a post office receipt, bill of lading or similar document from the shipper to be included as part of the chain-of-custody documentation. Return one copy of the chain-of-custody documentation to the project manager.

6) Equipment Cleaning

- Clean all equipment used as specified by the Project Manager and according to cleaning procedures prior to collecting a sample from the next sample location.

7) Sample Packaging and Shipment

- Carefully package the samples in a cooler with ice. Take care to wrap the sample containers in packing materials and place in sealed Zip top bags.

- Ship the samples via overnight courier as specified by the Project Manager. Be sure to secure all address labels with clear packing tape.

Attached Supporting Documents

- **Typical Chemical Analysis Methods and Preservatives**.

Other Supporting Documents

- **ASTM D4448-85a** *Standard Guide for Sampling Groundwater Monitoring Wells*.

Typical Chemical Analysis Methods and Preservatives

Analytical Test Method	Pilot Chemical Group	Sample Matrix			Sample Quantity (Minimum)										Preservative			Holding Time (Days)	Container Type
					Field Recovery Quantity			Laboratory Quantity											
		Soil	Water	Gas/Vapor	1500 grams	200 milliliters	2 Liters	400 milligrams	4 oz.	16 oz.	40 milliliters	200 milliliters	250 milliliters	500 milliliters	1 Liter	Cool 4°C, dark	pH<2, w/HNO_3	pH<2 w/HCL		
EPA 8260B	VOCs	X			X				X							X			14*	8 oz. CWM
			X				X				X							X	14*	40 mL GV
EPA 8270C	SVOCs	X			X				X							X			7**	8 oz. CWM
			X												X	X			7**	2 Liter AWM
EPA 8082	PCBs	X			X				X							X			7**	8 oz. CWM
			X				X								X	X			7**	2 Liter AWM
EPA 6010	Metals	X			X				X							X			180	4 oz. CWM
			X				X							X			X		180	500 mL HDPE
NIOSH 1501	Benzene, Toluene			X		X		X								X			14	Carbon Filter
EPA 9040A	pH	X			X				X							X			14*	4 oz. CWM
			X				X						X			X			14*	250 mL HDPE

* = Preserve within 2 days then analyze within 14 days
** = Extraction to occur within 7 days and analysis within 40 days after extraction
CWM = clear wide mouth
HDPE = High-density polyethylene bottles
GV = Glass vial
AWM = Amber wide mouth
AJ = Amber Jug

Terracon
STANDARD OPERATING PROCEDURE

E.480
SURFACE WATER SAMPLING

Last Review or Revision: August 2011

OBJECTIVE

To collect a representative surface water sample from the sampling point for chemical analysis. This includes the documentation of sampling methods, sampling supplies, and protocol to reduce potential for alteration and or cross-contamination during the sampling event.

This TSOP describes the procedure and equipment for collecting surface samples of water or other liquids using a dipper or equivalent. A pond sampler or dipper with extension handle allows the operator to sample streams, ponds, waste pits, and lagoons as far as 15 ft from the bank or other secure footing. The dipper is useful in filling a sample bottle without contaminating the outside of the bottle. This TSOP can be used to describe the sampling procedures to be used from a boat or from within the stream using hip-waders, however specific safety precautions should be implemented in these situations. See the project manager if sampling will not be conducted from the shore or other stable surface (boat dock, etc.).

EQUIPMENT

- Sampling and monitoring equipment specified by project manager;
- Disposable chemical-resistant gloves;
- Cleaning equipment;
- Proper forms, labels and indelible ink pen;
- Sample containers and packing material, tape, and labels;
- Cooler with ice pack and packing media;
- Dipper or equivalent sampling device; and
- Site map.

PROCEDURES

1. Clean sampling equipment in accordance with E.2410.
2. Locate sampling site at the designated point in the stream.
3. Attach extensions to the dipper as required to reach the distance from the shore specified by the project manager.

4. Submerge the container end of the dipper at sampling point such that mouth of dipper is at the depth specified by the project manager. If no depth has been specified, submerge the dipper about 2 to 3 inches below the water surface, if possible.
5. Allow the dipper to fill; rinse the dipper by shaking and discharging this water. Repeat this procedure three times.
6. Collect sample and transfer into a holding container or directly to the laboratory sample container. If a holding container is used for ease of transfer or to create a composite sample from multiple sampling locations, transfer water from the holding container into sampling bottles. If VOCs are included in the laboratory analysis, VOC samples must be free of air bubbles. If air bubble(s) persist within the sample container, note in the field log and on the chain-of-custody to notify the laboratory that the sample is unpreserved.
7. Fill out appropriate field form(s) documenting sample location, time, and other pertinent information before leaving sampling site.

OTHER SUPPORTING DOCUMENTS

ASTM D5358-93 *Standard Practice for Sampling with a Dipper or Pond Sampler*.

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STANDARD OPERATING PROCEDURE

E.500
pH FIELD SCREENING - SOIL

Last Review or Revision: June 2010

Objective and Application

To provide a qualitative and limited quantitative field screening of soil samples to aid in the evaluation of soil for acidity or causticity. This procedure is applicable for sites where moderately low or high pH soils are expected, such as coal pile storage.

Equipment

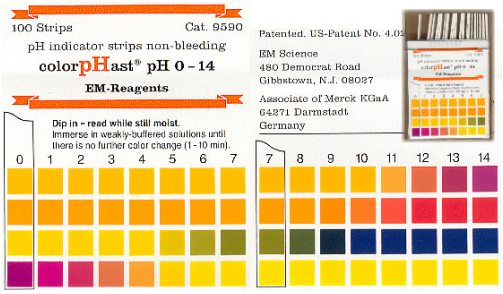
- Disposable chemical resistant gloves,
- Sterile container or clean zip top bag,
- ColorPHast® or equivalent litmus strips, and
- Deionized water.

Note regarding the ColorPHast® or equivalent litmus strips: These litmus strips are commonly available to cover a range of possible pHs. Typical ranges used for this procedure are 0-6, 5-10, and 7.5-14 standard units. Other ranges are also available. Before field mobilization, discuss the anticipated pH range of the soils to be tested and mobilize strips for the appropriate range. Strips should be stored in a dry location; it may be necessary to package the strips in a watertight zip top bag or equivalent before mobilization into the field.

Procedures

For each soil sample to be screened for pH, place a small (1oz.) amount of soil in a sterile container or clean zip top bag. Add an approximately equal amount of deionized water. Mix the solution until a slurry results or until all of the soil has been wetted. Place a litmus strip in the solution and allow the strip to become fully wetted. A test time of about 30 seconds is usually sufficient.

Compare the color of the test strip to the pH indicator chart on the front of the ColorPHast® package. The precision of the indicator chart will vary depending on the range of the strips used. The example shown indicates that for strips covering the 0-14 standard units range, a precision of 1 standard unit is available. Intermediate pH values may be estimated by



interpolating between the colors shown on the chart.

Documentation

Record the pH of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

Other Supporting Documents

ASTM D4972-01 *Standard Test Method for pH of Soils*.

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STANDARD OPERATING PROCEDURE

E.530
pH FIELD SCREENING - WATER

Last Revision or Review: June 2010

Objective and Application

To provide a qualitative and limited quantitative field screening of water samples to aid in the evaluation of water for acidity or causticity. This procedure is applicable for sites where moderately low or high pH water is expected.

Equipment

- Disposable chemical-resistant gloves,
- Sterile container or clean zip top bag,
- Corning Checkmate M90™ equipped with pH sensor,
- Corning Checkmate M90™ pH buffers (4, 7, and 10 standard units),
- ColorPHast® or equivalent litmus strips (to be used if pH meter is not being used or as a check of field equipment), and
- Deionized water.

Note regarding the ColorPHast® or equivalent litmus strips: These litmus strips are commonly available to cover a range of possible pHs. Typical ranges used for this procedure are 0-6, 5-10, and 7.5-14 standard units. Other ranges are also available. Before field mobilization, discuss the anticipated pH range of the water to be tested and mobilize strips for the appropriate range. Strips should be stored in a dry location; it may be necessary to package the strips in a watertight zip top bag or equivalent before mobilization into the field.

Procedures

- Install the pH sensor onto the Corning Checkmate M90™ meter.

    1. Locate the pins of the pH sensor and push firmly into the meter.
    2. Remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.

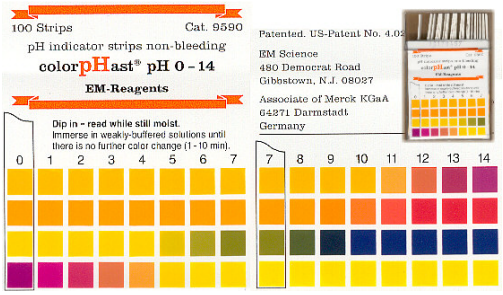
- Calibrate the pH meter at least daily in accordance with the manufacturer's specifications. The following procedure will calibrate the Corning Checkmate M90™ for pH measurements.

    1. Place the sensor in the calibrating medium (pH 7 buffer).

2. Press cal – cal 1 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.
3. Place the sensor in the second calibrating medium (pH 4 or 10 buffer). If samples are anticipated to be acidic, use the pH 4 buffer. If samples are anticipated to be basic, or caustic, use the pH 10 buffer.
4. Press cal – cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.

- For each water sample to be screened for pH, place a small amount of sample in a sterile container or clean zip top bag. Place the sensor into the sample. Automatic endpoint detection freezes the display when plateau is reached.

- ColorPHast® or equivalent litmus strips can be used to verify the results of the Corning Checkmate M90™ meter and to check if the meter requires re-calibration. Place a litmus strip in the sample and allow the strip to become fully wetted. A test time of about 30 seconds is usually sufficient. Compare the color of the test strip to the pH indicator chart on the front of the ColorPHast® package. The precision of the indicator chart will vary depending on the range of the strips used. The example shown indicates that for strips covering the 0-14 standard units range, a precision of 1 standard unit is available. Intermediate pH values may be estimated by interpolating between the colors shown on the chart.



Documentation

Record the pH of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

Attached Supporting Documentation

Corning Checkmate M90™ (or equivalent) Operating Instructions

Corning Checkmate M90™ (or equivalent) Sensor Information

Terracon
STANDARD OPERATING PROCEDURE

E.540
CONDUCTIVITY FIELD SCREENING - WATER

Last Revision or Review: June 2010

Objective and Application
To provide a qualitative and limited quantitative field screening of water samples to aid in the evaluation of water for the relative presence of ions in the sample.

Equipment
- Disposable chemical-resitant gloves,
- Sterile container or clean zip top bag,
- Corning Checkmate M90™ (or equivalent) equipped with conductivity sensor,
- Corning Checkmate M90™ (or equivalent) conductivity standard A or B (1413 uS or 12.88 mS), and
- Deionized water.

Procedures
- Install the conductivity sensor onto the Corning Checkmate M90™ meter.

    1. Locate the pins of the conductivity sensor and push firmly into the meter.
    2. Remove the sensor wetting cap and slide the vent sleeve to expose the fill hole.

- Calibrate the conductivity meter at least daily in accordance with the manufacturer's specifications. The following procedure will calibrate the Corning Checkmate M90™ for conductivity measurements.

    1. Hold the sensor in free air.
    2. Press cal – cal 1 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.
    3. Place the sensor in the second calibrating medium (conductivity standard A or B).
    4. Press cal – cal 2 is displayed. After endpointing the display automatically updates to the calibrated value shown, or the temperature compensated value.

- For each water sample to be screened for conductivity, place a small amount of sample in a sterile container or clean zip top bag. Place the sensor into the sample. Automatic endpoint detection freezes the display when plateau is reached.

Documentation

Record the conductivity of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

Attached Supporting Documentation

Corning Checkmate M90™ (or equivalent) Operating Instructions

Corning Checkmate M90™ (or equivalent) Sensor Information

STANDARD OPERATING PROCEDURE

E.550
Field Surface Screening – Soil / Photoionization Detector

Last Review or Revision: August 2011

Objective

To provide a qualitative and limited quantitative field screening of soil samples to aid in the evaluation of soil for the presence of volatile or semi-volatile organic chemicals.

The procedure is premised on the physical property of volatile compounds to move from the soil matrix of a freshly obtained soil sample (e.g., split spoon sample, grab sample) to the airborne state as vapor. The measurement of ambient air in close proximity to the surface of the soil will produce an indication of contaminants moving from the soil matrix to air. The relative strength of contaminants in ambient air around the sample will be considered an indication of the relative concentration of chemicals in the soil sample.

The procedure is semi-quantitative but valid only for the most preliminary qualitative decision-making. The procedure is highly susceptible to external air changes in wind velocity, ambient dilution by moving air and ambient influence by contaminants in moving air. The rate and degree of volatilization is susceptible to smearing of the exterior of the sample by the sampler wall in clay soils.

Measurements cannot be used as the sole indicator of soil contamination or in lieu of prescribed laboratory chemical testing for purposes of regulatory compliance. This procedure is only to be used for sites involving volatile or semi-volatile organic compounds, including tetrachloroethylene (perchloroethylene or PCE). This procedure <u>is not</u> to be used for purposes of health and safety monitoring.

Equipment

- Calibration gas from manufacturer
- Photoionization detector equipped with 10.0 eV lamp or greater.
- Forms and indelible ink pen
- Disposable chemical-resistant gloves

Procedures

On a daily basis, the unit should be gas calibrated to a manufacturer's gas standard and the results recorded in the field logbook. The unit is typically calibrated with isobutylene of 100 parts per million (ppm) concentration as the calibration gas. When checking the calibration, the unit should read within 5% of the span gas concentration used (e.g., between 95 and 105 ppm when 100 ppm span gas is used). If the unit does not calibrate, return it to the local Terracon equipment evaluation for evaluation and, if necessary, repair.

Immediately prior to making a field measurement the unit should be operated for approximately 1 minute and any background concentrations noted or zeroed out relative to measurements.

The opened sampler should be placed on disposable plastic material or a surface that can be cleaned between tests to avoid inadvertent contribution of ionizable vapor from previous tests.

Using a clean stainless steel knife or other cutting tool, slice through the sample to expose fresh soil material not smeared by the sampler or excavating equipment. Immediately begin the test procedure as follows.

- Place the tip of the PID probe between ½ and 1 inches from the surface of the exposed soil.

- Maintaining the proper and constant distance of the probe tip from the sample move the probe very slowly over the surface of sample material, giving good coverage to all portions of the sample.

- Total test time should be in excess of 1 minute for volatile chemicals of concern and in excess of 3 minutes for semi-volatile chemicals of concern.

- Record the highest reading obtained as parts per million (ppm) calibration gas equivalents (i.e., TEI580 calibrated to isobutylene would be expressed as ppmi). Record unusual fluctuations and any noticeable physical correlation to the sample (i.e., "highest readings at ½-1 feet below ground surface over a sand seam in the split spoon sample").

Sample Disposal

Soil samples should be returned to the site and included in auger soil or excavation soil for proper disposal.

Documentation

Record the highest reading in calibration gas equivalents on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded; date, job number, project name, sampling location, sample interval (if appropriate) sample identification, samplers name, and general observations.

Attached Supporting Documents

- **Thermo Environmental Instruments, Inc.** *OVM/Datalogger Model 580B Operating Manual*

STANDARD OPERATING PROCEDURE

E.552
Field Headspace Screening – Soil / Photoionization Detector

LAST REVIEW OR REVISION: August 2011

OBJECTIVE

To provide a qualitative and limited quantitative field screening of soil samples to aid in the evaluation of soil for the presence of volatile or semi-volatile organic chemicals.

The procedure is premised on the physical property of volatile compounds to move from the soil matrix to the airborne state as vapor. The amount of airborne material as vapor will be a relative concentration between samples if the volume of sample, volume of air, temperature and period of testing remain reasonably constant. The measurement is semi-quantitative for qualitative decision-making.

Measurements cannot be used as the sole indicator of soil contamination or in lieu of prescribed laboratory chemical testing for purposes of regulatory compliance. This procedure is only to be used for sites involving volatile organic compounds, including tetrachloroethylene (perchloroethylene or PCE). This procedure is not to be used for purposes of health and safety monitoring.

EQUIPMENT

- Calibration gas from manufacturer
- Photoionization detector equipped with 10.0 eV lamp or greater.
- Test chamber, reusable or disposable, examples:

    - Mason jar with aluminum foil
    - Mason jar equipped with charcoal filter
    - Ziploc® bags or other sealable container of at least 500 cubic centimeters to provide a fixed headspace volume for constancy between tests
    - Rigid, disposable concrete test cylinder mold with plastic cap

- Forms and indelible ink pen
- Disposable chemical-resistant gloves

PROCEDURES

On a daily basis, the unit should be gas calibrated to a manufacturer's gas standard and the results recorded in the field logbook. The unit is typically calibrated with isobutylene of 100 parts per million (ppm) concentration as the calibration gas. When checking the calibration, the unit should read within 5% of the span gas concentration used (e.g., between 95 and 105 ppm when 100 ppm span gas is used). If the unit does not calibrate, return it to the local Terracon equipment evaluation for evaluation and, if necessary, repair.

Immediately prior to making a field measurement the unit should be operated for approximately 1 minute and any background concentrations noted or zeroed out relative to measurements.

Prior to site testing an empty, unused test chamber should be sealed containing nothing but ambient air and allowed to stabilize for 1 minute. Test the chamber headspace to identify background contaminants contributed by the chamber itself. If anything is detected, change to another type of chamber which is inert relative to contributing ionizable materials to the headspace.

- Transfer soil sample representative of the condition to be measured from the sampling device to testing chamber (e.g., mason jar covered by aluminum foil, disposal concrete cylinder mold, Ziploc® bag, or other sealable container).
- Sample material should be representative of the vertical and horizontal cross-section of the sampled interval.
- The volume of soil should remain as constant as is practical for all site tests.
- The soil volume should not exceed 25% of the total volume of the air-filled testing chamber.
- Immediately seal the chamber after transfer and allow the sample to equilibrate for a minimum of 15 minutes at ambient temperatures above 50° F.
- Insert probe into sealed container for reading for 1 minute for volatile compounds of concern and 3 minutes for semi-volatile compounds of concern to account for varied response times.
- Record highest reading obtained as parts per million (ppm) calibration gas equivalents (i.e., TEI580 calibrated to isobutylene would be expressed as ppmi) .

SAMPLE DISPOSAL

Soil samples should be returned to the site and included in auger soil or excavation soil for proper disposal.

DOCUMENTATION

Record the highest reading in calibration gas equivalents on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded; date, job number, project name, sampling location, sample interval (if appropriate) sample identification, samplers name, and general observations.

OTHER SUPPORTING DOCUMENTS

- **Thermo Environmental Instruments, Inc.** *OVM/Datalogger Model 580B Operating Manual*

STANDARD OPERATING PROCEDURE

E.554
Field Screening – Air / Photoionization Detector

LAST REVIEW OR REVISION: August 2011

OBJECTIVE AND APPLICATION

To provide a qualitative and limited quantitative field screening of ambient air to aid in the evaluation of air quality relative to monitoring health and safety of workers involved in environmental field activities involving known or potential volatile or semi-volatile organic chemicals of concern.

The procedure is premised on the physical property of volatile compounds to move to the airborne state as vapor. The amount of airborne material as vapor will be a relative concentration dependent on air currents and dilution. The measurement is semi-quantitative for qualitative decision-making.

The intent is to monitor airborne concentrations of chemicals of concern in the breathing zone of workers. Unless otherwise specified by the health and safety plan, the breathing zone will be considered the height above ground surface generally corresponding from knees-to-chin to provide for upright, squatting and bending during field activity by workers.

This procedure is to be used primarily for purposes of health and safety monitoring. These readings are not a representative screening indication of the levels of chemicals of concern in soils or other media.

This procedure, though it may be used in combination with, does not replace similar methods and equipment used for confined space entry procedures. Use of this procedure as part of confined space entry testing must be discussed with the Corporate Health and Safety Manager and approved prior to such use.

EQUIPMENT

Equipment will only be operated by personnel trained and qualified as competent on use of the machine prior to field use and under the direction of an experienced operator. The following equipment will be used.

- Calibration gas from manufacturer.
- Photoionization detector equipped with 10.0 eV lamp or greater.
- Charger unit and source of 110v power when needed, not in the form of a generator.
- Forms and indelible ink pen
- Disposable chemical-resistant gloves

PROCEDURES

On a daily basis, the unit should be gas calibrated to a manufacturer's gas standard and the results recorded in the field logbook. The unit is typically calibrated with isobutylene of 100 parts per million (ppm) concentration as the calibration gas. When checking the calibration, the unit should read within 5% of the span gas concentration used (e.g., between 95 and 105 ppm when 100 ppm span gas is used). If the unit does not calibrate, return it to the local Terracon equipment evaluation for evaluation and, if necessary, repair.

Immediately prior to making a field measurement the unit should be operated for approximately 1 minute remote from activities engaged in sampling or disturbing contaminants. Any background concentrations should be noted or zeroed out relative to test measurements.

Readings will be taken in the breathing zone. Measurements can be taken continuously unattended (i.e., attached to the drill rig or excavator during equipment operations) or on an attended periodic basis specified by the project manager.

For continuous unattended readings the unit will placed secure from damage or interference from machinery exhausts at an elevation consistent with the breathing zone and in close proximity to workers. The unit will be operated in a mode which will record the maximum reading without having to continuously view the readout. The unit will be operated in alarm mode set at the first response level specified in the project health and safety plan. The maximum will be checked periodically by the operator, but on intervals never exceeding 30 minutes.

For periodic attended readings the unit will be handheld. The operator will hold the unit probe in the breathing zone, moving slowly through the zone in close proximity to workers without interfering with their activity. The reading will be made for no less than 3 minutes to account for response times of ionizable semi-volatile compounds of concern to account for varied response times.

Record readings obtained as parts per million (ppm) calibration gas equivalents (i.e., TEI580 calibrated to isobutylene would be expressed as ppmi) .

DOCUMENTATION

Record readings in calibration gas equivalents on the Acknowledgment Of Instruction Form of the project health and safety plan. Alternatively, they may be recorded on forms or the project field log book provided by the Project Manager. Copies of these records will be attached to and filed with the file copy of the health and safety plan.

Readings will clearly be recorded and labeled as separate from readings made for the purpose of field screening soils for environmental impacts.

OTHER SUPPORTING DOCUMENTS

- **Thermo Environmental Instruments, Inc.** *OVM/Datalogger Model 580B Operating Manual*

Terracon

STANDARD OPERATING PROCEDURE

E.570
TEMPERATURE FIELD SCREENING - WATER

Last Revision or Review: June 2010

Objective and Application

To provide a qualitative and limited quantitative field screening of water samples to aid in the evaluation of water temperature.

Equipment

- Disposable chemical-resistant gloves,
- Thermometer,
- Sterile container or zip top bag, and
- Deionized water.

Procedures

The sample containment area of the temperature meter or the measuring end of the thermometer should be cleaned with deionized water before each use.

As the temperature of the water sample will begin to normalize to ambient temperature as soon as the sample is removed from its point of origin (monitoring well, stream, etc.), temperature should be the first field measurement obtained. For each water sample to be tested, place a minimum of 100 milliliters (mL) of sample in the sterile container or zip top bag. If less than 100 mL is used, the temperature of the water sample may not stabilize prior to reaching ambient temperatures. Insert the measuring end of thermometer into the water sample. The thermometer should not rest against the sides of the sterile container while taking the measurement. Allow the thermometer readings to stabilize, and record the final temperature.

Documentation

Record the temperature of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

Other Supporting Documentation

ASTM E1 *Standard Specification for ASTM Thermometers*.

Terracon

STANDARD OPERATING PROCEDURE

E.580
TURBIDITY FIELD SCREENING - WATER

Last Revision or Review: June 2010

Objective and Application

To provide a qualitative and limited quantitative field screening of water samples to aid in the evaluation of water for turbidity, or the presence of visible suspended solids.

Equipment

- Disposable chmical-resistant gloves.
- LaMotte Turbidimeter Model 2008 or equivalent.
- Deionized water.

Procedures

Before testing water samples, calibrate the turbidity meter according to the manufacturer's recommendation. Calibration should occur as often as specified by the Project Manager, but at a minimum should be conducted at the manufacturers specified intervals, and at a minimum on a daily basis. The specialized sample containers supplied with the meter should be cleaned with deionized water prior to each use.

For each water sample to be tested, place a small amount of sample in the sample container supplied with the turbidity meter. Agitate the sample if needed to suspend any settled solids. Allow the turbidity readings to stabilize. Warning – do not allow the sample to remain still long enough for suspended solids to begin settling out of suspension; this could result in the meter underestimating the turbidity of the sample. Record the turbidity measured by the meter.

Documentation

Record the turbidity of the sample on forms provided by the Project Manager or in the field log book. Pertinent data will vary based on the parameter and the form; however, the following data must be recorded. Time, date, job number, project name, sampling location, samplers name, and general observations.

Attached Supporting Information

LaMotte Turbidimeter Model 2008 Operating Manual

Understanding Turbidity Measurement, HACH Technical Center for Applied Analytical Chemistry

Other Supporting Information

ASTM D1889-00 *Standard Test Method for Turbidity of Water*

Terracon
STANDARD OPERATING PROCEDURE

E.700
WELL CONSTRUCTION - TEMPORARY

Last Review or Revision: June 2010

Objective

To provide standard procedures for the design and installation of temporary monitoring wells that will result in reliable construction and provide for the collection of representative groundwater samples.

Background

The standard practice for design and installation of monitoring wells is contained in ASTM D 5092 - 90 "Design and Installation of Groundwater Monitoring Wells in Aquifers." Terracon procedures incorporate the provisions of ASTM D 5092 that describe the specific procedures to be used in installing monitoring wells in unconsolidated formations for subsurface characterization of sites. The ASTM standard should be referred to when installation of monitoring wells is required outside of the temporary monitoring wells described here.

Equipment

The augers required for 2-inch diameter monitoring wells are 4-1/4-inch inside diameter hollow stem augers. For 4-inch wells the 6-1/4-inch inside diameter hollow stem augers are recommended. Flight augers are not recommended for installation of monitoring wells unless aquifer conditions are well known and will allow installation of screen and riser pipe without formation cave-in.

Monitoring Well Material

a) Riser Pipe

The riser pipe used is threaded, flush jointed Tri-Loc (or equivalent) Schedule 40 PVC pipe. This riser pipe has been found to provide a reliable and durable well.

b) Screen

Slot openings generally used for monitoring wells are 0.010 and 0.020 inch. The slot opening commonly used is 0.010 which will be effective with the use of gravel pack #20-40. If a larger slot opening is desired (.020), a gravel pack of #10-20 is recommended.

c) Gravel Pack

The standard gravel pack used is graded as #20-40 which is suited for use with a slot size of .010. Gravel pack should extend approximately two feet above the slotted screen. Gravel pack grading should be specified

by the Project Manager or the Drilling Coordinator of the NE Division.

d) Bentonite

Bentonite pellets (1/4-inch size) are used to provide a relatively impermeable layer above the screen and gravel pack. One 5-gallon bucket of bentonite pellets should provide a two foot layer in the annulus of a boring created with 4 1/4-inch inside diameter augers and containing 2-inch monitoring well pipe.

e) Surface Security Materials

The well is secured with an expansion cap utilizing a keyed alike lock. A commercial flush mount protector or above-grade metal protector is used based on surface use conditions or client requirements.

Procedures

a) Background

Personnel involved in drilling operations must be familiar with the drilling procedures in ASTM D 5092. The Project Manager will specify the sampling interval, decontamination requirements, screen slot size, gravel pack size, and completion and survey requirements.

b) Mobilization

The project manager will prepare the mobilization sheets for the required drilling operation. The mobilization sheets should summarize types and quantities of well materials, drilling supplies, appropriate equipment, boring depths and boring locations. The enclosed tables should be referred to for calculating the volumes of gravel pack, bentonite, and grout required.

c) Field Operations

The field crews will review the mobilization sheets for adequacy. Unless otherwise specified the field crew will determine the specific use of equipment such as grout trailer, ready mix etc. The field crews will complete the monitoring wells as specified and complete forms for describing boring logs and well details. Any deviation from standard procedures or unusual observations should be noted by the field crews and reported to the project manager.

d) Well Security

The well is secured in accordance with the project manager's instructions and TSOPs E.900, E.905, E.910, or E.920. The proper well security materials to be used in completing the top of a monitoring well should be specified by the project manager and based on the expected ground surface use, regulatory requirements, and client desires. Care should be taken in installation of monitoring wells that will be subjected to vehicle traffic.

e) Well Documentation.

The well will be plugged and abandoned in accordance with TSOP E.1700.

e) Plugging and Abandonment.

The attached field boring log and monitoring well completion sheets are to be completed for each monitoring well.

Attached Supporting Documents

- Volume of Soil Boring, Annulus around 2" and 4" Casings, and Grout Mixtures
- **Terracon Form 130** – Log of Boring No., Monitoring Well Details, and Boring Elevations.
- **ASTM D 5092-90** *Design and Installation of Groundwater Monitoring Wells*
- Brainard Kilman Field Facts

VOLUME OF SOIL BORING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0.028	0.088	0.201	0.29	0.37	0.57
2	0.06	0.18	0.4	0.6	0.7	1.1
3	0.08	0.26	0.6	0.9	1.1	1.7
4	0.11	0.35	0.8	1.2	1.5	2.3
5	0.14	0.44	1.0	1.5	1.9	2.9
6	0.17	0.53	1.2	1.7	2.2	3.4
7	0.20	0.62	1.4	2.0	2.6	4.0
8	0.22	0.70	1.6	2.3	3.0	4.6
9	0.25	0.79	1.8	2.6	3.3	5.1
10	0.28	0.88	2.0	2.9	3.7	5.7
11	0.31	0.97	2.2	3.2	4.1	6.3
12	0.34	1.06	2.4	3.5	4.4	6.8
13	0.36	1.14	2.6	3.8	4.8	7.4
14	0.39	1.23	2.8	4.1	5.2	8.0
15	0.42	1.32	3.0	4.4	5.6	8.6
16	0.45	1.41	3.2	4.6	5.9	9.1
17	0.48	1.50	3.4	4.9	6.3	9.7
18	0.50	1.58	3.6	5.2	6.7	10.3
19	0.53	1.67	3.8	5.5	7.0	10.8
20	0.56	1.76	4.0	5.8	7.4	11.4
22	0.62	1.94	4.4	6.4	8.1	12.5
24	0.67	2.11	4.8	7.0	8.9	13.7
25	0.70	2.20	5.0	7.3	9.3	14.3
26	0.73	2.29	5.2	7.5	9.6	14.8
28	0.78	2.46	5.6	8.1	10.4	16.0
30	0.84	2.64	6.0	8.7	11.1	17.1
32	0.90	2.82	6.4	9.3	11.8	18.2
34	0.95	2.99	6.8	9.9	12.6	19.4
35	0.98	3.08	7.0	10.2	13.0	20.0
36	1.01	3.17	7.2	10.4	13.3	20.5
38	1.06	3.34	7.6	11.0	14.1	21.7
40	1.12	3.52	8.0	11.6	14.8	22.8
45	1.3	4.0	9.0	13.1	16.7	25.7
50	1.4	4.4	10.1	14.5	18.5	28.5
55	1.5	4.8	11.1	16.0	20.4	31.4
60	1.7	5.3	12.1	17.4	22.2	34.2
65	1.8	5.7	13.1	18.9	24.1	37.1
70	2.0	6.2	14.1	20.3	25.9	39.9
75	2.1	6.6	15.1	21.8	27.8	42.8
80	2.2	7.0	16.1	23.2	29.6	45.6
85	2.4	7.5	17.1	24.7	31.5	48.5
90	2.5	7.9	18.1	26.1	33.3	51.3
95	2.7	8.4	19.1	27.6	35.2	54.2
100	2.8	8.8	20.1	29.0	37.0	57.0
125	3.5	11.0	25.1	36.3	46.3	71.3
150	4.2	13.2	30.2	43.5	55.5	85.5
175	4.9	15.4	35.2	50.8	64.8	99.8
200	5.6	17.6	40.2	58.0	74.0	114.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0.17	0.66	1.5	2.14	2.78	4.29
2	0.3	1.3	3.0	4.3	5.6	8.6
3	0.5	2.0	4.5	6.4	8.3	12.9
4	0.7	2.6	6.0	8.6	11.1	17.2
5	0.9	3.3	7.5	10.7	13.9	21.5
6	1.0	4.0	9.0	12.8	16.7	25.7
7	1.2	4.6	10.5	15.0	19.5	30.0
8	1.4	5.3	12.0	17.1	22.2	34.3
9	1.5	5.9	13.5	19.3	25.0	38.6
10	1.7	6.6	15.0	21.4	27.8	42.9
11	1.9	7.3	16.5	23.5	30.6	47.2
12	2.0	7.9	18.0	25.7	33.4	51.5
13	2.2	8.6	19.5	27.8	36.1	55.8
14	2.4	9.2	21.0	30.0	38.9	60.1
15	2.6	9.9	22.5	32.1	41.7	64.4
16	2.7	10.6	24.0	34.2	44.5	68.6
17	2.9	11.2	25.5	36.4	47.3	72.9
18	3.1	11.9	27.0	38.5	50.0	77.2
19	3.2	12.5	28.5	40.7	52.8	81.5
20	3.4	13.2	30.0	42.8	55.6	85.8
22	3.7	14.5	33.0	47.1	61.2	94.4
24	4.1	15.8	36.0	51.4	66.7	103.0
25	4.3	16.5	37.5	53.5	69.5	107.3
26	4.4	17.2	39.0	55.6	72.3	111.5
28	4.8	18.5	42.0	59.9	77.8	120.1
30	5.1	19.8	45.0	64.2	83.4	128.7
32	5.4	21.1	48.0	68.5	89.0	137.3
34	5.8	22.4	51.0	72.8	94.5	145.9
35	6.0	23.1	52.5	74.9	97.3	150.2
36	6.1	23.8	54.0	77.0	100.1	154.4
38	6.5	25.1	57.0	81.3	105.6	163.0
40	6.8	26.4	60.0	85.6	111.2	171.6
45	7.7	29.7	67.5	96.3	125.1	193.1
50	8.5	33.0	75.0	107.0	139.0	214.5
55	9.4	36.3	82.5	117.7	152.9	236.0
60	10.2	39.6	90.0	128.4	166.8	257.4
65	11.1	42.9	97.5	139.1	180.7	278.9
70	11.9	46.2	105.0	149.8	194.6	300.3
75	12.8	49.5	112.5	160.5	208.5	321.8
80	13.6	52.8	120.0	171.2	222.4	343.2
85	14.5	56.1	127.5	181.9	236.3	364.7
90	15.3	59.4	135.0	192.6	250.2	386.1
95	16.2	62.7	142.5	203.3	264.1	407.6
100	17.0	66.0	150.0	214.0	278.0	429.0
125	21.3	82.5	187.5	267.5	347.5	536.3
150	25.5	99.0	225.0	321.0	417.0	643.5
175	29.8	115.5	262.5	374.5	486.5	750.8
200	34.0	132.0	300.0	428.0	556.0	858.0

VOLUME OF ANNULUS AROUND 2" CASING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0.065	0.178	0.26	0.34	0.54
2	0	0.13	0.4	0.5	0.7	1.1
3	0	0.20	0.5	0.8	1.0	1.6
4	0	0.26	0.7	1.0	1.4	2.2
5	0	0.33	0.9	1.3	1.7	2.7
6	0	0.39	1.1	1.6	2.0	3.2
7	0	0.46	1.2	1.8	2.4	3.8
8	0	0.52	1.4	2.1	2.7	4.3
9	0	0.59	1.6	2.3	3.1	4.9
10	0	0.65	1.8	2.6	3.4	5.4
11	0	0.72	2.0	2.9	3.7	5.9
12	0	0.78	2.1	3.1	4.1	6.5
13	0	0.85	2.3	3.4	4.4	7.0
14	0	0.91	2.5	3.6	4.8	7.6
15	0	0.98	2.7	3.9	5.1	8.1
16	0	1.04	2.8	4.2	5.4	8.6
17	0	1.11	3.0	4.4	5.8	9.2
18	0	1.17	3.2	4.7	6.1	9.7
19	0	1.24	3.4	4.9	6.5	10.3
20	0	1.30	3.6	5.2	6.8	10.8
22	0	1.43	3.9	5.7	7.5	11.9
24	0	1.56	4.3	6.2	8.2	13.0
25	0	1.63	4.5	6.5	8.5	13.5
26	0	1.69	4.6	6.8	8.8	14.0
28	0	1.82	5.0	7.3	9.5	15.1
30	0	1.95	5.3	7.8	10.2	16.2
32	0	2.08	5.7	8.3	10.9	17.3
34	0	2.21	6.1	8.8	11.6	18.4
35	0	2.28	6.2	9.1	11.9	18.9
36	0	2.34	6.4	9.4	12.2	19.4
38	0	2.47	6.8	9.9	12.9	20.5
40	0	2.60	7.1	10.4	13.6	21.6
45	0	2.9	8.0	11.7	15.3	24.3
50	0	3.3	8.9	13.0	17.0	27.0
55	0	3.6	9.8	14.3	18.7	29.7
60	0	3.9	10.7	15.6	20.4	32.4
65	0	4.2	11.6	16.9	22.1	35.1
70	0	4.6	12.5	18.2	23.8	37.8
75	0	4.9	13.4	19.5	25.5	40.5
80	0	5.2	14.2	20.8	27.2	43.2
85	0	5.5	15.1	22.1	28.9	45.9
90	0	5.9	16.0	23.4	30.6	48.6
95	0	6.2	16.9	24.7	32.3	51.3
100	0	6.5	17.8	26.0	34.0	54.0
125	0	8.1	22.3	32.5	42.5	67.5
150	0	9.8	26.7	39.0	51.0	81.0
175	0	11.4	31.2	45.5	59.5	94.5
200	0	13.0	35.6	52.0	68.0	108.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0.491	1.33	1.91	2.55	4.06
2	0	1.0	2.7	3.8	5.1	8.1
3	0	1.5	4.0	5.7	7.7	12.2
4	0	2.0	5.3	7.6	10.2	16.2
5	0	2.5	6.7	9.6	12.8	20.3
6	0	2.9	8.0	11.5	15.3	24.4
7	0	3.4	9.3	13.4	17.9	28.4
8	0	3.9	10.6	15.3	20.4	32.5
9	0	4.4	12.0	17.2	23.0	36.5
10	0	4.9	13.3	19.1	25.5	40.6
11	0	5.4	14.6	21.0	28.1	44.7
12	0	5.9	16.0	22.9	30.6	48.7
13	0	6.4	17.3	24.8	33.2	52.8
14	0	6.9	18.6	26.7	35.7	56.8
15	0	7.4	20.0	28.7	38.3	60.9
16	0	7.9	21.3	30.6	40.8	65.0
17	0	8.3	22.6	32.5	43.4	69.0
18	0	8.8	23.9	34.4	45.9	73.1
19	0	9.3	25.3	36.3	48.5	77.1
20	0	9.8	26.6	38.2	51.0	81.2
22	0	10.8	29.3	42.0	56.1	89.3
24	0	11.8	31.9	45.8	61.2	97.4
25	0	12.3	33.3	47.8	63.8	101.5
26	0	12.8	34.6	49.7	66.3	105.6
28	0	13.7	37.2	53.5	71.4	113.7
30	0	14.7	39.9	57.3	76.5	121.8
32	0	15.7	42.6	61.1	81.6	129.9
34	0	16.7	45.2	64.9	86.7	138.0
35	0	17.2	46.6	66.9	89.3	142.1
36	0	17.7	47.9	68.8	91.8	146.2
38	0	18.7	50.5	72.6	96.9	154.3
40	0	19.6	53.2	76.4	102.0	162.4
45	0	22.1	59.9	86.0	114.8	182.7
50	0	24.6	66.5	95.5	127.5	203.0
55	0	27.0	73.2	105.1	140.3	223.3
60	0	29.5	79.8	114.6	153.0	243.6
65	0	31.9	86.5	124.2	165.8	263.9
70	0	34.4	93.1	133.7	178.5	284.2
75	0	36.8	99.8	143.3	191.3	304.5
80	0	39.3	106.4	152.8	204.0	324.8
85	0	41.7	113.1	162.4	216.8	345.1
90	0	44.2	119.7	171.9	229.5	365.4
95	0	46.6	126.4	181.5	242.3	385.7
100	0	49.1	133.0	191.0	255.0	406.0
125	0	61.4	166.3	238.8	318.8	507.5
150	0	73.7	199.5	286.5	382.5	609.0
175	0	85.9	232.8	334.3	446.3	710.5
200	0	98.2	266.0	382.0	510.0	812.0

VOLUME OF ANNULUS AROUND 4" CASING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
	2	4	6	3.25	4.25	6.25
				7.25	8.25	10.25
1	0	0	0.11	0.2	0.26	0.46
2	0	0	0.2	0.4	0.5	0.9
3	0	0	0.3	0.6	0.8	1.4
4	0	0	0.4	0.8	1.0	1.8
5	0	0	0.6	1.0	1.3	2.3
6	0	0	0.7	1.2	1.6	2.8
7	0	0	0.8	1.4	1.8	3.2
8	0	0	0.9	1.6	2.1	3.7
9	0	0	1.0	1.8	2.3	4.1
10	0	0	1.1	2.0	2.6	4.6
11	0	0	1.2	2.2	2.9	5.1
12	0	0	1.3	2.4	3.1	5.5
13	0	0	1.4	2.6	3.4	6.0
14	0	0	1.5	2.8	3.6	6.4
15	0	0	1.7	3.0	3.9	6.9
16	0	0	1.8	3.2	4.2	7.4
17	0	0	1.9	3.4	4.4	7.8
18	0	0	2.0	3.6	4.7	8.3
19	0	0	2.1	3.8	4.9	8.7
20	0	0	2.2	4.0	5.2	9.2
22	0	0	2.4	4.4	5.7	10.1
24	0	0	2.6	4.8	6.2	11.0
25	0	0	2.8	5.0	6.5	11.5
26	0	0	2.9	5.2	6.8	12.0
28	0	0	3.1	5.6	7.3	12.9
30	0	0	3.3	6.0	7.8	13.8
32	0	0	3.5	6.4	8.3	14.7
34	0	0	3.7	6.8	8.8	15.6
35	0	0	3.9	7.0	9.1	16.1
36	0	0	4.0	7.2	9.4	16.6
38	0	0	4.2	7.6	9.9	17.5
40	0	0	4.4	8.0	10.4	18.4
45	0	0	5.0	9.0	11.7	20.7
50	0	0	5.5	10.0	13.0	23.0
55	0	0	6.1	11.0	14.3	25.3
60	0	0	6.6	12.0	15.6	27.6
65	0	0	7.2	13.0	16.9	29.9
70	0	0	7.7	14.0	18.2	32.2
75	0	0	8.3	15.0	19.5	34.5
80	0	0	8.8	16.0	20.8	36.8
85	0	0	9.4	17.0	22.1	39.1
90	0	0	9.9	18.0	23.4	41.4
95	0	0	10.5	19.0	24.7	43.7
100	0	0	11.0	20.0	26.0	46.0
125	0	0	13.8	25.0	32.5	57.5
150	0	0	16.5	30.0	39.0	69.0
175	0	0	19.3	35.0	45.5	80.5
200	0	0	22.0	40.0	52.0	92.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
	2	4	6	3.25	4.25	6.25
				7.25	8.25	10.25
1	0	0	0.84	1.48	1.95	3.46
2	0	0	1.7	3.0	3.9	6.9
3	0	0	2.5	4.4	5.9	10.4
4	0	0	3.4	5.9	7.8	13.8
5	0	0	4.2	7.4	9.8	17.3
6	0	0	5.0	8.9	11.7	20.8
7	0	0	5.9	10.4	13.7	24.2
8	0	0	6.7	11.8	15.6	27.7
9	0	0	7.6	13.3	17.6	31.1
10	0	0	8.4	14.8	19.5	34.6
11	0	0	9.2	16.3	21.5	38.1
12	0	0	10.1	17.8	23.4	41.5
13	0	0	10.9	19.2	25.4	45.0
14	0	0	11.8	20.7	27.3	48.4
15	0	0	12.6	22.2	29.3	51.9
16	0	0	13.4	23.7	31.2	55.4
17	0	0	14.3	25.2	33.2	58.8
18	0	0	15.1	26.6	35.1	62.3
19	0	0	16.0	28.1	37.1	65.7
20	0	0	16.8	29.6	39.0	69.2
22	0	0	18.5	32.6	42.9	76.1
24	0	0	20.2	35.5	46.8	83.0
25	0	0	21.0	37.0	48.8	86.5
26	0	0	21.8	38.5	50.7	90.0
28	0	0	23.5	41.4	54.6	96.9
30	0	0	25.2	44.4	58.5	103.8
32	0	0	26.9	47.4	62.4	110.7
34	0	0	28.6	50.3	66.3	117.6
35	0	0	29.4	51.8	68.3	121.1
36	0	0	30.2	53.3	70.2	124.6
38	0	0	31.9	56.2	74.1	131.5
40	0	0	33.6	59.2	78.0	138.4
45	0	0	37.8	66.6	87.8	155.7
50	0	0	42.0	74.0	97.5	173.0
55	0	0	46.2	81.4	107.3	190.3
60	0	0	50.4	88.8	117.0	207.6
65	0	0	54.6	96.2	126.8	224.9
70	0	0	58.8	103.6	136.5	242.2
75	0	0	63.0	111.0	146.3	259.5
80	0	0	67.2	118.4	156.0	276.8
85	0	0	71.4	125.8	165.8	294.1
90	0	0	75.6	133.2	175.5	311.4
95	0	0	79.8	140.6	185.3	328.7
100	0	0	84.0	148.0	195.0	346.0
125	0	0	105.0	185.0	243.8	432.5
150	0	0	126.0	222.0	292.5	519.0
175	0	0	147.0	259.0	341.3	605.5
200	0	0	168.0	296.0	390.0	692.0

GROUT MIXTURES

PORTLAND CEMENT GROUT

CUBIC FEET	CEMENT 94 lb. sack	BENTONITE lbs.	WATER gallons
1	0.6339	3.17	5.07
1.577	1.0	5.0	8.0
2	1.27	6.3	10.1
3	1.90	9.5	15.2
4	2.54	12.7	20.3
5	3.17	15.9	25.4
6	3.80	19.0	30.4
7	4.44	22.2	35.5
8	5.07	25.4	40.6
9	5.71	28.5	45.6
10	6.34	31.7	50.7
11	6.97	34.9	55.8
12	7.61	38.0	60.8
13	8.24	41.2	65.9
14	8.87	44.4	71.0
15	9.51	47.6	76.1
16	10.14	50.7	81.1
17	10.78	53.9	86.2
18	11.41	57.1	91.3
19	12.04	60.2	96.3
20	12.68	63.4	101.4
22	13.95	69.7	111.5
24	15.21	76.1	121.7
25	15.85	79.3	126.8
26	16.48	82.4	131.8
28	17.75	88.8	142.0
30	19.02	95.1	152.1
32	20.28	101.4	162.2
34	21.55	107.8	172.4
35	22.19	111.0	177.5
36	22.82	114.1	182.5
38	24.09	120.5	192.7
40	25.36	126.8	202.8
45	28.5	142.7	228.2
50	31.7	158.5	253.5
55	34.9	174.4	278.9
60	38.0	190.2	304.2
65	41.2	206.1	329.6
70	44.4	221.9	354.9
75	47.5	237.8	380.3
80	50.7	253.6	405.6
85	53.9	269.5	431.0
90	57.1	285.3	456.3
95	60.2	301.2	481.7
100	63.4	317.0	507.0
125	79.2	396.3	633.8
150	95.1	475.5	760.5
175	110.9	554.8	887.3
200	126.8	634.0	1014.0

BENTONITE GROUT

CUBIC FEET	BENTONITE 50 lb sack	WATER gallons
1	0.444	6.22
2	0.9	12.4
2.25	1	14
3	1.3	18.7
4	1.8	24.9
5	2.2	31.1
6	2.7	37.3
7	3.1	43.5
8	3.6	49.8
9	4.0	56.0
10	4.4	62.2
11	4.9	68.4
12	5.3	74.6
13	5.8	80.9
14	6.2	87.1
15	6.7	93.3
16	7.1	99.5
17	7.5	105.7
18	8.0	112.0
19	8.4	118.2
20	8.9	124.4
22	9.8	136.8
24	10.7	149.3
25	11.1	155.5
26	11.5	161.7
28	12.4	174.2
30	13.3	186.6
32	14.2	199.0
34	15.1	211.5
35	15.5	217.7
36	16.0	223.9
38	16.9	236.4
40	17.8	248.8
45	20.0	279.9
50	22.2	311.0
55	24.4	342.1
60	26.6	373.2
65	28.9	404.3
70	31.1	435.4
75	33.3	466.5
80	35.5	497.6
85	37.7	528.7
90	40.0	559.8
95	42.2	590.9
100	44.4	622.0
125	55.5	777.5
150	66.6	933.0
175	77.7	1088.5
200	88.8	1244.0

Terracon
STANDARD OPERATING PROCEDURE

E.800
WELL CONSTRUCTION –PERMANENT

Last Revision or Review: June 2010

Objective

To provide standard procedures for the design and installation of routine monitoring wells that will result in reliable construction and provide for the collection of representative groundwater samples.

Background

The standard practice for design and installation of monitoring wells is contained in ASTM D 5092 - 90 "Design and Installation of Groundwater Monitoring Wells in Aquifers." Terracon procedures incorporate the provisions of ASTM D 5092 which describe the specific procedures to be used in installing monitoring wells in unconsolidated formations. The ASTM standard should be referred to when installation of monitoring wells is required outside of the routine monitoring wells described here.

Equipment

The augers required for 2-inch diameter monitoring wells are 4-1/4-inch inside diameter hollow stem augers. For 4-inch wells the 6-1/4-inch inside diameter hollow stem augers are recommended. Flight augers are not recommended for installation of monitoring wells unless aquifer conditions are well known and will allow installation of screen and riser pipe without formation cave-in.

Monitoring Well Material

a) Riser Pipe

The riser pipe used is threaded, flush jointed Tri-Loc (or equivalent) Schedule 40 PVC pipe. This riser pipe has been found to provide a reliable and durable well.

b) Screen

Slot openings generally used for monitoring wells are 0.010 and 0.020 inch. The slot opening commonly used is 0.010 which will be effective with the use of gravel pack #20-40. If a larger slot opening is desired (.020), a gravel pack of #10-20 is recommended.

c) Gravel Pack

The standard gravel pack used is graded as #20-40 which is suited for use with a slot size of 0.010. Gravel pack should extend approximately two feet above the slotted screen. Gravel pack grading should be

specified by the Project Manager or the Drilling Coordinator of the NE Division.

d) Bentonite

Bentonite pellets (1/4-inch size) are used to provide a relatively impermeable layer above the screen and gravel pack. One 5-gallon bucket of bentonite pellets should provide a two foot layer in the annulus of a boring created with 4 and 1/4-inch inside diameter augers and containing 2-inch monitoring well pipe.

e) Surface Security Materials

The well is secured in accordance with the project manager's instructions and TSOPs E.900, E.905, E.910, or E.920.

Procedures

a) Background

Personnel involved in drilling operations must be familiar with the drilling procedures in ASTM D 5092. The Project Manager will specify the sampling interval, decontamination requirements, screen slot size, gravel pack size, and completion and survey requirements.

b) Mobilization

The project manager will prepare the mobilization sheets for the required drilling operation. The mobilization sheets should summarize types and quantities of well materials, drilling supplies, appropriate equipment, boring depths and boring locations. The enclosed tables should be referred to for calculating the volumes of gravel pack, bentonite, and grout required.

c) Field Operations

The field crews will review the mobilization sheets for adequacy. Unless otherwise specified the field crew will determine the specific use of equipment such as grout trailer, ready mix etc. The field crews will complete the monitoring wells as specified and complete forms for describing boring logs and well details. Any deviation from standard procedures or unusual observations should be noted by the field crews and reported to the project manager.

d) Well Security

The well is secured in accordance with the project manager's instructions and TSOPs E.900, E.905, E.910, or E.920. The proper well security materials to be used in completing the top of a monitoring well should be specified by the project manager and based on the expected ground surface use, regulatory requirements, and client desires. Care should be taken in installation of monitoring wells that will be subjected to vehicle traffic.

e) Well Documentation.

The attached field boring log and monitoring well completion sheets are to be completed for each monitoring well.

Attached Supporting Documents

- Volume of Soil Boring, Annulus around 2" and 4" Casings, and Grout Mixtures
- **Terracon Form 130** – Log of Boring No., Monitoring Well Details, and Boring Elevations.
- **ASTM D 5092-90** *Design and Installation of Groundwater Monitoring Wells*
- Brainard Kilman Field Facts

VOLUME OF SOIL BORING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING					
				INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0.028	0.088	0.201	0.29	0.37	0.57
2	0.06	0.18	0.4	0.6	0.7	1.1
3	0.08	0.26	0.6	0.9	1.1	1.7
4	0.11	0.35	0.8	1.2	1.5	2.3
5	0.14	0.44	1.0	1.5	1.9	2.9
6	0.17	0.53	1.2	1.7	2.2	3.4
7	0.20	0.62	1.4	2.0	2.6	4.0
8	0.22	0.70	1.6	2.3	3.0	4.6
9	0.25	0.79	1.8	2.6	3.3	5.1
10	0.28	0.88	2.0	2.9	3.7	5.7
11	0.31	0.97	2.2	3.2	4.1	6.3
12	0.34	1.06	2.4	3.5	4.4	6.8
13	0.36	1.14	2.6	3.8	4.8	7.4
14	0.39	1.23	2.8	4.1	5.2	8.0
15	0.42	1.32	3.0	4.4	5.6	8.6
16	0.45	1.41	3.2	4.6	5.9	9.1
17	0.48	1.50	3.4	4.9	6.3	9.7
18	0.50	1.58	3.6	5.2	6.7	10.3
19	0.53	1.67	3.8	5.5	7.0	10.8
20	0.56	1.76	4.0	5.8	7.4	11.4
22	0.62	1.94	4.4	6.4	8.1	12.5
24	0.67	2.11	4.8	7.0	8.9	13.7
25	0.70	2.20	5.0	7.3	9.3	14.3
26	0.73	2.29	5.2	7.5	9.6	14.8
28	0.78	2.46	5.6	8.1	10.4	16.0
30	0.84	2.64	6.0	8.7	11.1	17.1
32	0.90	2.82	6.4	9.3	11.8	18.2
34	0.95	2.99	6.8	9.9	12.6	19.4
35	0.98	3.08	7.0	10.2	13.0	20.0
36	1.01	3.17	7.2	10.4	13.3	20.5
38	1.06	3.34	7.6	11.0	14.1	21.7
40	1.12	3.52	8.0	11.6	14.8	22.8
45	1.3	4.0	9.0	13.1	16.7	25.7
50	1.4	4.4	10.1	14.5	18.5	28.5
55	1.5	4.8	11.1	16.0	20.4	31.4
60	1.7	5.3	12.1	17.4	22.2	34.2
65	1.8	5.7	13.1	18.9	24.1	37.1
70	2.0	6.2	14.1	20.3	25.9	39.9
75	2.1	6.6	15.1	21.8	27.8	42.8
80	2.2	7.0	16.1	23.2	29.6	45.6
85	2.4	7.5	17.1	24.7	31.5	48.5
90	2.5	7.9	18.1	26.1	33.3	51.3
95	2.7	8.4	19.1	27.6	35.2	54.2
100	2.8	8.8	20.1	29.0	37.0	57.0
125	3.5	11.0	25.1	36.3	46.3	71.3
150	4.2	13.2	30.2	43.5	55.5	85.5
175	4.9	15.4	35.2	50.8	64.8	99.8
200	5.6	17.6	40.2	58.0	74.0	114.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING					
				INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0.17	0.66	1.5	2.14	2.78	4.29
2	0.3	1.3	3.0	4.3	5.6	8.6
3	0.5	2.0	4.5	6.4	8.3	12.9
4	0.7	2.6	6.0	8.6	11.1	17.2
5	0.9	3.3	7.5	10.7	13.9	21.5
6	1.0	4.0	9.0	12.8	16.7	25.7
7	1.2	4.6	10.5	15.0	19.5	30.0
8	1.4	5.3	12.0	17.1	22.2	34.3
9	1.5	5.9	13.5	19.3	25.0	38.6
10	1.7	6.6	15.0	21.4	27.8	42.9
11	1.9	7.3	16.5	23.5	30.6	47.2
12	2.0	7.9	18.0	25.7	33.4	51.5
13	2.2	8.6	19.5	27.8	36.1	55.8
14	2.4	9.2	21.0	30.0	38.9	60.1
15	2.6	9.9	22.5	32.1	41.7	64.4
16	2.7	10.6	24.0	34.2	44.5	68.6
17	2.9	11.2	25.5	36.4	47.3	72.9
18	3.1	11.9	27.0	38.5	50.0	77.2
19	3.2	12.5	28.5	40.7	52.8	81.5
20	3.4	13.2	30.0	42.8	55.6	85.8
22	3.7	14.5	33.0	47.1	61.2	94.4
24	4.1	15.8	36.0	51.4	66.7	103.0
25	4.3	16.5	37.5	53.5	69.5	107.3
26	4.4	17.2	39.0	55.6	72.3	111.5
28	4.8	18.5	42.0	59.9	77.8	120.1
30	5.1	19.8	45.0	64.2	83.4	128.7
32	5.4	21.1	48.0	68.5	89.0	137.3
34	5.8	22.4	51.0	72.8	94.5	145.9
35	6.0	23.1	52.5	74.9	97.3	150.2
36	6.1	23.8	54.0	77.0	100.1	154.4
38	6.5	25.1	57.0	81.3	105.6	163.0
40	6.8	26.4	60.0	85.6	111.2	171.6
45	7.7	29.7	67.5	96.3	125.1	193.1
50	8.5	33.0	75.0	107.0	139.0	214.5
55	9.4	36.3	82.5	117.7	152.9	236.0
60	10.2	39.6	90.0	128.4	166.8	257.4
65	11.1	42.9	97.5	139.1	180.7	278.9
70	11.9	46.2	105.0	149.8	194.6	300.3
75	12.8	49.5	112.5	160.5	208.5	321.8
80	13.6	52.8	120.0	171.2	222.4	343.2
85	14.5	56.1	127.5	181.9	236.3	364.7
90	15.3	59.4	135.0	192.6	250.2	386.1
95	16.2	62.7	142.5	203.3	264.1	407.6
100	17.0	66.0	150.0	214.0	278.0	429.0
125	21.3	82.5	187.5	267.5	347.5	536.3
150	25.5	99.0	225.0	321.0	417.0	643.5
175	29.8	115.5	262.5	374.5	486.5	750.8
200	34.0	132.0	300.0	428.0	556.0	858.0

VOLUME OF ANNULUS AROUND 2" CASING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0.065	0.178	0.26	0.34	0.54
2	0	0.13	0.4	0.5	0.7	1.1
3	0	0.20	0.5	0.8	1.0	1.6
4	0	0.26	0.7	1.0	1.4	2.2
5	0	0.33	0.9	1.3	1.7	2.7
6	0	0.39	1.1	1.6	2.0	3.2
7	0	0.46	1.2	1.8	2.4	3.8
8	0	0.52	1.4	2.1	2.7	4.3
9	0	0.59	1.6	2.3	3.1	4.9
10	0	0.65	1.8	2.6	3.4	5.4
11	0	0.72	2.0	2.9	3.7	5.9
12	0	0.78	2.1	3.1	4.1	6.5
13	0	0.85	2.3	3.4	4.4	7.0
14	0	0.91	2.5	3.6	4.8	7.6
15	0	0.98	2.7	3.9	5.1	8.1
16	0	1.04	2.8	4.2	5.4	8.6
17	0	1.11	3.0	4.4	5.8	9.2
18	0	1.17	3.2	4.7	6.1	9.7
19	0	1.24	3.4	4.9	6.5	10.3
20	0	1.30	3.6	5.2	6.8	10.8
22	0	1.43	3.9	5.7	7.5	11.9
24	0	1.56	4.3	6.2	8.2	13.0
25	0	1.63	4.5	6.5	8.5	13.5
26	0	1.69	4.6	6.8	8.8	14.0
28	0	1.82	5.0	7.3	9.5	15.1
30	0	1.95	5.3	7.8	10.2	16.2
32	0	2.08	5.7	8.3	10.9	17.3
34	0	2.21	6.1	8.8	11.6	18.4
35	0	2.28	6.2	9.1	11.9	18.9
36	0	2.34	6.4	9.4	12.2	19.4
38	0	2.47	6.8	9.9	12.9	20.5
40	0	2.60	7.1	10.4	13.6	21.6
45	0	2.9	8.0	11.7	15.3	24.3
50	0	3.3	8.9	13.0	17.0	27.0
55	0	3.6	9.8	14.3	18.7	29.7
60	0	3.9	10.7	15.6	20.4	32.4
65	0	4.2	11.6	16.9	22.1	35.1
70	0	4.6	12.5	18.2	23.8	37.8
75	0	4.9	13.4	19.5	25.5	40.5
80	0	5.2	14.2	20.8	27.2	43.2
85	0	5.5	15.1	22.1	28.9	45.9
90	0	5.9	16.0	23.4	30.6	48.6
95	0	6.2	16.9	24.7	32.3	51.3
100	0	6.5	17.8	26.0	34.0	54.0
125	0	8.1	22.3	32.5	42.5	67.5
150	0	9.8	26.7	39.0	51.0	81.0
175	0	11.4	31.2	45.5	59.5	94.5
200	0	13.0	35.6	52.0	68.0	108.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0.491	1.33	1.91	2.55	4.06
2	0	1.0	2.7	3.8	5.1	8.1
3	0	1.5	4.0	5.7	7.7	12.2
4	0	2.0	5.3	7.6	10.2	16.2
5	0	2.5	6.7	9.6	12.8	20.3
6	0	2.9	8.0	11.5	15.3	24.4
7	0	3.4	9.3	13.4	17.9	28.4
8	0	3.9	10.6	15.3	20.4	32.5
9	0	4.4	12.0	17.2	23.0	36.5
10	0	4.9	13.3	19.1	25.5	40.6
11	0	5.4	14.6	21.0	28.1	44.7
12	0	5.9	16.0	22.9	30.6	48.7
13	0	6.4	17.3	24.8	33.2	52.8
14	0	6.9	18.6	26.7	35.7	56.8
15	0	7.4	20.0	28.7	38.3	60.9
16	0	7.9	21.3	30.6	40.8	65.0
17	0	8.3	22.6	32.5	43.4	69.0
18	0	8.8	23.9	34.4	45.9	73.1
19	0	9.3	25.3	36.3	48.5	77.1
20	0	9.8	26.6	38.2	51.0	81.2
22	0	10.8	29.3	42.0	56.1	89.3
24	0	11.8	31.9	45.8	61.2	97.4
25	0	12.3	33.3	47.8	63.8	101.5
26	0	12.8	34.6	49.7	66.3	105.6
28	0	13.7	37.2	53.5	71.4	113.7
30	0	14.7	39.9	57.3	76.5	121.8
32	0	15.7	42.6	61.1	81.6	129.9
34	0	16.7	45.2	64.9	86.7	138.0
35	0	17.2	46.6	66.9	89.3	142.1
36	0	17.7	47.9	68.8	91.8	146.2
38	0	18.7	50.5	72.6	96.9	154.3
40	0	19.6	53.2	76.4	102.0	162.4
45	0	22.1	59.9	86.0	114.8	182.7
50	0	24.6	66.5	95.5	127.5	203.0
55	0	27.0	73.2	105.1	140.3	223.3
60	0	29.5	79.8	114.6	153.0	243.6
65	0	31.9	86.5	124.2	165.8	263.9
70	0	34.4	93.1	133.7	178.5	284.2
75	0	36.8	99.8	143.3	191.3	304.5
80	0	39.3	106.4	152.8	204.0	324.8
85	0	41.7	113.1	162.4	216.8	345.1
90	0	44.2	119.7	171.9	229.5	365.4
95	0	46.6	126.4	181.5	242.3	385.7
100	0	49.1	133.0	191.0	255.0	406.0
125	0	61.4	166.3	238.8	318.8	507.5
150	0	73.7	199.5	286.5	382.5	609.0
175	0	85.9	232.8	334.3	446.3	710.5
200	0	98.2	266.0	382.0	510.0	812.0

VOLUME OF ANNULUS AROUND 4" CASING

CUBIC FEET PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0	0.11	0.2	0.26	0.46
2	0	0	0.2	0.4	0.5	0.9
3	0	0	0.3	0.6	0.8	1.4
4	0	0	0.4	0.8	1.0	1.8
5	0	0	0.6	1.0	1.3	2.3
6	0	0	0.7	1.2	1.6	2.8
7	0	0	0.8	1.4	1.8	3.2
8	0	0	0.9	1.6	2.1	3.7
9	0	0	1.0	1.8	2.3	4.1
10	0	0	1.1	2.0	2.6	4.6
11	0	0	1.2	2.2	2.9	5.1
12	0	0	1.3	2.4	3.1	5.5
13	0	0	1.4	2.6	3.4	6.0
14	0	0	1.5	2.8	3.6	6.4
15	0	0	1.7	3.0	3.9	6.9
16	0	0	1.8	3.2	4.2	7.4
17	0	0	1.9	3.4	4.4	7.8
18	0	0	2.0	3.6	4.7	8.3
19	0	0	2.1	3.8	4.9	8.7
20	0	0	2.2	4.0	5.2	9.2
22	0	0	2.4	4.4	5.7	10.1
24	0	0	2.6	4.8	6.2	11.0
25	0	0	2.8	5.0	6.5	11.5
26	0	0	2.9	5.2	6.8	12.0
28	0	0	3.1	5.6	7.3	12.9
30	0	0	3.3	6.0	7.8	13.8
32	0	0	3.5	6.4	8.3	14.7
34	0	0	3.7	6.8	8.8	15.6
35	0	0	3.9	7.0	9.1	16.1
36	0	0	4.0	7.2	9.4	16.6
38	0	0	4.2	7.6	9.9	17.5
40	0	0	4.4	8.0	10.4	18.4
45	0	0	5.0	9.0	11.7	20.7
50	0	0	5.5	10.0	13.0	23.0
55	0	0	6.1	11.0	14.3	25.3
60	0	0	6.6	12.0	15.6	27.6
65	0	0	7.2	13.0	16.9	29.9
70	0	0	7.7	14.0	18.2	32.2
75	0	0	8.3	15.0	19.5	34.5
80	0	0	8.8	16.0	20.8	36.8
85	0	0	9.4	17.0	22.1	39.1
90	0	0	9.9	18.0	23.4	41.4
95	0	0	10.5	19.0	24.7	43.7
100	0	0	11.0	20.0	26.0	46.0
125	0	0	13.8	25.0	32.5	57.5
150	0	0	16.5	30.0	39.0	69.0
175	0	0	19.3	35.0	45.5	80.5
200	0	0	22.0	40.0	52.0	92.0

GALLONS PER LINEAR FOOT

LENGTH (ft.)	DIAMETER OF SOIL BORING			INSIDE DIAMETER OF HOLLOW STEM AUGER		
				3.25	4.25	6.25
	2	4	6	7.25	8.25	10.25
1	0	0	0.84	1.48	1.95	3.46
2	0	0	1.7	3.0	3.9	6.9
3	0	0	2.5	4.4	5.9	10.4
4	0	0	3.4	5.9	7.8	13.8
5	0	0	4.2	7.4	9.8	17.3
6	0	0	5.0	8.9	11.7	20.8
7	0	0	5.9	10.4	13.7	24.2
8	0	0	6.7	11.8	15.6	27.7
9	0	0	7.6	13.3	17.6	31.1
10	0	0	8.4	14.8	19.5	34.6
11	0	0	9.2	16.3	21.5	38.1
12	0	0	10.1	17.8	23.4	41.5
13	0	0	10.9	19.2	25.4	45.0
14	0	0	11.8	20.7	27.3	48.4
15	0	0	12.6	22.2	29.3	51.9
16	0	0	13.4	23.7	31.2	55.4
17	0	0	14.3	25.2	33.2	58.8
18	0	0	15.1	26.6	35.1	62.3
19	0	0	16.0	28.1	37.1	65.7
20	0	0	16.8	29.6	39.0	69.2
22	0	0	18.5	32.6	42.9	76.1
24	0	0	20.2	35.5	46.8	83.0
25	0	0	21.0	37.0	48.8	86.5
26	0	0	21.8	38.5	50.7	90.0
28	0	0	23.5	41.4	54.6	96.9
30	0	0	25.2	44.4	58.5	103.8
32	0	0	26.9	47.4	62.4	110.7
34	0	0	28.6	50.3	66.3	117.6
35	0	0	29.4	51.8	68.3	121.1
36	0	0	30.2	53.3	70.2	124.6
38	0	0	31.9	56.2	74.1	131.5
40	0	0	33.6	59.2	78.0	138.4
45	0	0	37.8	66.6	87.8	155.7
50	0	0	42.0	74.0	97.5	173.0
55	0	0	46.2	81.4	107.3	190.3
60	0	0	50.4	88.8	117.0	207.6
65	0	0	54.6	96.2	126.8	224.9
70	0	0	58.8	103.6	136.5	242.2
75	0	0	63.0	111.0	146.3	259.5
80	0	0	67.2	118.4	156.0	276.8
85	0	0	71.4	125.8	165.8	294.1
90	0	0	75.6	133.2	175.5	311.4
95	0	0	79.8	140.6	185.3	328.7
100	0	0	84.0	148.0	195.0	346.0
125	0	0	105.0	185.0	243.8	432.5
150	0	0	126.0	222.0	292.5	519.0
175	0	0	147.0	259.0	341.3	605.5
200	0	0	168.0	296.0	390.0	692.0

GROUT MIXTURES

PORTLAND CEMENT GROUT

CUBIC FEET	CEMENT 94 lb. sack	BENTONITE lbs.	WATER gallons
1	0.6339	3.17	5.07
1.577	1.0	5.0	8.0
2	1.27	6.3	10.1
3	1.90	9.5	15.2
4	2.54	12.7	20.3
5	3.17	15.9	25.4
6	3.80	19.0	30.4
7	4.44	22.2	35.5
8	5.07	25.4	40.6
9	5.71	28.5	45.6
10	6.34	31.7	50.7
11	6.97	34.9	55.8
12	7.61	38.0	60.8
13	8.24	41.2	65.9
14	8.87	44.4	71.0
15	9.51	47.6	76.1
16	10.14	50.7	81.1
17	10.78	53.9	86.2
18	11.41	57.1	91.3
19	12.04	60.2	96.3
20	12.68	63.4	101.4
22	13.95	69.7	111.5
24	15.21	76.1	121.7
25	15.85	79.3	126.8
26	16.48	82.4	131.8
28	17.75	88.8	142.0
30	19.02	95.1	152.1
32	20.28	101.4	162.2
34	21.55	107.8	172.4
35	22.19	111.0	177.5
36	22.82	114.1	182.5
38	24.09	120.5	192.7
40	25.36	126.8	202.8
45	28.5	142.7	228.2
50	31.7	158.5	253.5
55	34.9	174.4	278.9
60	38.0	190.2	304.2
65	41.2	206.1	329.6
70	44.4	221.9	354.9
75	47.5	237.8	380.3
80	50.7	253.6	405.6
85	53.9	269.5	431.0
90	57.1	285.3	456.3
95	60.2	301.2	481.7
100	63.4	317.0	507.0
125	79.2	396.3	633.8
150	95.1	475.5	760.5
175	110.9	554.8	887.3
200	126.8	634.0	1014.0

BENTONITE GROUT

CUBIC FEET	BENTONITE 50 lb sack	WATER gallons
1	0.444	6.22
2	0.9	12.4
2.25	1	14
3	1.3	18.7
4	1.8	24.9
5	2.2	31.1
6	2.7	37.3
7	3.1	43.5
8	3.6	49.8
9	4.0	56.0
10	4.4	62.2
11	4.9	68.4
12	5.3	74.6
13	5.8	80.9
14	6.2	87.1
15	6.7	93.3
16	7.1	99.5
17	7.5	105.7
18	8.0	112.0
19	8.4	118.2
20	8.9	124.4
22	9.8	136.8
24	10.7	149.3
25	11.1	155.5
26	11.5	161.7
28	12.4	174.2
30	13.3	186.6
32	14.2	199.0
34	15.1	211.5
35	15.5	217.7
36	16.0	223.9
38	16.9	236.4
40	17.8	248.8
45	20.0	279.9
50	22.2	311.0
55	24.4	342.1
60	26.6	373.2
65	28.9	404.3
70	31.1	435.4
75	33.3	466.5
80	35.5	497.6
85	37.7	528.7
90	40.0	559.8
95	42.2	590.9
100	44.4	622.0
125	55.5	777.5
150	66.6	933.0
175	77.7	1088.5
200	88.8	1244.0

Terracon

STANDARD OPERATING PROCEDURE

E.900
WELL SECURITY – TYPE A (Simple Cap)

LAST REVIEW OR REVISION: June 2010

OBJECTIVE: To provide standard procedures for capping a monitoring well in the field in conditions where;

- The potential threat of persons deliberately contaminating groundwater used for sampling and testing through the surface well casing is non-existent
- The well is within a secured area or enclosure otherwise providing security
- The probability of inadvertent structural damage to the well casing is not likely

The procedure does not provide an indicator if the well has been entered by other than authorized project personnel, thereby not allowing the sampler to halt sampling or flag samples as suspect.

This procedure is low cost, low maintenance and provides no wellhead protection for sample integrity except against fugitive airborne dust. It provides the minimum level of protection for exposed well casing, inadvertent or deliberate, surface traffic.

Special Note: This procedure may used at client request for other higher threat conditions providing the client understands and accepts the risk associated with sabotaged wells.

EQUIPMENT

- Copy of completed field well construction log, field or report version.
  - Terracon *Form No. 130 MONITORING WELL DETAILS* , see figure enclosed.
  - Terracon Final Report Log from project report.
- Cap compatible with well casing material
  - PVC slip-type end cap of proper inside diameter to match well casing. For wells greater than 3-inches in diameter the flange of the slip cap should be slotted with a hacksaw free of oils prior to installation to prevent seizing of the cap to well casing in temperature changes.
  - PVC or metal threaded caps manufactured to match the well casing thread and diameter.

- Hand-operated compression pipe cutter appropriate to material and without interference to the integrity of the sampling (requires no solvents or oils).

- Wellhead marking materials ONLY FOR EXTERNAL APPLICATION AWAY FROM WELLHEAD CASING OPENING
    - Compatible markers or adhesive labels to permanently mark casing without solvent/residue interference with potential chemicals of analysis.

PROCEDURES

Inspect the casing opening for adhered soils, sand or other materials which might interfere with the slip cap or threaded plug. Clear and clean appropriate with project cleaning or decontamination procedures. The top of casing should be visually true-and-round, visually free of chips, cracks or other physical damage which could interfere with the expanding gasket of the locking cap. The casing should be flat and level across the opening, within approximately ±10-degrees of perpendicular relative to the casing side wall.

If the top of casing is damaged, use the pipe cutter to square and repair the casing opening. Record the change in length to 0.1-inch relative to the original casing rise recorded on Form 130 or Report Log Form and report to the Terracon Project Manager.

Hand place the slip cap over the well casing, do NOT drive in place mechanically. For threaded plugs hand tighten the cap only, USE NO TOOLS.

Physically mark the casing with well identification. Record and return documentation to the Project Manager and file.

DOCUMENTATION

Record any adjustments or repairs on completed Form 130 (Back) *Monitoring Well Details* and return to Project Manager.

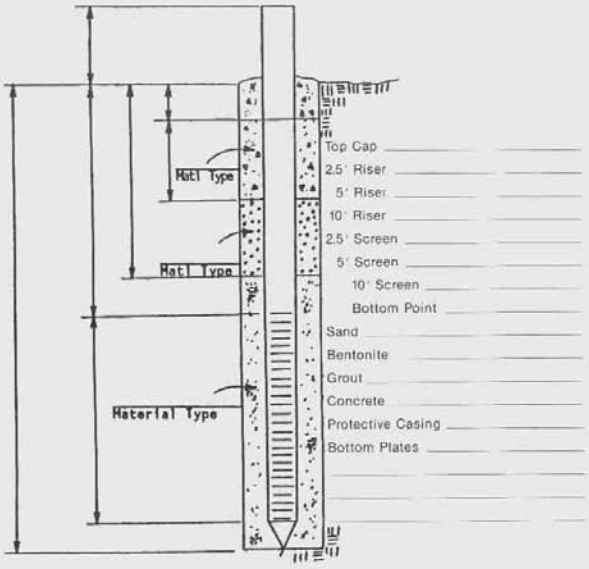
OTHER SUPPORTING DOCUMENTS

TSOP E.700	Well Construction – Temporary	August 2000
TSOP E.800	Well Construction – Permanent	November 2001

FORM 130 - BACK

MONITORING WELL DETAILS

1. Fill in applicable dimensions and identify backfill materials
2. Sketch screen installation (if more than one section used)



Top Cap _____
2.5' Riser _____
5' Riser _____
10' Riser _____
2.5' Screen _____
5' Screen _____
10' Screen _____
Bottom Point _____
Sand _____
Bentonite _____
Grout _____
Concrete _____
Protective Casing _____
Bottom Plates _____

Protective steel cover? ☐ Yes (Lock Type) _____)
Type _____ (Key number _____)
☐ No
Screen slot size or fabric type _____
Well Installation Accepted by _____ Driller _____

BORING ELEVATIONS

Benchmark _____
Elevation _____ ☐ Assumed ☐ Given

Boring/ Turning Point	Backsight	Height Instrument	Foresight	Elevation

Terracon

STANDARD OPERATING PROCEDURE

E.905
WELL SECURITY – TYPE B (Locking Expansion Cap)

LAST REVIEW OR REVISION: June 2010

OBJECTIVE: To provide standard procedures for physically securing monitoring wells in the field where the potential threat of persons deliberately contaminating groundwater used for sampling and testing through the surface well casing is low and the probability of inadvertent structural damage to the well casing is not likely.

The procedure provides an indicator if the well has been entered by other than authorized project personnel, allowing the sampler to halt sampling or flag samples as suspect.

This procedure is low cost, low maintenance and provides the lowest amount of wellhead protection for sample integrity. It provides the minimum level of protection for exposed well casing, inadvertent or deliberate, by surface traffic.

Special Note: This procedure may used at client request for other higher threat conditions providing the client understands and accepts the risk associated with sabotaged wells.

EQUIPMENT

- Copy of completed field well construction log, field or report version.
  - Terracon *Form No. 130 MONITORING WELL DETAILS* , see figure enclosed.
  - Terracon Final Report Log from project report.

- Lockable caps
  - With inert or chemically compatible expanding gasket of material not affected as to loss of expansion or damage down to temperatures of –10 degrees Fahrenheit.
  - Cherne Industries, Inc. Gripper® or Morrison-Dubuque #678XA, or equivalents approved by Terracon Equipment/Supplies Manager.
- Keyed-alike padlocks, hardened shank not required.
- Hand-operated compression pipe cutter appropriate to material and without interference to the integrity of the sampling (requires no solvents or oils).
- Wellhead marking materials ONLY FOR EXTERNAL APPLICATION AWAY FROM WELLHEAD CASING OPENING
  - Compatible markers or adhesive labels to permanently mark cap without solvent/residue interference with potential chemicals of analysis.
  - 2-inch diameter brass tag-and-ring to physically attach to padlock or cap
  - Steel number punch set for brass tags, if applicable.

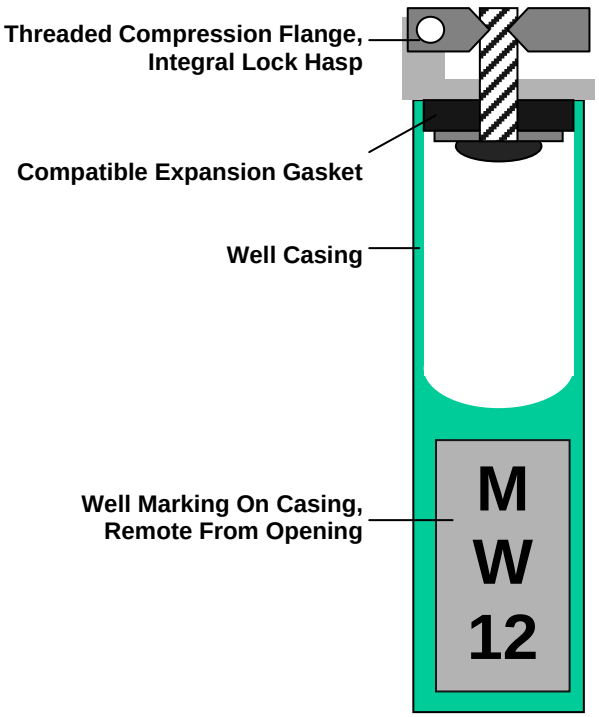
- Hammer for punch set, if applicable.
- Small metal block or hand anvil for punch, set.

- Pliers or small box end wrench

PROCEDURES

Inspect the casing opening for adhered soils, sand or other materials which might interfere with the expanding seal. Clear and clean appropriate with project cleaning or decontamination procedures. The top of casing should be visually true-and-round, visually free of chips, cracks or other physical damage which could interfere with the expanding gasket of the locking cap. The casing should be flat and level across the opening, within approximately ±10-degrees of perpendicular relative to the casing side wall.

TYPICAL TYPE A CAP ASSEMBLY



If the top of casing is damaged, use the pipe cutter to square and repair the casing opening. Record the change in length to 0.1-inch relative to the original casing rise recorded on Form 130 or Report Log Form and report to the Terracon Project Manager.

Loosen the compression nut/flange on the lockable expansion cap, but do not remove the compression nut/flange. Insert the cap into the well opening. Holding the cap in place with one hand against the casing, with the other hand tighten the compression nut/flange. Tighten to maximum hand pressure.

Using a hand tool, rotate the compression nut/flange less than one revolution to align it with the padlock hasp of the cap. DO NOT over tighten. Excessive compression can split or crack PVC well casing and can damage or break the expansion cap inside steel well casing.

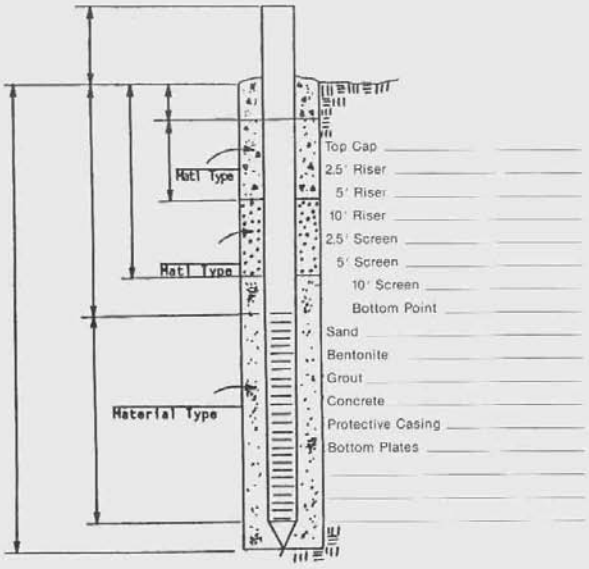
Insert the brass well tag onto the padlock shank, if applicable. Insert the keyed-alike padlock into the expansion cap.

Physically mark or tag the key with the project number, and the well identification if more than one type of keyed-lock is used for the project. Return the key(s) with documentation and return to the Project Manager for future access.

FORM 130 - BACK

MONITORING WELL DETAILS

1. Fill in applicable dimensions and identify backfill materials
2. Sketch screen installation (if more than one section used)



Top Cap _____
2.5' Riser _____
5' Riser _____
10' Riser _____
2.5' Screen _____
5' Screen _____
10' Screen _____
Bottom Point _____
Sand _____
Bentonite _____
Grout _____
Concrete _____
Protective Casing _____
Bottom Plates _____

Protective steel cover? ☐ Yes (Lock Type) _____)
Type _____ (Key number _____)
☐ No
Screen slot size or fabric type _____
Well Installation Accepted by _____ Driller _____

BORING ELEVATIONS

Benchmark _____
Elevation _____ ☐ Assumed ☐ Given

Boring/ Turning Point	Backsight	Height Instrument	Foresight	Elevation

DOCUMENTATION

Record any adjustments or repairs on completed Form 130 (Back) *Monitoring Well Details* and return with key to Project Manager.

OTHER SUPPORTING DOCUMENTS

TSOP E.700	Well Construction – Temporary	August 2000
TSOP E.800	Well Construction – Permanent	November 2001

Terracon
STANDARD OPERATING PROCEDURE

E.910
WELL SECURITY – TYPE C (Protective Casing)

LAST REVIEW OR REVISION: June 2010

OBJECTIVE: To provide standard procedures for physically securing monitoring wells in the field where the potential threat of persons deliberately contaminating groundwater used for sampling and testing through the surface well casing is moderate to high or the probability of inadvertent structural damage to the well casing is likely.

The procedure provides an indicator if the well has been entered by other than authorized project personnel intent on unauthorized access to the well, allowing the sampler to halt sampling or flag samples as suspect.

This procedure is of moderate cost, requires a moderate technical efficiency on the part of the field person and provides a high degree of wellhead protection for sample integrity. It provides a high level of protection against damage, inadvertent or deliberate, for exposed well casing from surface traffic.

This procedure should be conducted at the time of original well construction, but no later than 48 hours afterward. Until final security construction can be completed, the wellhead should be temporarily secured using *TSOPs E.900 Well Security- Type A (Simple Cap) or E.905 Well Security – Type B (Locking Expansion Cap)*.

EQUIPMENT

- Copy of field well construction log.
  - Terracon *Form No. 130 MONITORING WELL DETAILS* , see figure enclosed.

- Drilling or excavating equipment capable of soil removal from around the well casing to provide sufficient dimensions for construction, if the protective casing is not installed in the original boring annulus at the time of drilling and well construction.

- Steel protective casing of commercial manufacture matched by Terracon Project Manager to diameter of monitoring well. The protective casing will extend over and enclose the well casing. The protective casing will be of heavy gauge metal, if painted or coated against corrosion, the coating will be commercially applied and cured well before field mobilization – DO NOT re-paint protective casings in the field to prevent potential chemical contamination of the well.

- A 0.125-inch diameter weep hole will be drilled into the protective casing at a distance of at least 24-inches below the top edge of the opened protective casing. The weep hole allows water accumulation from sampling or intruding rain to escape the interior of the protective casing. <u>Accumulating water confined within the protective casing can crush the interior well casing when it freezes.</u> Alternatively, the weep hole can be drilled after construction for best position using a portable battery-powered drill.

- Well casing caps or plugs, expansion or threaded.
  - With inert or chemically compatible expanding gasket of material not affected as to loss of expansion or damage down to temperatures of –10 degrees Fahrenheit.
  - Simple slip caps as used in TSOP E.900

- Keyed-alike padlocks, hardened >0.25-inch diameter hasp required.

- Hand-operated compression pipe cutter appropriate to material and without interference to the integrity of the sampling (requires no solvents or oils).

- Wellhead marking materials ONLY FOR EXTERNAL APPLICATION AWAY FROM WELLHEAD CASING OPENING
  - Compatible markers or adhesive labels to permanently mark cap without solvent/residue interference with potential chemicals of analysis.
  - 2-inch diameter brass tag-and-ring to physically attach to padlock or cap
  - Steel number punch set for brass tags, if applicable.
  - Hammer for punch set, if applicable.
  - Small metal block or hand anvil for punch, set.
  - Stencils and spray latex paint for marking closed casing, no petroleum-based paint.

- Soft wire and wire cutters

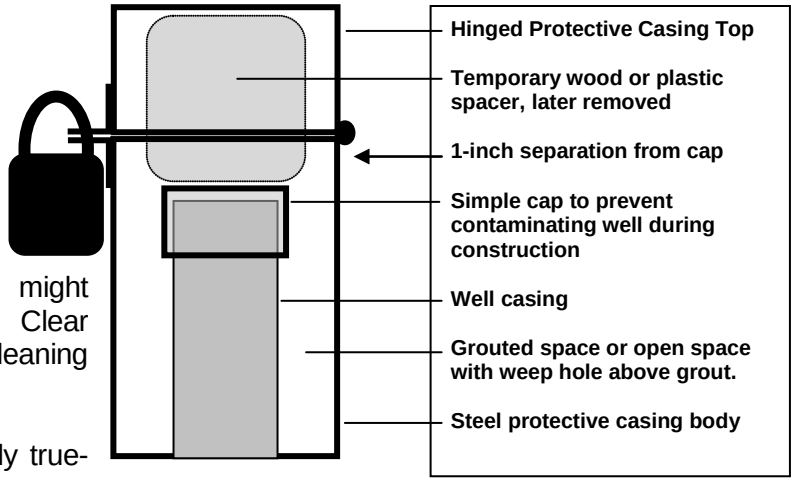
- Pliers or small box end wrench

- Battery-powered drill and 1/8th (0.125) –inch steel drill bit, if constructing weep holes in the field.

- Casing spacer(s) of at least one-half the interior diameter of the protective steel casing, flat on top and bottom and constructed of plastic or wood. The spacer will rest temporarily on top of the sealed well casing inside protective casing's hinged top when closed. The spacer should position the top of the well casing 2-inches, ±0.5 inches, below the top edge of the opened steel protective casing. This spacer is later removed after the cement grout has set and holds the protective casing in place.

PROCEDURES

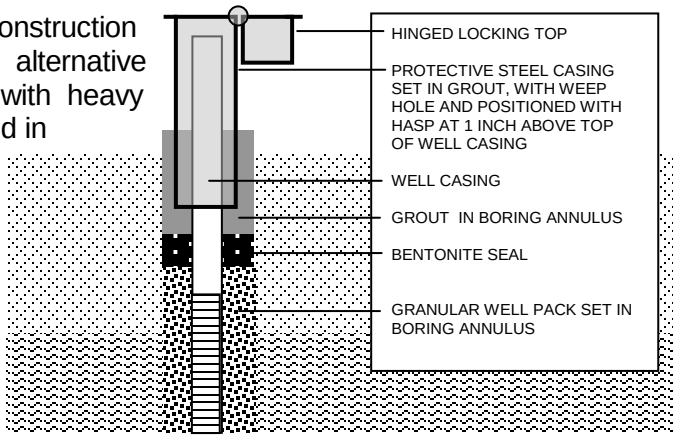
Visually inspect the top well casing after well construction and placement of the bentonite seal in the boring annulus. Inspect the casing opening for adhered soils, sand or other materials which might interfere with the expanding seal. Clear and clean appropriate with project cleaning or decontamination procedures.



The top of casing should be visually true-

and-round, visually free of chips, cracks or other physical damage which could interfere with the expanding gasket of the locking cap. The casing should be flat and level across the opening, within approximately ±10-degrees of perpendicular relative to the casing side wall. If the top of casing is damaged, use the pipe cutter to square and repair the casing opening. Record the change in length to 0.1-inch relative to the original casing riser length recorded on Form 130.

Temporarily seal the well casing during well construction using a simple cap as in TSOP E.905. An alternative method is to cover the top of the casing with heavy gauge (4-mil or greater) clean plastic bags held in place by rubber bands. Avoid use of tape to prevent residual adhesives in the immediate vicinity of the well opening.



Have at hand the spacer and pre-cut piece of soft steel wire approximately 4-inches in length. Within 10 minutes of placement of the cement grout to surface, lower the steel protective casing over the well casing. Take care to visually place the well casing at the center of the protective casing. While one person holds the protective casing centered, the second person should place the spacer on top of the well casing, carefully close the hinged cover and tightly wire the padlock flanges together. Wire the padlock flanges such that the padlock hasp can still passthrough the hole. DO NOT rely on the padlock to hold the hinged cover and protective casing in position while the grout hardens.

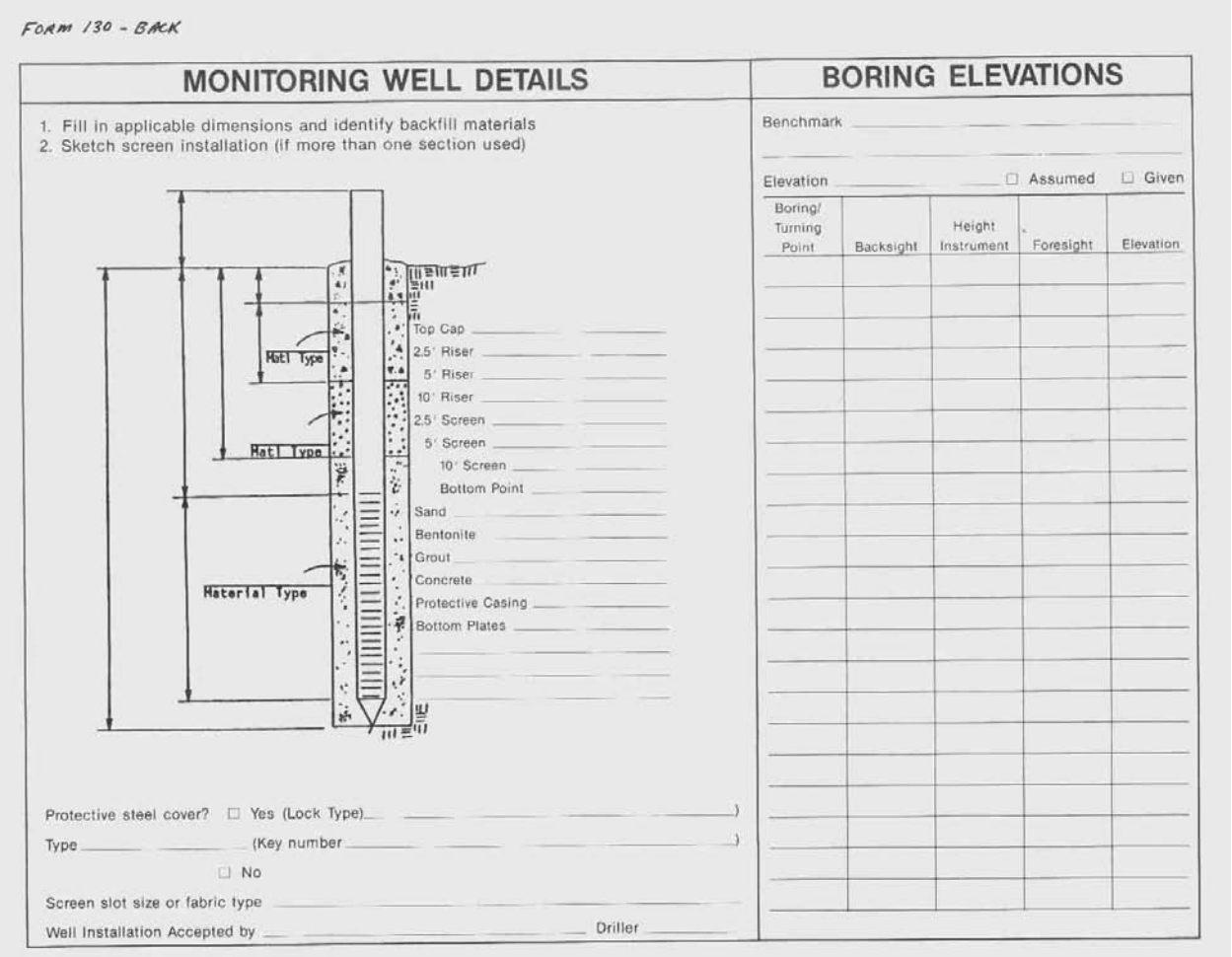
Mark, label or stencil the well identification on the exterior of the protective casing. Insert the brass well tag onto the padlock shank, if applicable. Place the padlock hasp through the padlock flanges and secure.

Physically mark or tag the key with the project number, and the well identification if more than one type of keyed-lock is used for the project. Return the key(s) with documentation and return to the Project Manager for future access.

The wire and spacer may be removed after the grout has cured (hardened) for at least 8 hours.

SPECIAL NOTE: If project specifications require the interior space of the protective casing to be filled with grout, do so after the initial construction. Thereafter DO NOT introduce neat cement grout (pure Portland cement and water), the heat of hydration and expansion of the grout confined within the steel casing will crush PVC well casings. Use grout consisting of an approximate 1:3 dry volume mixture of bentonite powder and Portland cement, respectively. Add water to a pourable slurry. With the simple well cap still in place, introduce slurry by funnel into the open space around the well casing. Take care not to spill grout on the well cap or cover/hinge assembly. Bring the grout volume to a height of 3-inches, ±0.5-inch, below the bottom of the cap to allow for some slight expansion of the grout during hydration. DO NOT inadvertently grout the cap in place.

DOCUMENTATION



FORM 130 - BACK

MONITORING WELL DETAILS

1. Fill in applicable dimensions and identify backfill materials
2. Sketch screen installation (if more than one section used)

Material Type
Material Type
Material Type

Top Cap
2.5' Riser
5' Riser
10' Riser
2.5' Screen
5' Screen
10' Screen
Bottom Point
Sand
Bentonite
Grout
Concrete
Protective Casing
Bottom Plates

Protective steel cover? ☐ Yes (Lock Type)
Type (Key number)
☐ No
Screen slot size or fabric type
Well Installation Accepted by Driller

BORING ELEVATIONS

Benchmark
Elevation ☐ Assumed ☐ Given

Boring/ Turning Point	Backsight	Height Instrument	Foresight	Elevation

Record dimensions, or other adjustments or repairs on completed Form 130 (Back) *Monitoring Well Details* and return with key to Project Manager.

OTHER SUPPORTING DOCUMENTS

TSOP E.900	Well Security – Type A (Simple Cap)
TSOP E.800	Well Construction – Permanent

Terracon

STANDARD OPERATING PROCEDURE

E.920
WELL SECURITY – TYPE D (Flushmount)

LAST REVIEW OR REVISION: June 2010

OBJECTIVE and INTENT: To provide standard procedures for physically securing monitoring wells in the field where the potential threat of persons deliberately contaminating groundwater used for sampling and testing through the surface well casing is moderate to high and the probability of inadvertent structural damage by traffic is very high or client operations require no interference with traffic for safety issues.

The procedure provides an indicator if the well has been entered by other than authorized project personnel intent on unauthorized access to the well, allowing the sampler to halt sampling or flag samples as suspect.

This procedure is of moderate to high cost, requires a moderate to high technical efficiency on the part of field staff and provides a high degree of wellhead protection for sample integrity. It provides a high level of protection against mechanical damage, inadvertent or deliberate, from surface traffic. Flushmount construction requires field staff to exercise greater care in construction and to be more precise in positioning the top of well casing relative to final elevation. The ability to shorten "too long" or trim damaged well casing is greatly restricted inside the flushmount covers. Flushmount well covers have a higher degree of post-construction maintenance to prevent possible contamination of the groundwater system being evaluated. Flush mount wells should NOT be the first choice of protection and should only be used where necessity or the client specifications demand, see Special Notes below.

This procedure should be conducted at the time of original well construction, but no later than 48 hours afterward.

Special Technical Note 1: Although designed with water-resistant gaskets, flushmount well covers are historically subject to surface water intrusion; from rain, flood, spillage during groundwater purging/sampling or floor washings. Seals deteriorate and cast-metal lids can crack under temperature fluctuations. Water collecting inside the cover basin, possibly contaminated by contact with surface, can then enter a vented well casing and contaminate the groundwater intended for sampling. Accumulated water can readily freeze if the well is constructed outside of heated structures. Confined within the cover, expanding ice can crush/crack the well casing and allow water into the well when it thaws. While the well casing may often resist freeze-thaw damage to some extent, the accumulated water frozen over the well casing will prevent access to the well without extraordinary measures. It is imperative that the seals be maintained and replaced regularly and that the covers be tightened uniformly when sealing.

Special Safety Note 2: As a precaution against seal failure, the well casing cannot be vented and must also be permanently secured using *TSOP E.905 Well Security – Type B (Locking Expansion Cap)*. However, different from E.905, the tightening of the compression nut/flange should be hand tight plus two revolutions to align the hasp. While this provides a watertight seal for the well casing inside the flushmount cover, the unvented column of air trapped inside the well casing has no where to escape as groundwater levels fluctuate up and down in the well casing. Pressure can build up and care should be taken in removing the expansion caps for sampling. In addition, in some hydrologic conditions the trapped air column, if not dissipated through un-submerged well screen, can artificially depress water levels measured within the well casing. Water level measurements may NOT be representative of stabilized groundwater levels in the subsurface.

EQUIPMENT

- Copy of field well construction log.
  - Terracon *Form No. 130 MONITORING WELL DETAILS* , see figure enclosed.

- Drilling or excavating equipment capable of soil removal from around the well casing to provide sufficient dimensions for construction, if the flushmount cover is not installed in the original boring annulus at the time of drilling and well construction.

- Steel flushmount vault and cover of commercial manufacture matched by Terracon Project Manager to diameter of monitoring well. The flushmount vault and cover will extend over and enclose the well casing. The flushmount vault and cover will be of heavy gauge metal, if painted or coated against corrosion, the coating will be commercially applied and cured well before field mobilization – DO NOT re-paint flushmount vault and covers in the field to prevent potential chemical contamination of the well.

- Well casing expansion locking plugs with inert or chemically compatible expanding gasket of material not affected as to loss of expansion or damage down to temperatures of –10 degrees Fahrenheit.

- Keyed-alike padlocks, hardened hasp required.

- Hand-operated compression pipe cutter appropriate to material and without interference to the integrity of the sampling (requires no solvents or oils).

- Wellhead marking materials ONLY FOR EXTERNAL APPLICATION AWAY FROM WELLHEAD CASING OPENING
  - Compatible markers or adhesive labels to permanently mark flushmount cover without solvent/residue interference with potential chemicals of analysis.
  - 2-inch diameter brass tag-and-ring to physically attach to padlock or cap
  - Steel number punch set for brass tags, if applicable.
  - Hammer for punch set, if applicable.
  - Small metal block or hand anvil for punch, set.

- Special socket tool by manufacturer matched to flushmount cover security bolts.

- Piece of 0.5- to 0.75-inch thick plywood or lumber to use as spacer/gauge for well cap clearance.

PROCEDURES

Visually inspect the top well casing after well construction and placement of the bentonite seal in the boring annulus. Inspect the casing opening for adhered soils, sand or other materials which might interfere with the expanding seal. Clear and clean appropriate with project cleaning or decontamination procedures.

The top of casing should be visually true-and-round, visually free of chips, cracks or other physical damage which could interfere with the expanding gasket of the locking cap. The casing should be flat and level across the opening, within approximately ±10-degrees of perpendicular relative to the casing side wall. The top of casing should be at final design elevation, considering these three essential items in exercising professional judgment in the field;

1. With the flushmount vault and cover bolted in place placed over the well, the locking expansion plug and padlock should have no less than 0.5-inch clearance from the bottom of the vault cover. Vault designs differ! Do not assume the same amount of clearance for all vault/cover assemblies (e.g., One manufacturer incorporates ribbed strengthening members cast in the underside of the flushmount cover that can increase maximum cross-sectional thickness for clearance by ~1-inch).

2. Trimming the well casing height should always be done prior to grouting in the vault/cover assembly. The top of cover is not truly flushmount, it should rise approximately 0.125-inches above surface to prevent small amounts of rain or standing water on the cover as a low place and yet clear snowplows, sweepers, etc.

3. Do not set the top of casing too low to allow for clearance, it should be set as high as possible inside the vault. This protects against minor water accumulation and freezing inside the vault (i.e., minor infiltration through cracked cover or deteriorated seal between sampling events) which might occur, yet maximizes the potential for keeping the lock and expansion cap from being covered by water/ice.

If the top of casing is damaged, use the pipe cutter to square and repair the casing opening. Record the change in length to 0.1-inch relative to the original casing riser length recorded on Form 130. Seal the well casing at all times during well construction using the expansion locking cap with padlock in place as in TSOP E.905.

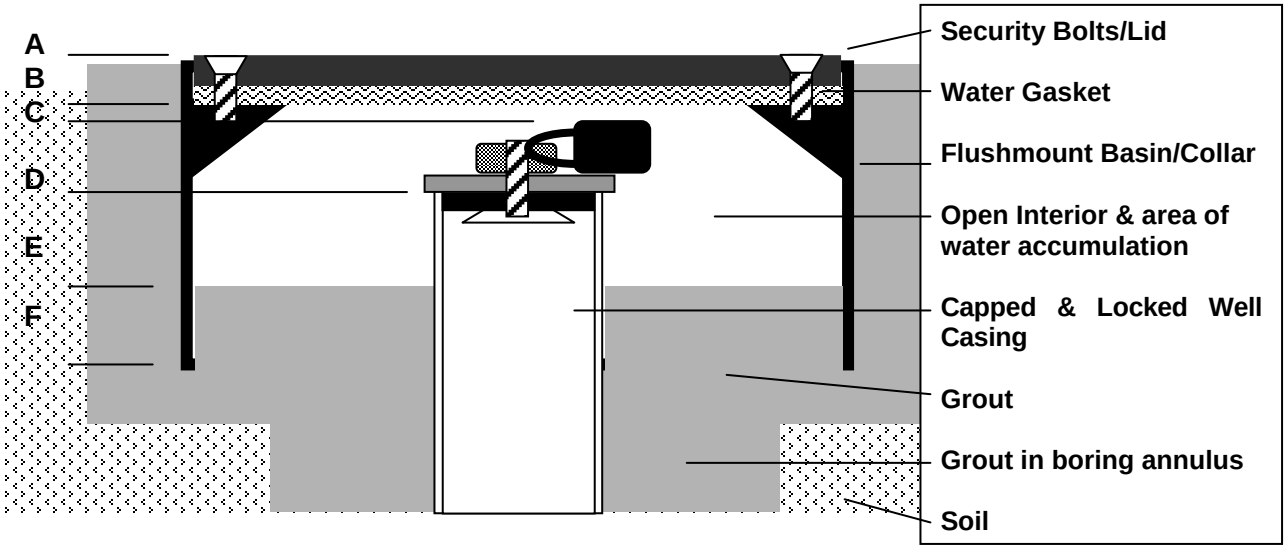
Have at hand a piece of 0.5- to 0.75-inch thick plywood to use as a temporary spacer/gauge for clearance. For example, referencing figure below;

A - Ground surface	0.0 inches elevation
B - Flushmount cover thickness	-0.5 inches
C - Clearance to cover from cap/lock (spacer)	-0.5 inches
D - Thickness of cap/lock above top of casing	-1.0 inches

Top of well casing from surface	-2.0 inches

Place the final cement grout to an elevation of surface minus the length of the vault assembly collar less ~2 inches. The intent is to bring the grout up into and around the base/collar of the flushmount basin/assembly approximately 2 inches to seal the assembly, yet not interfere with the locking expansion cap and still provide the maximum interior volume for small amounts of unplanned water infiltration. For example, referencing figure below;

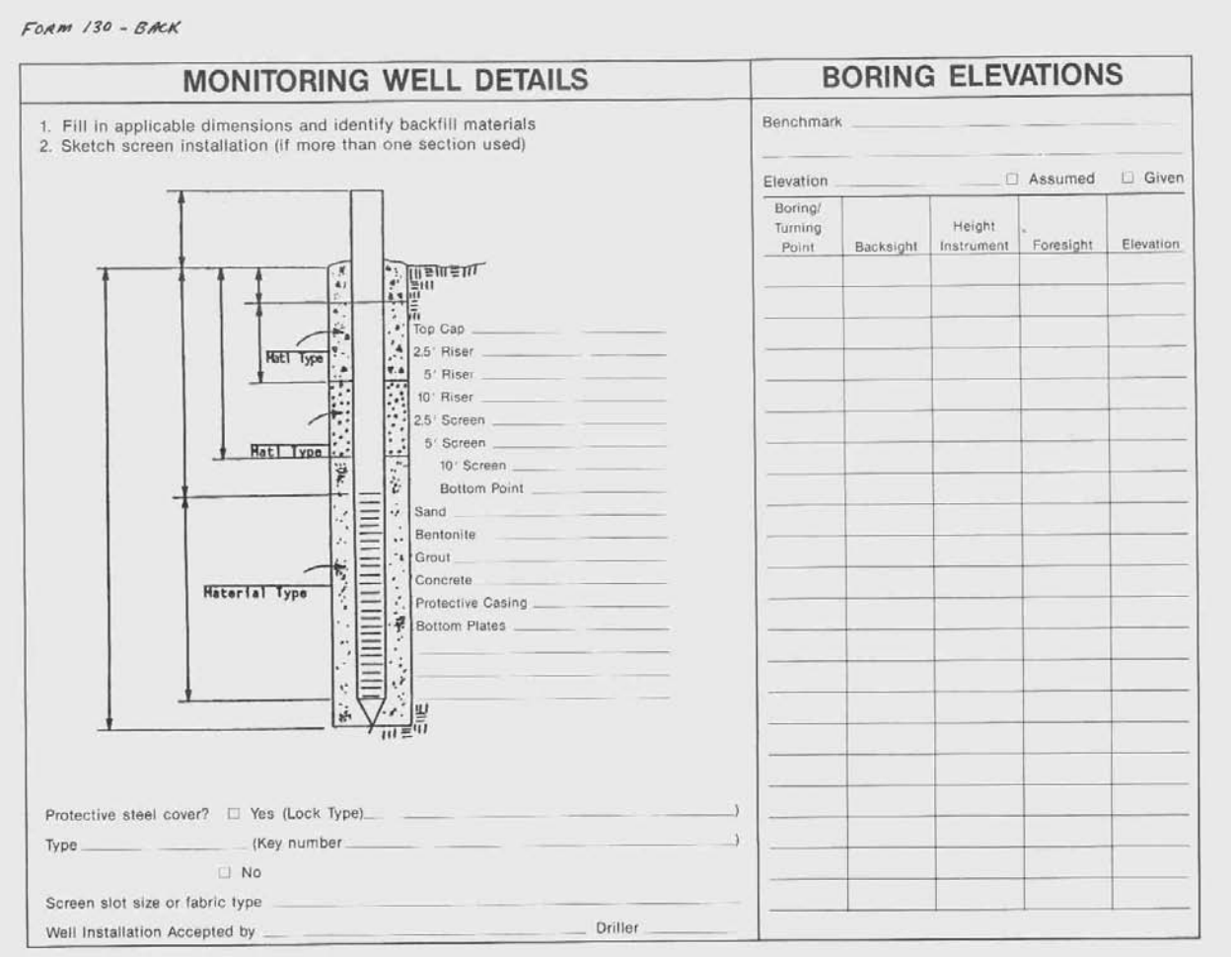
A - Ground surface	0.0 inches elevation
Length of collar/basin	-8.5 inches
F - Distance grout rise inside the basin/collar	+2.0 inches
Top of grout from surface	-6.5 inches from surface



With the spacer held in place inside the cover, set the closed (lid bolted in place) flushmount cover over the well and into the grout no later than 10 minutes after initial grout placement. Although the well casing should already be centered in the middle of the boring and area removed by hand tools or coring (in the case of slabs), take care to visually place the well casing at the center of the flushmount vault and cover. Allow the grout to undergo a firm set, typically 15 to 30 minutes for cement mixtures.

Grout the exterior of the basin, free of water and debris to surface with a stiff, but flowable neat cement grout. Standard air-entrained mixtures or commercial concrete may be used against freeze-thaw if approved by a Terracon geotechnical engineer. Slope grout upward to edges of vault cover for final finishing.

Mark, label or stencil the well identification on the exterior of the flushmount vault and cover, and/or insert the brass well tag onto the padlock shank. Place the padlock hasp through the padlock flanges and secure.



FORM 130 - BACK

MONITORING WELL DETAILS

1. Fill in applicable dimensions and identify backfill materials
2. Sketch screen installation (if more than one section used)

Material Type
Material Type
Material Type

Top Cap
2.5' Riser
5' Riser
10' Riser
2.5' Screen
5' Screen
10' Screen
Bottom Point
Sand
Bentonite
Grout
Concrete
Protective Casing
Bottom Plates

Protective steel cover? ☐ Yes (Lock Type)
Type (Key number)
☐ No
Screen slot size or fabric type
Well Installation Accepted by Driller

BORING ELEVATIONS

Benchmark
Elevation ☐ Assumed ☐ Given

Boring/ Turning Point	Backsight	Height Instrument	Foresight	Elevation

Physically mark or tag the key with the project number, and the well identification if more than one type of keyed-lock is used for the project. Return the key(s) with documentation and return to the Project Manager for future access.

Take care not to spill grout on the well cap or cover/hinge assembly. Bring the grout volume to a height of 3-inches, +.5-inch, below the bottom of the cap to allow for some slight expansion of the grout during hydration. DO NOT inadvertently grout the cap in place.

DOCUMENTATION

Record dimensions, or other adjustments or repairs on completed Form 130 (Back) *Monitoring Well Details* and return with key to Project Manager.

OTHER SUPPORTING DOCUMENTS

TSOP E.900	Well Security – Type B (Locking Expansion Cap)
TSOP E.800	Well Construction – Permanent

Appendix C – Table of Terracon Standard Operating Procedures (Enclosed on CD)

REFERENCE NO.		TITLE	LAST REVISED OR REVIEWED
E.20		Standard Safe Operating Procedures for Hazardous Waste Operations	June 2010
E.30		Chain of Custody Documentation	June 2010
E.50		Sampling – Environmental Representativeness	June 2010
	E.1XX	Soil Sampling	
	E.100	Surface & Near Surface Soil Sampling – Grab	June 2010
	E.150/155	Soil Sampling – Volatiles By TerraCore™	August 2011
E.300		Sampling & Drilling Platforms	June 2010
E.400		Subsurface Sampling – Geoprobe© Platform	August 2011
	E.4XX	Sample Handling	
	E.468	Sample Handling – Soil (Level D)	June 2010
	E.470	Sample Handling – Groundwater (Non-Hazardous)	June 2010
	E.480	Surface Water Sampling	August 2011
E.5XX		Field Screening	
E.500		pH Field Screening – Soil	June 2010
E.530		pH Field Screening – Water	June 2010
E.540		Conductivity Field Screening – Water	June 2010
E.550		Field Surface Screening – Soil / Photoionization Detector	August 2011
E.552		Field Headspace Screening – Soil / Photoionization Detector	August 2011
E.554		Field Screening – Air / Photoionization Detector	August 2011
E.570		Temperature Field Screening	June 2010
E.580		Turbidity Field Screening	June 2010
	E.700	Well Construction – Temporary	June 2010
	E.800	Well Construction – Permanent	June 2010

E.9XX		Well Security	
E.900		Well Security – Type A (Simple Cap)	June 2010
E.905		Well Security – Type B (Locking Expansion)	June 2010
E.910		Well Security – Type B (Protective Casing)	June 2010
E.920		Well Security – Type C (Flush Mount)	June 2010
	E.1300	Well Development – Volumetric	June 2010
	E.1400	Well Development – Parametric	June 2010
	E.1500	Boring Abandonment – Commercial Sealant	June 2010
E.18XX		Physical Field Measurements	June 2010
E.1800		Field Measurement – Surface Layout	June 2010
E.1805		Field Measurement – Elevations	June 2010
E.1810		Field Measurement – Subsurface Soils	June 2010
E.1820		Field Measurement – Groundwater	June 2010
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E.4000		Sampling of Potential Lead-Based Paint	September 2011

Appendix D
Pace Analytical Services, Inc. Information
(Enclosed on CD)

File Name	Description
60_Illinois 2011 Certification.pdf	IEPA Certification
Illinois 20050 exp 8-3-12.pdf	IEPA Certification
Quality Manual Rev 14.pdf	Quality Assurance Manual
S-ALL-GB-I-015-Rev.01 (pH).pdf	SOP – SW-846 Method 9040B and 9045C
S-GB-C-008-Rev.01 (% Moisture).pdf	SOP -- ASTM D 2974-87
S-GB-I-002-Rev.05 (Ignitability-SW846 1010A).pdf	SOP – SW-846 Method 1010A
S-GB-I-013-Rev.02 (Paint Filter_9095A).pdf	SOP – SW-846 Method 9095A
S-GB-I-037-Rev.03 (Walkley Black).pdf	SOP -- TOC
S-GB-I-059-Rev.02 (Apollo TOC by Tekmar Fusion).pdf	SOP -- EPA Method 9060A
S-GB-I-063-Rev.00 (TOC by Tekmar Fusion).pdf	SOP – TOC SW-846 Method 9060A Tekmar
S-GB-I-064-Rev.00 (SmartChem CN_9012A).pdf	SOP – SW-846 Method 9012A and EPA 335.4
S-GB-I-067-Rev.00 (TCLP, SPLP,ASTM).pdf	SOP – SW-846 Method 1311 and 1312
S-GB-I-068-Rev.00 (TSS, TDS, TVS, OM, FOC).pdf	SOP -- Method 2540, STM D2974 and EPA 160.4
S-GB-M-004-Rev.03 Mercury 7470A_7471A_B.pdf	SOP – SW-846 Method 7470A & 7471 A/B
S-GB-M-005-Rev.03 (6010C).pdf	SOP -- SW-846 Method 5030B and 5035
S-GB-O-005-Rev.03 (SW846-5035-5030B).pdf	SOP -- SW-846 Method 6010C with 3010A and 3050B
S-GB-O-008-Rev.04 (SW846-8015).pdf	SOP – SW-846 Method 8015C
S-GB-O-010-Rev.03 (SW846-5030B Aq Sample Prep).pdf	SOP – SW-846 Method 5030B
S-GB-O-023-Rev.04 (TPHSOP).pdf	SOP – SW-846 Method 8015B
S-GB-O-027-Rev.03 (Pesticides SOP).pdf	SOP – SW-846 Method 8081A
S-GB-O-041-Rev.03 (PCBs By 3541).pdf	SOP – SW-846 Method 3541
S-GB-O-045-Rev.03 (Microwave Extraction for PAH, BNA, and TPH So).pdf	SOP – SW-846 Method 3546
S-GB-O-047-Rev.02 (PCB 8082A SOP).pdf	SOP – SW-846 Method 8082A
S-GB-O-049-Rev.03 (8270 SVOA).pdf	SOP – SW-846 Method 8270
S-GB-O-050-Rev.00 (8270 SVOA SIM).pdf	SOP – SW-846 Method 8270 sim
S-GB-O-053-Rev.00 (Sep Funnel Extraction).pdf	SOP – EPA Method 3510
S-GB-O-054-Rev.00 (Ultrasonic Extraction).pdf	SOP – EPA Method 3550
S-GB-O-056-Rev.02 (VOA Analytical_Prep).pdf	SOP – SW-846 Method 8260B w/ 5030B & 5035

QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures

Revision 14.0

Pace Analytical Services – *Green Bay, WI*
1241 E Bellevue Street, Suite 9
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CORPORATE APPROVAL

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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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PACE ANALYTICAL SERVICES – *GREEN BAY, WI*
LOCAL APPROVAL

Laboratory General Manager — Date: 04/04/11
920-469-2436

Laboratory Quality Manager — Date: 4/3/11
920-469-2436

Laboratory Technical Director — Date: 04/04/2011
920-469-2436

Laboratory Technical Director — Date: 04/04/11
920-469-2436

Laboratory Technical Director — Date: 04/05/11
920-469-2436

Additional Signatures

Signature	Title	Date

Effective Date is the date of the last signature.

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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

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.

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

1.2. Statement of Purpose

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

1.3. Quality Policy Statement and Goals of the Quality System

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.

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.

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, such as the NELAC, TNI, NVLAP, and ISO 17025 standards, and are required to perform all tests in accordance with stated methods and customer requirements.

1.4. Core Values

- **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.
- **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.
- **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.
- **Honor Commitments-** Pace labs focus on making solid commitments with regards to quality, capacity, and agreed upon turnaround time to our customers.
- **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.
- **Pursue Opportunities-** Pace is committed to pursuing opportunities for the growth of the company by constantly exploring markets and areas where we can expand.
- **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

PASI's fundamental ethical principles are as follows:

- Each PASI employee is responsible for the propriety and consequences of his or her actions;
- Each PASI employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where PASI does business or seeks to do business;
- Each PASI employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.
- 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:

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.

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

1.6. Standards of Conduct

1.6.1. Data Integrity

The accuracy and integrity of the analytical results 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.

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

1.6.2. Confidentiality

PASI employees must not 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.

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

1.6.3. Conflict of Interest

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

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

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

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

The PASI Corporate Office centralizes company-wide accounting, business development, financial management, human resources development, information systems, marketing, quality, safety, and training activities. PASI's Director of Quality is responsible for assisting the development, implementation and monitoring of quality programs for the company. See Attachment IIB for the Corporate Organizational structure.

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

A General Manager (GM) supervises each regional laboratory. Some operations may have an Assistant General Manager (AGM) in situations where the General Manager is responsible for multiple laboratory facilities and is not necessarily in the facility on a regular basis. Quality Managers (QM) at each laboratory report directly to their General Manager or Assistant General Manager and will also receive guidance and direction from the Director of Quality.

The General Manager bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of the General Manager or Assistant General Manager, the Quality Manager serves as the next in command. He or she assumes the responsibilities of the GM until the GM is available to resume the duties of their position. In the absence of the GM and QM, management responsibility of the laboratory is passed to the Technical Director – provided such a position is identified – and then to the most senior department manager until the return of the GM or QM. The most senior department manager in charge may include the Client Services Manager or the Administrative Business Manager at the discretion of the General Manager.

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

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

Under the direction of the General Manager, the technical staff of the laboratory is generally organized into the following functional groups:

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

Appropriate support groups are present in each laboratory. The actual organizational structure for PASI – *Green Bay* is listed in Attachment IIA. In the event of a change in General Manager, Quality Manager, 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 Quality Manual, 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 General Managers within their designated region;
- Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Oversees the preparation of budgets and staffing plans for all operations within their designated region;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.2. General Manager (local laboratory)

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

1.8.3. Quality Manager

- Oversees the laboratory Quality Systems while functioning independently from laboratory operations. Reports directly to the General Manager;
- Monitors Quality Assurance policies and Quality Control procedures to ensure that the laboratory achieves established standards of quality;
- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews and maintains records of proficiency testing results;
- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS);
- Monitors correctives actions;
- Maintains the currency of the Quality Manual.

1.8.4. Technical Director

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

1.8.5. Administrative Business Manager

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

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

1.8.6. Client Services Manager

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

1.8.7. Project Manager

- Coordinates daily activities including taking orders, reporting data and analytical results;
- Serves as the primary technical and administrative liaison between customers and 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.

1.8.8. Project Coordinator

- Enters the sample information into the Laboratory Information Management System (LIMS) for tracking and reporting;
- 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.

1.8.9. Department Manager/Supervisor

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

1.8.10. 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 Quality Manager.

1.8.11. 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.

1.8.12. 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.

1.8.13. Sample Management Personnel

- Signs for incoming samples and verifies the data entered on the Chain of custody forms;
- Stages samples according to EPA requirements; Fills bottle orders and sample shipments.

1.8.14. 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.

1.8.15. 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.

1.8.16. 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

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.

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.

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

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.

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 Orientation and *Training Procedures* or its equivalent revision or replacement.

1.10. Data Integrity System

The data integrity system at PASI provides assurances to management that a highly ethical approach is being applied to all planning, training and implementation of methods. Data integrity is crucial to the success of our company and Pace Analytical is committed to 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. 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:
   a. Need for honesty and transparency in analytical reporting
   b. Process for reporting data integrity issues
   c. Specific examples of unethical behavior and improper practices
   d. Documentation of non-conforming data that is still useful to the data user
   e. Consequences and punishments for unethical behavior
   f. Examples of monitoring devices used by management to review data and systems
2. Signed data integrity documentation for all employees: this includes a written quiz following the Ethics training session and written agreement to abide by the Code of Ethics and Standards of Conduct explained in the employee manual.
3. In-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.
4. Documentation of any review or investigation into possible data integrity infractions. This documentation, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be available for review for laboratory assessors and must be retained for a minimum of five years.

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

Corporate management also provides all PASI facilities a mechanism for confidential reporting of data integrity issues that ensures confidentiality and a receptive environment in which all employees are comfortable discussing items of ethical concern. The anonymous message line is monitored by the Corporate Director of Quality who will ensure that all concerns are evaluated and, where necessary, brought to the attention of executive management and investigated. **The message line voice mail box number is available in the Pace Employee Handbook.**

1.11. Laboratory Safety

It is the policy of PASI to make safety and health an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety 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

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 GM, QM, or TD specify otherwise. In this instance, the staff member will escort the visitor

back to the reception area at the end of his/her visit where he/she signs out. The last staff member to leave their department for the day should ensure that all outside access points to that area are secure.

Additional security is provided where necessary, e.g., specific secure areas for sample, data, and customer report storage, as requested by customers or cases where national security is of concern. These areas are lockable within the facilities, or are 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.

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.

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).

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.

2.0. SAMPLE CUSTODY

2.1. Sampling Support

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. PASI – *Green Bay* may provide pick-up and delivery services to their customers when needed.

2.2. Field Services

Pace Analytical has a large Field Services Division which is based in their Minneapolis facility as well as limited field service capabilities in some of 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

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

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.

The laboratory maintains records of all such reviews, including discussions with customers. Routine analytical project documentation of quotes, notes, dates, initials, and/or recordings is maintained in a

project folder by project management. Conditions for new and more complex contracts are determined by the General Managers and sales representatives. Quality Management is consulted on technical requirements and operations staff provides input on volume capacities. Evidence of these reviews is maintained in the form of awarded Request for Proposals (RFPs), signed quotes or contracts, and a Customer Relationship Management (CRM) database. If a review identifies a potential mismatch between customer requirements and laboratory capabilities and/or capacities, Pace will specify its level of commitment by listing these exceptions to the requirements within the RFP, quote or contract.

Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-ALL-C-006 *Review of Analytical Requests* or its equivalent revision or replacement.

2.4. Chain of Custody

A chain of custody (COC) 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.

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.

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

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

- Customer project name
- Project location or number
- Field sample number/identification
- Date and time sampled
- Sample 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

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.

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

2.5. Sample Acceptance Policy

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

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

All samples must:

- Have unique customer identification that are clearly marked 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 must be received frozen at ≤0°C.

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

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

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

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

2.6. Sample Log-in

After sample inspection, all sample information on the chain of custody is entered into the Laboratory Information Management System.

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

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.

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

Current region codes are noted below. More may be added without updating this document.

10 = Minnesota
92 = Asheville and Charlotte
60 = Kansas
50 = Indianapolis
3038 = Pittsburgh Radiological
25 = Seattle
51 = Columbus
35 = Florida
20 = Gulf Coast
30 = Pittsburgh
40 = Green Bay
17 = Pace Life Sciences
65 = Schenectady (NEA)

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

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

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

2.7. Sample Storage

2.7.1. Storage Conditions

Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. 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.

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.

2.7.2. Temperature Monitoring

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.

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 if necessary.
- The Quality Manager and/or laboratory management are notified if the problem persists.
- The samples are relocated to a proper environment if the temperature cannot be maintained after corrective actions are implemented.
- The affected customers are notified.
- Documentation is provided on analytical report.

2.7.3. Hazardous Materials

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

2.7.4. Foreign/Quarantined Soils

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

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

2.8. Sample Protection

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.

Samples are removed from storage areas by designated personnel and returned to the storage areas, if necessary, immediately after the required sample quantity has been taken.

Upon customer request, additional and more rigorous chain of custody protocols for samples and data can be implemented. For example, some projects may require complete documentation of sample custody within the secure laboratory.

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

2.9. Subcontracting Analytical Services

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 subcontracting laboratory's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations.

Prior to subcontracting samples to a laboratory outside Pace Analytical, the potential sub-contract laboratory will be pre-qualified by verifying that the subcontractor meets the following criteria:

- All certifications required for the proposed subcontract are in effect,
- Sufficient professional liability and other required insurance coverage is in effect, and
- Is not involved in legal action by any federal, state, or local government agency for data integrity issues and has not been convicted in such investigation at any time during the past 5 years.

The contact and preliminary arrangements are made between the PASI Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract laboratory agreement include:

- Method of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Laboratory certification requirement
- Price per analysis
- Turn-around time requirements

Chain of custody forms are generated for samples requiring subcontracting to other laboratories. Sample receiving personnel re-package the samples for shipment, create a transfer chain of custody form and record the following information:

- Pace Analytical Laboratory Number
- Matrix
- Requested analysis
- Special instructions regarding turnaround, required detection or reporting limits, or any unusual information known about the samples or analytical procedure.
- Signature in "Relinquished By"

All subcontracted sample data reports are sent to the PASI Project Manager. Pace will provide a copy of the subcontractor's report to the client when requested.

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-NELAC work is clearly identified. PASI will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

Additional information can be found in SOP S-ALL-Q-027 *Evaluation & Qualification of Vendors* or its equivalent revision or replacement.

2.10. Sample Retention and Disposal

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.

Unused portions of samples are retained by each laboratory based on program or customer requirements for sample retention and storage. The sample retention time is a minimum of 45 days from receipt of the samples. Samples requiring storage beyond this time due to special requests or contractual obligations will not be stored under temperature controlled conditions unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

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

Additional information can be found in SOP S-GB-S-006 *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

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.

In the event of a customer-specific need, instrumentation constraint or regulatory requirement, PASI laboratories reserve the right to use valid versions of methods that may not be the most recent edition available.

3.2. Analytical Method Documentation

The primary form of 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).

The SOPs may be supplemented by other training materials that further detail how methods are specifically performed. This training material will undergo periodic, documented review along with the other Quality System documentation.

3.3. Analytical Method Validation

In some situations, PASI develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods 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 determination of the limit of detection and limit of quantitation, evaluation of precision and bias, and evaluation of selectivity of each analyte of interest.

3.4. Demonstration of Capability (DOC)

Analysts complete an initial demonstration of capability (IDOC) study prior to performing a method or when there is a change in instrument type, personnel, or test method, 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.

For Continuing Demonstrations of Capability, the laboratories may use Performance Testing (PT) samples in lieu of the 4-replicate approach listed above. For methods or procedures that do not lend themselves to the "4-replicate" approach, the demonstration of capability requirements will be specified in Section 13 – Method Performance of the applicable SOP.

Additional information can be found in SOP S-ALL-Q-020 *Training Procedures* or its equivalent revision or replacement.

3.5. Regulatory and Method Compliance

PASI understands that expectations of our customers commonly include the assumption that laboratory data will satisfy specific regulatory requirements. Therefore PASI attempts to ascertain, prior to beginning a project, what applicable regulatory jurisdiction, agency, or protocols apply to that project. This information is also required on the chain of custody submitted with samples.

PASI makes every effort to detect regulatory or project plan inconsistencies, based upon information from the customer, and communicate them immediately to the customer in order to aid in the decision making process. PASI will not be liable if the customer chooses not to follow PASI recommendations.

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

4.0. QUALITY CONTROL PROCEDURES

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

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.

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.

The method blank consists of a matrix similar to the associated samples that is known to be free of analytes of interest. Laboratories will characterize a representative matrix as "clean" if the matrix contains contaminants at less than ½ the laboratory's reporting limit.

Method blanks are not applicable for certain analyses, such as pH, conductivity, flash point and temperature.

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. Corrective actions include the re-preparation and re-analysis of all the samples (where possible) along with the full set of required quality control samples. Data qualifiers must be applied to any result reported that is associated with a contaminated method blank.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.2. Laboratory Control Sample

The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis.

An LCS is processed at a minimum frequency of 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.

The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes.

The LCS contains **all** analytes specified by a specific method or by the customer or regulatory agency (which may include full list of target compounds, with certain exceptions. These exceptions may include analyzing only specific Aroclors when PCB analysis is requested or not spiking with all

EPA Appendix IX compounds when a full Appendix IX list of compounds is requested). In the absence of specified components, the laboratory will spike with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
  - For methods with 1-10 target compounds, the 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.

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.

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

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

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

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

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

A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch unless the MS is actually used as the LCS.

A **Matrix Spike/Matrix Spike Duplicate** (MS/MSD) set is processed at a frequency specified in a particular method or as determined by a specific customer 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.

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.

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 with the same number of compounds as previously discussed in the LCS section.

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

A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.4. Surrogates

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

Surrogates are added to each customer sample (for organics), method blank, LCS, and MS prior to extraction or analysis. The surrogates are evaluated against the method or laboratory derived acceptance criteria 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.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.5. Sample Duplicate

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

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

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.6. Internal Standards

Internal Standards are method-specific analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. At a minimum, the laboratory will follow method specific guidelines for the treatment of internal standard recoveries as they are related to the reporting of data.

Deviations made from this policy must be approved by the Quality Manager prior to release of the data.

4.7. Field Blanks

Field blanks are blanks prepared at the sampling site in order to monitor for contamination that may be present in the environment where samples are collected. These field quality control samples are often referenced as field blanks, 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

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)

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.

The LOD is initially established for the compounds of interest for each method in a clean matrix with no target analytes present and no interferences at a concentration that would impact the results. The LOD is then determined every time there is a change in the test method that affects how the test is performed or when there has been a change in the instrument that affects the sensitivity. If required by customer, method or accreditation body, the LOD will be re-established annually for all applicable methods.

Unless otherwise noted, the method used by PASI laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. Where required by regulatory program or customer, the above referenced procedure will be followed.

Where specifically stated in the published method, LODs or MDLs will be performed at the listed frequency.

The validity of the LOD must be shown by detection (a value above zero) of the analytes in a QC sample in each quality system matrix. The QC sample must contain the analyte at no more than 3X the LOD for a single analyte test and 4X the LOD for multiple analyte tests. This verification must be performed on each instrument used for sample analysis and reporting of data. The validity of the LOD must be verified as part of the LOD determination process. This verification must be done prior to the use of the LOD for sample analysis.

An LOD study is not required for any analyte for which spiking solutions or quality control samples are not available such as temperature.

The LOD, if required, shall be verified annually for each quality system matrix, technology and analyte. In lieu of performing full LOD (MDL) studies annually, the 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).

Additional information can be found in SOP S-ALL-Q-004 *Method Detection Limit Studies* or its equivalent revision or replacement.

4.10. Limit of Quantitation (LOQ)

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

The LOQ must be higher than the LOD.

To verify the LOQ, the laboratory will prepare a sample in the same matrix used for the LCS. The sample will be spiked with target analytes at the concentration(s) equivalent to or less than the RL(s). This sample must undergo the routine sample preparation procedure including any routine sample cleanup steps. The samples extracted for LOD determination can be used for this purpose. 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.

Additional information can be found in SOP S-ALL-Q-004 *Method Detection Limit Studies* or its equivalent revision or replacement.

4.11. Estimate of Analytical Uncertainty

PASI laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, PASI laboratories base this estimation on the recovery data obtained from the Laboratory Control Spikes. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-GB-Q-010 *Estimation of Measurement Uncertainty* or its equivalent revision or replacement.

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

4.12. Proficiency Testing (PT) Studies

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

The laboratory initiates an investigation whenever PT results are deemed 'unacceptable' by the PT provider. All findings and corrective actions taken are reported to the Quality Manager. A corrective action plan 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.

PT samples are treated as typical customer samples, utilizing the same staff, methods, equipment, facilities, and frequency of analysis. PT samples are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

Comparison of analytical results with anyone participating in the same PT study is prohibited prior to the close of the study.

Additional information can be found in SOP S-ALL-Q-010 *PE/PT Program* or its equivalent revision or replacement.

4.13. Rounding and Significant Figures

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

4.13.1. 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).

4.13.2. Significant Digits

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

5.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

5.1. Document Management

Additional information can be found in SOP S-ALL-Q-002 *Document Management* or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

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).

A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. This establishes that there are no invalid or obsolete documents in use in the facility. All documents are reviewed periodically and revised if necessary. Obsolete documents are systematically discarded or archived for audit or knowledge preservation purposes.

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

SOPs, specifically, are available to all 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.

5.1.1. Quality Assurance Manual (QAM)

The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for PASI. The base QAM template is distributed by the Corporate Quality Department to each of the regional Quality Managers. The regional management personnel modify the necessary and permissible sections of the base template and submit those modifications to the Corporate Director of Quality for review. Once approved and signed by both the CEO and the Director of Quality; the General Manager, Quality Manager, and any Technical Directors sign the Quality Assurance Manual. Each regional Quality Manager is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual

template is reviewed on an annual basis by all of the PASI Quality Managers and revised accordingly by the Director of Quality.

5.1.2. Standard Operating Procedures (SOPs)

SOPs fall into two categories: company-wide documents and facility specific documents. Company-wide SOPs start with the prefix S-ALL- and local SOPs start with the individual facility prefix.

The purpose of the company-wide SOPs is to establish policies and procedure that are common and applicable to all PASI facilities. Company-wide SOPs are document-controlled by the corporate quality office and signed copies are distributed to all of the regional Quality Managers. The regional management personnel sign the company-wide SOPs. The regional Quality Manager is then in charge of distribution to employees, external customers, or regulatory agencies and maintaining a distribution list of controlled document copies.

Regional PASI facilities are responsible for developing facility-specific SOPs applicable to their respective facility. The regional facility develops these facility-specific SOPs based on the corporate-wide SOP template. This template is written to incorporate a set of minimum method requirements and PASI best practice requirements. The regional facilities may add to or modify the corporate-wide SOP template provided there are no contradictions to the minimum method or best practice requirements. Facility-specific SOPs are controlled by the regional Quality Manager according to the corporate document management policies.

SOPs are reviewed every two years at a minimum although a more frequent review may be required by some state or federal agencies or customers. A review of the document does not necessarily constitute issuing a new revision. Documentation of this review and any applicable revisions are made in the last section of each SOP. This provides a historical record of all revisions.

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

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

5.1.3. Other Documentation

Additional documents such as Forms and Spreadsheets are controlled through the document management system

5.2. Document Change Control

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

revisions are approved, a revision number is assigned and the previous version of the document is officially retired. Copies may be kept for audit or knowledge preservation purposes.

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

5.3. Management of Change

The process for documenting necessary changes within the laboratory network are not typically handled using the corrective or preventive action system as outlined in section 9.0. Management of Change is a proactive approach to dealing with change to minimize the potential negative impact of systematic change in the laboratory and to ensure that each change has a positive desired outcome. This process will primarily be used for the implementation of large scale projects and information system changes as a means to apply consistent systems or procedures within the laboratory network. The request for change is submitted by the initiator and subsequently assigned to an individual or team for development and planning. The final completion of the process culminates in final approval and verification that the procedure was effectively implemented. Additional information can be found in SOP S-ALL-Q-034 *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

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

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

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

All prepared standard or reagent containers include the PASI identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials. This ensures traceability back to the standard preparation logbook.

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.

If a second source standard is required to verify an existing calibration or spiking standard, this standard should be obtained from a different manufacturer or from a different lot unless client specific QAPP requirements state otherwise.

Additional information concerning standards and reagent traceability can be found in the SOP S-ALL-Q-025 *Standard and Reagent Preparation and Traceability* or its equivalent revision or replacement.

6.2. General Analytical Instrument Calibration Procedures

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

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

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

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

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

6.2.1. General Organic Calibration Procedures

Calibration standards are prepared at a minimum of five concentrations for organic analyses. Results from all calibration standards analyzed must be included in constructing the calibration curve with the following exceptions:

- The lowest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The reporting limit must be adjusted to the lowest concentration included in the calibration curve.

- The highest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done an individual analyte basis. The upper limit of quantitation must be adjusted to the highest concentration included in the calibration curve.
- Multiple points from either the high end or the low end of the calibration curve may be excluded as long as the remaining points are contiguous in nature and the minimum number of levels remains as established by method or standard operating procedure. The reporting limit or quantitation range, which is appropriate, must be adjusted accordingly.
- Results from a concentration level between the lowest and highest calibration levels can be excluded from the calibration curve for an acceptable cause with approval from the responsible department supervisor if the results for all analytes are excluded and the point is replaced by re-analysis. Re-analysis must occur within the same 12 hour tune time period for GC/MS methodologies and within 8 hours of the initial analysis for non-GC/MS methodologies. All samples analyzed prior to the re-analyzed calibration curve point must be re-analyzed after the calibration curve is completed.

Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Curves that do not meet the appropriate criteria require corrective action that may include re-running the initial calibration curve. All initial calibrations are verified with 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.

The calibration curve is periodically verified by the analysis of a mid-level continuing calibration verification (CCV) standard during the course of sample analysis. 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.

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 mat 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 two consecutive passing CCVs have been analyzed. If required by specific state, program, or customer specification, the instrument is re-calibrated after two consecutive CCV failures. All samples analyzed since the last compliant CCV are re-analyzed for methodologies utilizing external calibration.

When instruments are operating unattended, autosamplers may be programmed to inject consecutive CCVs as a preventative measure against CCV failure with no corrective action. In this case, both CCVs must be evaluated to determine potential impact to the results. A summary of the decision tree and necessary documentation are listed below:

- If both CCVs meet the acceptance criteria, the analytical sequence is allowed to continue without corrective action. The 12 hour clock begins with the injection of the second CCV.
- If the first CCV does not meet the acceptance criteria and the second CCV is acceptable, the analytical sequence is continued and the results are reported.
- If the first CCV meets the acceptance criteria and the second CCV is out of control, the samples preceded by the out of control CCV must be re-analyzed in a compliant analytical sequence.
- If both CCVs are out of control, all samples since the last acceptable CCV must be re-analyzed in a compliant analytical sequence.

Some analytical methods require that samples be bracketed by passing CCVs analyzed both before and after the samples. This is specific to each method but, as a general rule, all external calibration methods require bracketing CCVs. Most internal standard calibrations do not require bracketing CCVs.

Some analytical methods require verification based on a time interval; some methods require a frequency based on an injection interval. The type and frequency of the calibration verifications is dependent on both the analytical method and possibly on the quality program associated with the samples. The type and frequency of calibration verification will be documented in the method specific SOP employed by each laboratory.

6.2.2. General Inorganic Calibration Procedures

The instrument is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. A calibration blank is also included. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. The number of calibration standards used depends on the specific method criteria or customer project requirements, although normally a minimum of three standards is used.

The ICP and ICP/MS can be standardized with a zero point and a single point calibration if:

- Prior to analysis, the zero point and the single point calibration are analyzed and a linear range 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.

All initial calibrations are verified with a 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.

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.

A calibration blank is also run with each calibration verification standard to verify the cleanliness of the system. All reported results must be bracketed by acceptable CCVs. Instrument and method specific calibration acceptance criteria are explained within the specific analytical standard operating procedures for each facility.

Interference check standards are also analyzed per method requirements and must meet acceptance criteria for metals analyses.

6.3. Support Equipment Calibration Procedures

All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until repaired. The laboratory maintains records to demonstrate the correction factors applied to working thermometers.

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.

6.3.1. Analytical Balances

Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Calibration weights are ASTM Class 1 or other class weights that have been calibrated against a NIST standard weight and are re-certified annually against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the Quality department.

6.3.2. Thermometers

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

Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures. Each thermometer is individually numbered and assigned a correction factor based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

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

6.3.3. pH/Electrometers

The meter is calibrated before use each day, using fresh buffer solutions. Additional information regarding pH/Electrometers can be found in SOP S-ALL-GB-I-015 *Measurement of pH in Water, Soil, and Waste*.

6.3.4. Spectrophotometers

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

6.3.5. Mechanical Volumetric Dispensing Devices

Mechanical volumetric dispensing devices including bottle top dispensers, pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis. The accuracy of glass microliter syringes is verified and documented prior to initial use.

Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-ALL-Q-013 *Support Equipment* or its equivalent revision or replacement.

6.4. Instrument/ Equipment Maintenance

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

The Laboratory Operations Manager and department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

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

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

All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation 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

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

When maintenance is performed to repair an instrument problem, depending on the initial problem, demonstration of return to control may be satisfied by the successful analysis of a tune, reagent blank, or continuing calibration standard depending on the instrument problem. 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.

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

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.

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 then compiles the initial data package for verification. This compilation must include sufficient documentation for data review. It may include standard calibrations, chromatograms, manual integration documentation, electronic printouts, chain of custody forms, and logbook copies.

Some agencies or customers require different levels of data reporting. For these special levels, the primary analyst may need to compile additional project information, such as initial calibration data or extensive spectral data, before the data package proceeds to the verification step.

7.2. Data Verification

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

Analysts performing the analysis and subsequent data reduction have primary responsibility for quality of the data produced. The primary analyst initiates the data verification process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. Data review checklists, either hardcopy or electronic, are used to document the data review process. The primary analyst is responsible for the initial input of the data into the LIMS.

The completed data package is then sent to a designated qualified reviewer (this cannot be the primary analyst). The following criteria have been established to qualify someone as a data reviewer. To perform secondary data reviewer, the reviewer must:

1. Have a current Demonstration of Capability (DOC) study on file and have an SOP acknowledgement form on file for the method/procedure being reviewed; or, See Note

2. Have a DOC on file for a similar method/technology (i.e. GC/MS) and have an SOP acknowledgment form on file for the method/procedure being reviewed; or, See Note

3. Supervise or manage a Department and have an SOP acknowledgment form on file for the method/procedure being reviewed; or,

4. Have significant background in the department/methods being reviewed through education or experience and have an SOP acknowledgment form on file for the method/procedure being reviewed.

Note: Secondary reviewer status must be approved personally by the Quality Manager or General Manager in the event that this person has no prior experience on the specific method or general technology.

This reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer also validates the data entered into the LIMS.

Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data (or designating the review of data electronically). The Operations or Project Manager examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution.

Additional information regarding data review procedures can be found in SOP S-ALL-Q-037 *Data Review* or its equivalent revision or replacement.

7.3. Data Reporting

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.

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

1. A title which designates the report as “Final Report”, “Laboratory Results”, “Certificate of Results”, etc.
2. Name and address of laboratory (or subcontracted laboratories, if used).
3. Phone number and name of laboratory contact to where questions can be referred.

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.
5. Name and address of customer and name of project
6. Unique identification of samples analyzed as well as customer sample IDs.
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.
8. Date and time of collection of samples, date of sample receipt by the laboratory, dates of sample preparation and analysis, and times of sample preparation and analysis when the holding time for either is 72 hours or less.
9. Identification of the test methods used.
10. Identification of sampling procedures if sampling was conducted by the laboratory.
11. Deviations from, additions to, or exclusions from the test methods. These can include failed quality control parameters, deviations caused by the matrix of the sample, etc., and can be shown as a case narrative or as defined footnotes to the analytical data.
12. Identification of whether calculations were performed on a dry or wet-weight basis.
13. Reporting limits used.
14. Final results or measurements, supported by appropriate chromatograms, charts, tables, spectra, etc.
15. A signature and title, electronic or otherwise, of person accepting responsibility for the content of the report.
16. Date report was issued.
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.
18. If necessary, a statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory.
19. Identification of all test results provided by a subcontracted laboratory or other outside source.
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:

21. Deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions.
22. Where relevant, a statement of compliance/non-compliance with requirements and/or specifications (e.g., the TNI standard).
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.
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.
25. Additional information which may be required by specific methods, regulatory agencies, or customers.

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.

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

The following positions are the only approved signatories for PASI final reports:

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

7.4. Data Security

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

All records compiled by PASI are maintained legible and retrievable and stored secured in a suitable environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. These records may include, but are not limited to, customer data reports, calibration and maintenance of equipment, raw data from instrumentation, quality control documents, observations, calculations, and logbooks. These records are retained in order to provide for possible historical reconstruction including sampling, receipt, preparation, analysis, and personnel involved. NELAP-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the Quality Manager or a designated Data Archivist.

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

In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained by the acquiring entity for a minimum of five years. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

7.6. Data Disposal

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

8.0. QUALITY SYSTEM AUDITS AND REVIEWS

8.1. Internal Audits

8.1.1. Responsibilities

The Quality Manager is responsible for designing and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be functionally independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The Quality Manger evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the 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

The complete internal audit process consists of the following four sections:

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

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

Examples of system-wide elements that can be audited include:

- Quality Systems documents, such as Standard Operating Procedures, training documents, Quality Assurance Manual, and all applicable addenda
- 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.

When the operations of a specific department are evaluated, a number of additional functions are reviewed including:

- Detection limit studies
- Internal chain of custody documentation
- Documentation of standard preparations
- Quality Control limits and Control charts

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

A representative number of data audits are completed annually. The report format of any non-conformance is similar to that of other internal audits.

The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the 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

Additional information can be found in SOP S-ALL-Q-011 *Audits and Inspections* or its equivalent revision or replacement.

A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the Quality Manager writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

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

Once completed, the internal audit report is issued jointly to the Laboratory General Manager and the manager(s)/supervisor(s) of the audited operation at a minimum. The responsible manager(s)/supervisor(s) responds within 14 days with a proposed plan to correct all of the deficiencies cited in the audit report. The Quality Manager may grant additional time for responses to large or complex deficiencies (not to exceed 30 days). Each response must include timetables for completion of all proposed corrective actions.

The Quality Manager reviews the audit responses. If the response is accepted, the Quality Manager uses the action plan and timetable as a guideline for verifying completion of the corrective action(s). If the Quality Manager determines that the audit response does not adequately address the correction of cited deficiencies, the response will be returned for modification.

To complete the audit process, the Quality Manager performs a re-examination of the areas where deficiencies were found to verify that all proposed corrective actions have been implemented. An audit deficiency is considered closed once implementation of the necessary corrective action has been audited and verified. This is usually within 60-90 days after implementation. If corrective action cannot be verified, the associated deficiency remains open until that action is completed.

8.2. External Audits

PASI laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

Audit teams external to the company review the laboratory to assess the effectiveness of systems and degree of technical expertise. The Quality Manager and other QA staff host the audit team and assist in facilitation of the audit process. Generally, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. In some cases, items of concern are discussed during a debriefing convened at the end of the on-site review process.

The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the Quality Manager. The Laboratory General Manager provides the necessary resources for staff to develop and implement the corrective action plans. The Quality Manager collates this information and provides a written response to the audit team. The response contains the corrective action plan and expected completion dates for each element of the plan. The Quality Manager follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

8.3. Quarterly Quality Reports

The Quality Manager is responsible for preparing a quarterly report to management summarizing the effectiveness of the laboratory Quality Systems. This status report will include:

- Overview of quality activities for the quarter
- Certification status
- Proficiency Testing study results
- SOP revision activities
- Company-wide 3P Document implementation (internal program)
- External audit findings
- Internal audit (method/system) findings
- Manual integration audit findings (Mintminer)
- Raw Data and Final Report review findings
- MDL activities
- Corrective action activities
- Training activity status

- Other significant Quality System items

The Corporate Director of Quality utilizes the information from each laboratory to make decisions impacting the Quality Systems of the company as a whole. Each General Manager utilizes the quarterly report information to make decisions impacting Quality Systems and operational systems at a local level.

Additional information can be found in SOP S-ALL-Q-014 *Quality System Review* or its equivalent revision or replacement.

8.4. Annual Managerial Review

A managerial review of Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements.

The managerial review must include the following topics of discussion:

- Suitability of quality management policies and procedures
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventative actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources, and staffing.

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

8.5. Customer Service Reviews

As part of the annual managerial review listed previously, the sales staff is responsible for reporting on customer feedback, including complaints. The acquisition of this information is completed by performing surveys.

The sales staff continually receives customer feedback, both positive and negative, and reports this feedback to the laboratory management in order for them to evaluate and improve their management system, testing activities and customer service.

In addition, the labs must be willing to cooperate with customers or their representatives to clarify customer requests and to monitor the laboratory's performance in relation to the work being performed for the customers.

9.0. CORRECTIVE ACTION

Additional information can be found in SOP S-ALL-Q-012 *Corrective Action/Preventive Action Process* 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

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

Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g. matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

The person who discovers the deficiency or non-conformance initiates the corrective action documentation on the Non-Conformance Corrective/ Preventative Action report and/or LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

In the event that the laboratory is unable to determine the cause, laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, 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 Lab Track or on the Corrective/Preventative Action Report.

After all the documentation is completed, the routing of the Corrective/Preventative Action Report and /or Lab Track will continue from the person initiating the corrective action, to their immediate supervisor or the Project Manager and finally to the Quality Manager, who is responsible for final review and signoff of all formal corrective/preventative actions.

In the event that analytical testing or results do not conform to laboratory procedures or customer requirements, the laboratory shall investigate the significance of the non-conformance and take appropriate corrective actions. Where necessary, the customer is notified and work is recalled. The procedures for handling non-conforming work are detailed in SOP S-ALL-Q-012 *Corrective Action/Preventive Action Process* or its equivalent revision or replacement.

9.2. Corrective Action Completion

9.2.1. Internal Laboratory Non-Conformance Trends

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

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting error

See applicable analytical SOPs for further guidance on QC acceptance criteria.

9.2.2. PE/PT Sample Results

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 Quality Manager reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the Quality Manager and reported to the applicable regulatory authorities.

9.2.3. Internal and External Audits

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

9.2.4. Data Review

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

9.2.5. Client Complaints

Project Managers are responsible for issuing corrective action forms, 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

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

9.2.7. Holding Time Violations

In the event that a holding time has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the Quality Manager must be made aware of all holding time violations.

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 Quality Manager includes a list of all missed holding times in their Quarterly Report to the corporate QA office.

9.3. Preventive Action Documentation

Pace laboratories can take advantage of several available information sources in order to identify needed improvements in all of their systems 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 NELAC 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.

When improvement opportunities are identified or if preventive action is required, the laboratory can develop, implement, and monitor preventive action plans.

10.0. GLOSSARY

3P Program	The Pace Analytical continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all PASI labs.
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. In the context of NELAP, this process is voluntary.
Accrediting Authority	The Territorial, State or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.
Accrediting (or 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 that are due to sampling and analytical operations; a data quality indicator.
Aliquot	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.
Analyst	The designated individual who performs the "hands-on" analytical methods and associated techniques and who is 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.
Assessment	The evaluation process used to measure the performance or effectiveness of a system and its elements against specific criteria (this 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	A process in which a sample is converted to free atoms.
Audit	A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch	Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same 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) that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
Bias	The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
Blind Sample	A sample submitted for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test analyst or laboratory proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.
Calibration	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	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 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	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of custody Form (COC)	A record that documents the possession of samples from the time of collection to receipt in the laboratory. This record generally includes the number and type of containers, mode of collection, collector, time of collection, preservation, and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Confirmation	Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: • second-column confirmation; • alternate wavelength; • derivatization; • mass spectral interpretation; • alternative detectors; or • additional cleanup procedures.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g. PCBs, PCDDs).
Consensus Standard	A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.

Continuing Calibration Verification	The verification of the initial calibration 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	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	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	The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more usable form.
Definitive Data	Analytical data of known quality, concentration and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making.
Demonstration of Capability	A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.
Detection Limit (DL)	The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration 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	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 (Management)	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled (managed) to ensure use of the correct version at the location where the prescribed activity is performed.
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (or Replicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g. PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental 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.

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.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Finding	An assessment conclusion 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. (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	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 (40 CFR Part 136). (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	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.

Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP-AES that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Verification (ICV)	A standard 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	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	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Internal Standards	A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties.

Laboratory Control Sample (LCS)	(however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of the total analytical system, including all preparation and analysis steps.
Laboratory Duplicate	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	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)	(TNI) An estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect. (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 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	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.

Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	The substrate of a test sample.
Matrix Duplicate	A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of precision of the recovery of each analyte.
Method	A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis) systematically presented in the order in which they are to be executed.
Method Blank	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures: and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.
MintMiner	Program used by Pace Analytical to review large amounts of chromatographic data to monitor for errors or data integrity issues.
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 (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.

Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Performance 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.
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	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
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	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	Any conditions under which a sample must be kept in order to maintain the chemical and/or biological integrity of the sample.
Proficiency Testing	A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Proficiency Testing Sample	A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within the specified acceptance criteria.
Protocol	A detailed written procedure for field and/or laboratory operation that must be strictly followed.
Quality Assurance (QA)	(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)	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)	The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality 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.

Quality System Matrix	These matrix definitions are to be used for purposes of batch and quality control requirements: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin • **Chemical Waste**: A product or by-product or an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other saltwater source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	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	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.
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	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.
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	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.
Sample Condition Upon Receipt Form (SCURF)	Form used by Pace Analytical sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selective Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector.

Selectivity	The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	The capability of a 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 as long as the requirement is fulfilled.
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 a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.

Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
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 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	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	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	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	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 objective evidence that specified requirements have been met. (DoD) 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).

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 revision 13.0	Increased font size of entire document. Section 1.7, fifth paragraph: changed length of time Technical Director can be gone before contacting primary authority (from 65 down to 35 days per TNI standard). Section 1.8.2: Reworded definition for Assistant GM to say "all departments". Fixed numbering issue with sub-sections for section 1.8 and used bullet points instead of numbers. Section 1.8.19: revised position title to capture requirement of some labs. Section 1.9: added language to second bullet point regarding LMS. Section 1.9: added bullet point for on-line courses. Section 2.5: added third note per request from GB (in red text). Section 2.6: Added chart of 2-digit codes (laboratory designations) per audit finding from GB laboratory (matches corporate SOPs). Section 2.7.4: added reference for Waste Handling and Management SOT. Section 3.1: added more method agency references. Section 3.4: added reference to Training SOP at end of section. Section 4.1: fixed numbering issue. Removed anonymous phone number and added reference to the Employee Handbook. Section 4.2: added paragraph of Ohio VAP required language (red text). Section 4.3, fifth paragraph: reworded second sentence for clarity. Section 4.3: added paragraphs of Ohio VAP and DoD required language (red text). Section 4.4, first paragraph: added qualifier to end of paragraph that MS limits are used to assess the batch if the MS is used in place of the LCS. Section 4.4: added paragraph of Ohio VAP required language (red text). Section 4.6: added paragraph of Ohio VAP required language (red text). Section 4.7: added paragraph of Ohio VAP required language (red text). Section 4.10: added paragraph of Ohio VAP required language (red text). Section 4.11: added paragraph of DoD required language (red text). Section 5.1, fifth paragraph: changed wording from LAN/WAN to local server (as opposed to hardcopies) and added language about LMS access. Section 5.1.2: added paragraphs of Ohio VAP and DoD required language (red text). Added new section 5.3- Management of Change. Section 6.2.1: added paragraph of Ohio VAP required language (red text). Section 6.3.2: changed NIST thermometer calibration frequency to every 3 years to match current practice. Section 7.3: added comment about Ohio VAP reporting (red text). Section 8.1.2, last sentence: reworded to match current practice. Section 8.1.3, last paragraph: reworded sentences regarding verification of corrective actions. Section 8.3: revised list of Quarterly Quality report items to match the revised SOP. Section 8.4: added last two bullet points and added second line of last paragraph to match ISO language. Section 9.1: changed bullet point items to match CAR SOP. Section 9.2.1: revised language to match SOP. Section 9.2.2: moved language from old 9.2.8 to 9.2.2. Section 9.2.4: added language to data review section.	30Apr2010

Document Number	Reason for Change	Date
	Glossary: Added definitions for analytical uncertainty, audit, bias, field of accreditation, finding, legal COC, matrix duplicate, method, PT sample, sampling, verification (per TNI standard). Glossary: Added definitions for reporting limit verification standard and initial calibration verification per request. Glossary: revised the following definitions to match new TNI language: DOC, LCS, LOD, MS, MSD, preservation, QA, QC, QC sample, raw data, reference standard, SOPs, and traceability. Also revised language within the definition for Quality System Matrix (previously just called Matrix). Glossary: deleted definition for 'detection limit'. Glossary: added definitions from company Acronym form from IT. Glossary: added definitions for LabTrack and MintMiner. Attachment VIII: added more tests to the chart per QM input including a line item for concentrated waste matrix for VOA 8260. General: changed all references to "Director of Quality, Safety, and Training" to "Director of Quality". General: revised document references to SOTs for Waste Handling and Management and Sample Management. Removed corporate org chart from Attachment IIB and will provide this as a separate document to the QMs. In this way, revisions to the corporate org chart will not necessitate a new QAM revision.	
Quality Assurance Manual 14.0	Cover page: moved signatures to cover page; added new form identification box in header. Added names of signatories Section 1.1 edited for clarity Section 1.3: added language to 3rd paragraph regarding compliance with method and client requirements (TNI V1M2, section 4.2). edited for clarity Section 1.4: Added language describing each of the Core Values, including wording from TNI V1M2, section 4.1 into the "Value Employees" line item. Section 1.5 added fourth bullet point Sections 1.6.2, 1.6.2, 1.6.3, 1.6.4 edited for clarity Section 1.7 edited for clarity Section 1.8.4: added bullet point to QM responsibilities pertaining to corrective actions (TNI V1M2, section 4.1.7.1). Section 1.8.4: added bullet point to QM responsibilities pertaining to the QAM (TNI V1M2, section 4.2.8.2). Section 1.9: reworded and reorganized parts of this section to match current practices using the LMS and for clarity. Section 1.10: moved Data Integrity section from Chapter 4 to Chapter 1.edited for clarity. Section 1.11 edited for clarity Sections 2.1, 2.3, 2.4, 2.5, 2.8, 2.9, 2.10 edited for clarity Section 2.5: added additional bullet point regarding qualified data (TNI V1M2, section 5.8.6.g). Section 2.6: added DoD red letter paragraph regarding time of collection (DoD gray box 14). Section 2.7.1: added new second paragraph to discuss storage blanks (DoD gray box 19). Section 2.7.2: reworded 2nd paragraph- temperatures are checked and documented each day of use (TNI V1M2, section 5.5.13.1.d). Section 2.9: added red letter section to the end of this section regarding DoD subcontract labs (DoD gray box 10). Section 2.9: added wording to the end of the fifth paragraph in this section regarding providing copies of subcontract reports to the client (TNI V1M2, section 4.5.5). 3.1 removed A2LA nad NVLAP and added Standard methods Section 3.2, 3.3 edited for clarity Section 3.4: added phrase to first paragraph (TNI V1M4, section 1.6.1). Section 3.4: added language about running DOC if a method had not been	17Feb2011

Document Number	Reason for Change	Date
	run for 12 months (TNI V1M4, section 1.6.2). Also removed language regarding 'work cells' (removed from TNI standard). Section 4.0; new general paragraph added to explain specifics regarding QC samples (TNI V1M2, section 5.9 and DoD gray box 45). Section 4.1, 4.2, 4.3, 4.4, 4.5, 4.6, 4.9, 4.10, 4.12 edited for clarity Section 4.1: added DoD red letter paragraph regarding method blank contamination (DoD gray box D-1). Section 4.1: added new 4th paragraph regarding method blanks (TNI V1M4, section 1.7.3.1.d). Section 4.2: added wording to 5th paragraph regarding exceedance of LCS limits and data qualifiers (TNI V1M4, section 1.7.4.2.a.i and ii). Section 4.3: added red-letter paragraph regarding MS/MSD requirements for DoD (DoD gray boxes D-7 and D-8). Section 4.4: added wording to 2nd paragraph regarding client-specific surrogate criteria (DoD gray box D-12). Section 4.10: removed sentence from 2nd paragraph ('sufficient buffer' was too vague). Section 4.11: adjusted title of section. Section 5.1: added reference to electronic signature policy in Document Mgmt SOP (TNI V1M2, section 4.2.8.4.r). Section 5.1.2: added language to DoD red letter paragraph (DoD gray box 20).edited for clarity Section 6.1: reworded 5th paragraph to compensate for change in TNI language regarding second source standards (TNI V1M4, section 1.7.1.1.d). Added small vial labeling requirement. Added paragraph about minimum requirements for labeling. Section 6.2, 6.2.1, 6.2.2, 6.3, 6.3.5, 6.4, 7.3, 8.1.1, 8.4 edited for clarity Section 6.3: reworded 2nd paragraph- that all measurements are performed on each day if use and documented (TNI V1M2, section 5.5.13.1.d). Section 6.3.1 added "bracketing the range of use" also edited for clarity Section 7.2: added additional red-letter section for DoD data review steps to be added (DoD gray box 44) and added reference to Data Review SOP. Section 7.3: added DoD red letter paragraph regarding the recording of sample prep and analysis time on final reports (DoD gray box 14). Section 7.3: Added extra requirements for final reports when needed for interpretation of results (TNI V1M2, section 5.10). Section 8.4: edited for clarity Section 9.1: added paragraph regarding non-conforming work (TNI V1M2, section 4.9). Section 9.2.1 added login error Section 9.3 edited for clarity Section 11: added DoD QSM version 4.2 as a reference. Glossary: **added definitions** for NIST, reference material, standard (document), selectivity, acceptance criteria, accreditation, accrediting authority, accrediting body, analyst, assessment, atomization, calibration method, calibration range, calibration standard, certified reference material, chain of custody (as opposed to COC form), client (customer), congener, conformance, consensus standard, continuing calibration verification (as opposed to CCV standard), data audit, definitive data, detection limit, digestion, eluent, elute, elution, environmental data, environmental monitoring, homologue, inspection, interference (spectral and chemical), SI, instrument blank, isomer, laboratory duplicate, LOQ (added DoD version), manager, management, management system, matrix, method of standard additions, negative control, nonconformance, performance audit, positive control, quality manual, quantitation range, reagent blank, reference standard (DoD version), reporting limit (DoD version), requirement, retention time, sample, shall, should, signal-to-noise ratio, spike, standard method, standard reference material, supervisor, target analytes, technical	

Document Number	Reason for Change	Date
	director, test, test method, traceability (DoD version), tuning, validation. Glossary: **revised definitions** for standard (chemical), accuracy, aliquot, analyte, batch, calibration, calibration curve, confirmation, corrective action, document control, duplicate, finding, holding time (added DoD version too), LCS, LOD (added DoD version too), MDL (adopted DoD clarification since TNI dropped MDL), precision, PT sample, quality assurance (added DoD version), QAPP, raw data, ICV, verification (added DoD note). Glossary: **removed definition** for calibration verification.	

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 ($\overline{X}$)

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

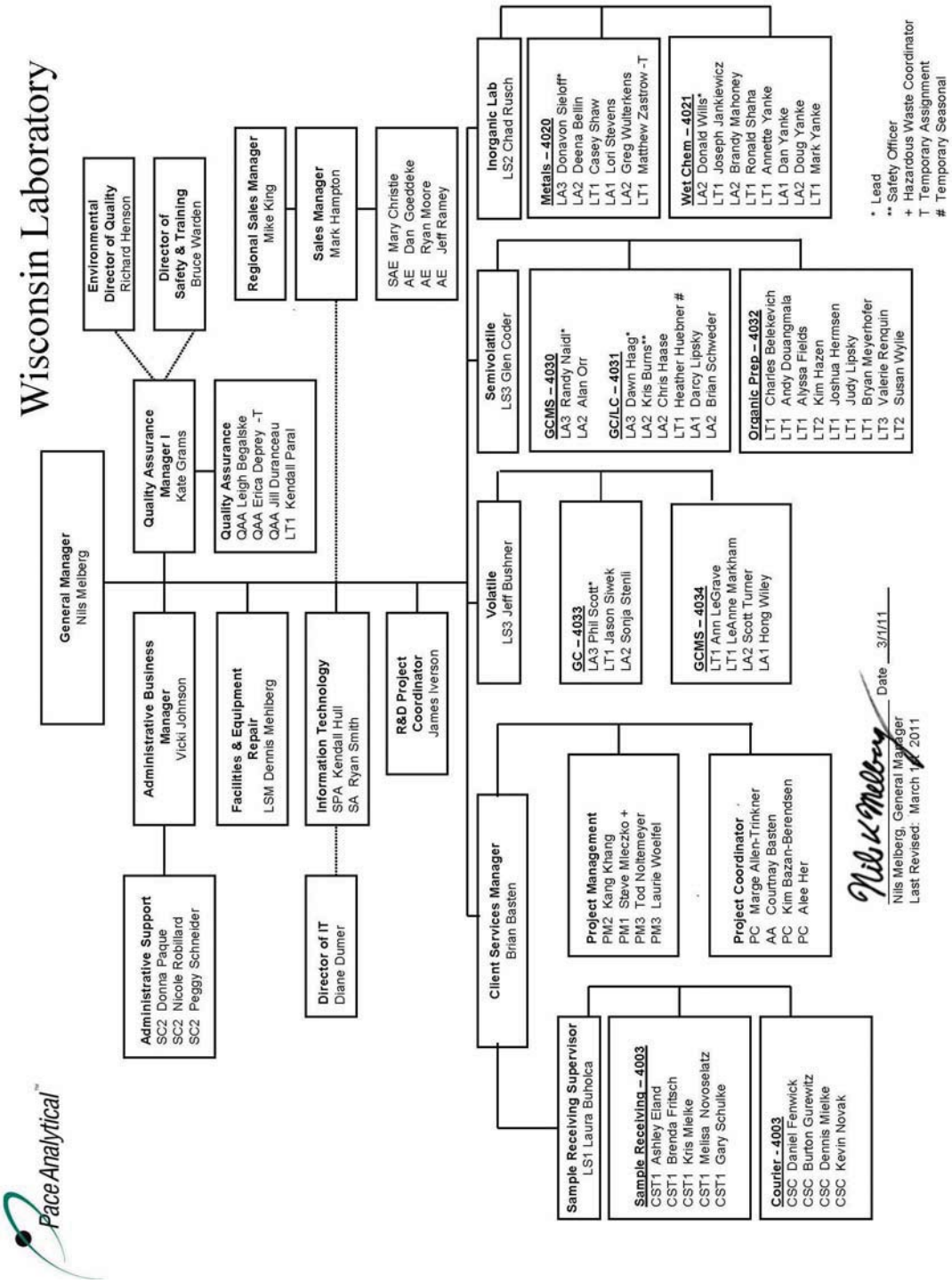
where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

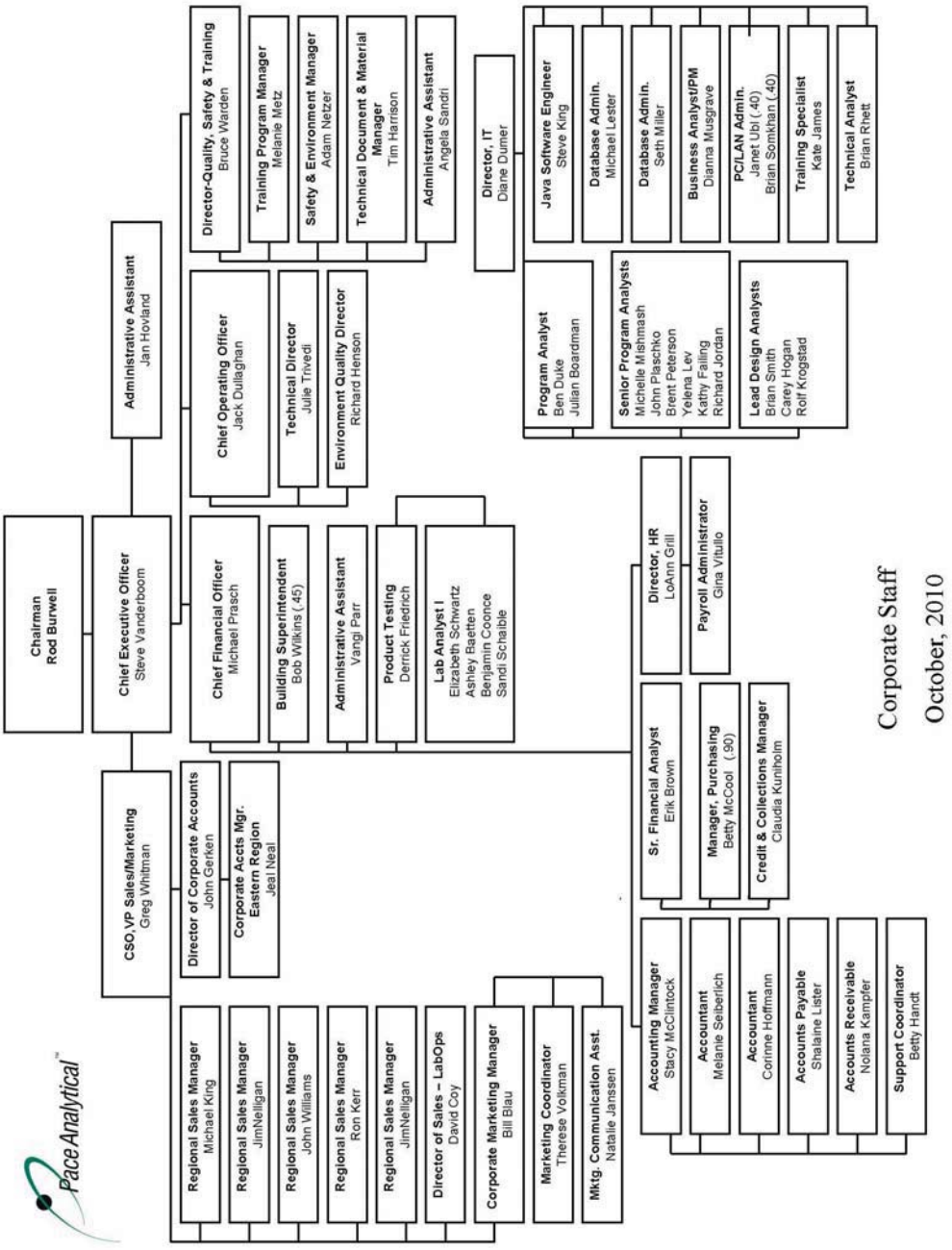
$$RSD = \frac{S}{\overline{X}} * 100$$

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

ATTACHMENT IIA- PACE GREEN BAY LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



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



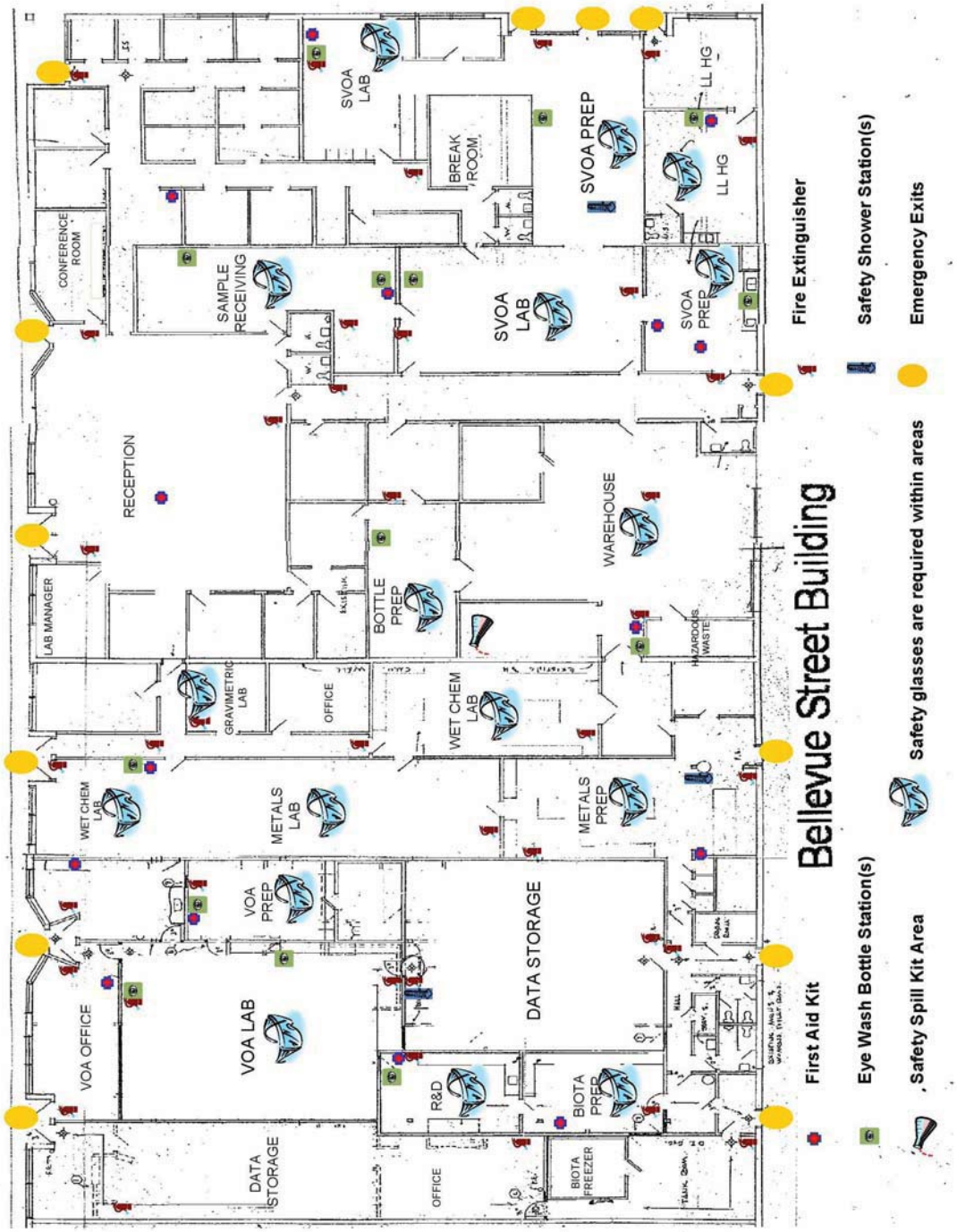
ATTACHMENT III- EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
SR	Total Solids Oven	40OVN1	Precision Scientific	STG80		ASTM D2974-87
SR	Total Solids Oven	40OVN2	Precision Scientific	1025		ASTM D2974-87
SVOA	GC/MS	40MSS1	Hewlett-Packard	5890	MS	8270C/625
SVOA	GC	40GCS6	Hewlett-Packard	5890	ECD/ECD	8081
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	40GCS7	Hewlett-Packard	5890	ECD/ECD	
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/MS	40MSS2	Hewlett-Packard	6890	MS	8270C-SIM
SVOA	GC/MS	40MSS3	Hewlett-Packard	6890	MS	8270C-SIM
SVOA	GC/MS	40MSS4	Hewlett-Packard	6890	MS	8270C-SIM
SVOA	GC/MS	40MSS5	Hewlett-Packard	5890	MS	8270 SCREEN
SVOA	GC/MS	40MSS6	Hewlett-Packard	5890	MS	8270C
SVOA	GC/MS	40MSS7	Hewlett-Packard	7890A	MS	8270C
SVOA	GC	40GCS1	Hewlett-Packard	5890	FID	WI-DRO/8015
SVOA	GC	40GCS2	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	40GCSF	Hewlett-Packard	5890	FID	WI-DRO/8015
SVOA	Separatory Funnel Extractor	#1	Lab Line	-	-	SW846 3510C
SVOA	Separatory Funnel Extractor	#2	Lab Line	-	-	SW846 3510C
SVOA	Separatory Funnel Extractor	#3	Lab Line	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	#1	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	#2	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	#3	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	#4	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	#5	Zymark	-	-	SW846 3510C
SVOA	Accuprep MPS GPC Cleanup System	John	J2 Scientific	-	-	SW846 3640A
SVOA	Accuprep MPS GPC Cleanup System	Jane	J2 Scientific	-	-	SW846 3640A
SVOA	Mars Xtraction Microwave System		CEM	907501	-	SW846 3546
SVOA	Soxtherm Accelerated Soxhlet Extractor	#1	Gerhardt Soxtherm	SE-30	-	SW846 3541
SVOA	Soxtherm Accelerated Soxhlet Extractor	#2	Gerhardt Soxtherm	SE-30	-	SW846 3541
SVOA	Soxtherm Accelerated Soxhlet Extractor	#3	Gerhardt Soxtherm	SE-30	-	SW846 3541
SVOA	Soxtherm Accelerated Soxhlet Extractor	#4	Gerhardt Soxtherm	SE-30	-	SW846 3541
SVOA	Soxtherm Accelerated Soxhlet Extractor	#1	Gerhardt Soxtherm	SE-416	-	SW846 3541
SVOA	Soxtherm Accelerated Soxhlet Extractor	#2	Gerhardt Soxtherm	SE-416	-	SW846 3541
SVOA	Soxtherm Accelerated Soxhlet Extractor	#3	Gerhardt Soxtherm	SE-416	-	SW846 3541
SVOA	Soxtherm Accelerated Soxhlet Extractor	#4	Gerhardt Soxtherm	SE-416	-	SW846 3541
SVOA	Sonifier Cell Disruptors with Horns	Bob	-	-	-	SW846 3550B
SVOA	Sonifier Cell Disruptors with Horns	Ralph	-	-	-	SW846 3550B
SVOA	Oven	40OVN7	VWR Scientific	1370 FM		Air Dry Dry Wt. Sed/Tissue Dry Wt.
SVOA	Oven	40OVN8	National Appliance Co	5861-8		Regulated Soil Disposal
SVOA	Muffle Furnace	40OVN9	American Scientific	FP-41		Sodium Sulfate Baking
SVOA	Oven	40OVNA	VWR Scientific	1350 FM		Air Dry Dry Wt. Sed/Tissue Dry Wt.
SVOA	Oven	40OVNH	VWR Scientific	1370 GM		Air Dry Dry Wt. Sed/Tissue Dry Wt.
VOA	GC/MS	40MSV1	Hewlett-Packard	5890	MS	SW846 8260B/5030
VOA	GC/MS	40MSV3	Agilent	6850	MS	SW846 8260B/624/5030B
VOA	GC/MS	40MSV4	Hewlett-Packard	5890	MS	SW846 8260B/624/5030B

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
VOA	GC/MS	40MSV5	Hewlett-Packard	6890	MS	SW846 8260B/624/5030B
VOA	GC/MS	40MSV6	Hewlett-Packard	5890	MS	SW846 8260B/624/5030B
VOA	GC/MS	40MSV7	Hewlett-Packard	6890	MS	SW846 8260B/624/5030B
VOA	GC/MS	40MSV8	Agilent	6850	MS	SW846 8260B/624/5030B
VOA	GC/MS	40MSV9	Hewlett-Packard	6890	MS	SW846 8260B/624/5030B
VOA	GC/MS	40MSV2	Hewlett-Packard	6890	MS	SW846 8260B/624/5030B
VOA	GC/MS	40MSVA	Hewlett-Packard	7890	MS	SW846 8260B/624/5030B
VOA	GC	40GCV1	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV2	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
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	Screen 1	Hewlett-Packard	5890	FID	Screener
VOA	GC	Screen 2	Hewlett-Packard	5890	FID	Screener
VOA	Sonicator	1	9314	294	-	VOA Prep
VOA	Sonicator	2	9333	0194-03	-	VOA Prep
VOA	Oven	40OVNB	Scientific Products	M8620-10A		Glassware Drying
WET	Fusion	40WTA7	Teledyne	14-9600-100	-	EPA 9060, SM 5310C
WET	Apollo	40WTA5	Tekmar/Dohrmann	Apollo 9000	-	EPA 9060
WET	Spectronic 21D Spectrophotometer	40WTA8	Milton Roy	-	-	EPA 1629 (AVS)
WET	SmartChem	40WTA9	Westco Scientific	Smartchem 200		350.1, 365.4, 9012A, 310.2, 351.2, 353.2
WET	pH Meter	40WET8	Orion	720A	-	pH Meter
WET	pH Meter	-	Orion	720A	-	pH Meter
WET	pH Meter	-	Corning	320	-	pH Meter
WET	EH Meter	40WET9	Accument	AB15	-	EH Meter
WET	Radiometer	40WET0	Titration	TIM840		Alkalinity - 2320B
WET	Oxygen Meter	40WET1	YSI	5000	YSI 5010	SM 5210B--01, SM 4500-O G
WET	Turbidimeter	40WET6	Hach	-	-	EPA 180.1, SM2130B
WET	Conductivity Meter	40WET7	Accument	30	-	EPA 120.1
WET	Flashpoint Instrument A	40WET4	Precision	-	-	EPA 1010A
WET	Flashpoint Instrument B	40WET5	Fisher	-	-	EPA 1010A
WET	TDS Oven	Oven #3	VWR	1305U	-	TVS Analysis
WET	TSS Oven	Oven #1	Yamato	DKN 600	-	TDS, TSS, TS Analysis
WET	Total Solids Oven	Oven #5	-	-	-	
WET	Lindberg Muffle Furnace	Muffle Furnace	Lindberg	51714	-	TVS, OM, FOC
WET	MICRO DIST Rapid Distillation System	A	Lachat	-	-	350.1, 9012A
WET	MICRO DIST Rapid Distillation System	B	Lachat	-	-	350.1, 9012A
WET	MICRO DIST Rapid Distillation System	C	Lachat	-	-	350.1, 9012A
WET	Block Digestion System	-	Westco Scientific	40/20	-	365.4, 351.2

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
WET	Block Digestion System	-	Tecator	2040	-	365.4, 351.2
WET	COD Reactor	-	Hach	45600	-	410.4
WET	COD Reactor	-	Bioscience INC	100 003	-	410.4
WET	Ion Chromatograph	40WTA3	Dionex	DX-120	-	EPA 300.0, 9056A
WET	Ion Chromatograph	40WTA4	Dionex	DX-120	-	EPA 300.0, 9056A
WET	Quik Chem 8000	40WTA2	Lachat	8000	-	9012A, 310.2, 351.2, 353.2
WET	Direct Reading Spectrophotometer	40WTA1	Hach	DR 2000	-	SM3500Cr-B, Hach 8146, Walkley Black TOC
WET	Hellige Colony Counter	-	Hellige	-	-	
WET	Incubator	40INC05	Millipore	-	-	
WET	Incubator	40INC06	Lab-Line	-	-	
WET	Stereomaster Stereoscope	-	Fisher	FW 99-20-1385	-	
MET	ICP	40ICP2	Thermo	ICAP 6500	-	6010
MET	Chiller	40ICP2	Neslab/Thermo	Flex 900	-	6010
MET	ICPMS	40ICM2	Thermo	X Series 2	-	6020
MET	Chiller	40ICM2	Neslab/Thermo	Merlin M75	-	6020
MET	ICPMS	40ICM3	Thermo	X Series 2	-	6020
MET	Chiller	40ICM3	Neslab/Thermo	2500	-	6020
MET	PE FIMS (Flow Injection Hg System)	40HG1	Perkin Elmer	FIMS 100	-	7470/7471
MET	Hot Block Metals Digestion System	#1	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	#2	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	#3	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	#4	Environmental Express	SC154	-	6010/6020
MET	Hot Block Metals Digestion System	#5	Environmental Express	SC100	-	6010/6020
MET	Low-level Mercury Analyzer	40LHG1	Tekran	2500	2500	1631E
MET	Low-level Mercury Analyzer	40LHG2	Tekran	2500	2500	1631E
MET	Low Level Automated Mercury Analyzer	40LHG3	Tekran	2600	2600	1631E
MET	Low Level Mercury Analyzer	40LHG4	Analytik Jena	Mercu		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 Publication	Signature Date	Department
S-ALL-C-002	01	Bottle Order Database	4-Oct-06	22-Sep-09	Sample Management
S-ALL-C-005	00	Operation of PacePort Customer Feedback Form	27-Nov-07	4-Mar-09	Sample Management
S-ALL-C-006	00	Review of Analytical Requests	23-Dec-08	4-Mar-09	Sample Management
S-GB-C-001	03	Procedure to Preserve Samples for Volatile Organic Analysis of Solid Matrices by Method 5035	12-Apr-05	7-Jun-10	Sample Management
S-GB-C-002	03	Extruding Sample from 25 g EnCores for Volatile and Semivolatile Analyses	12-Apr-05	7-Jun-10	Sample Management
S-GB-C-005	03	Maintenance of Ice Chests and Shipping Containers	1-Jun-05	15-Sep-10	Sample Management
S-GB-C-007	02	Laboratory Tracking of Samples	6-Jun-05	22-Sep-08	Sample Management
S-GB-C-008	01	Measurement of Percent Moisture in Soils and Solids	19-Dec-06	4-Nov-09	Sample Management
S-GB-C-009	01	Subcontracting Samples	31-Mar-05	1-Mar-11	Sample Management
S-GB-C-010	01	Sample Management	12-Jul-04	1-Mar-11	Sample Management
S-GB-C-011	00	Bottle Preparation	4-Oct-06	30-Aug-10	Sample Management
S-GB-M-001	01	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry by 1631	1-Dec-06	30-Jan-09	Metals
S-GB-M-003	01	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry Solids - Manual System	12-Oct-07	16-Sep-10	Metals
S-GB-M-004	03	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy (7470A/7471A_B)	28-Aug-06	16-Sep-10	Metals
S-GB-M-005	02	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010C	23-Sep-08	13-May-10	Metals
S-GB-M-006	03	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020A	25-Sep-08	16-Sep-10	Metals
S-GB-M-007	01	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020	13-May-10	16-Sep-10	Metals
S-GB-M-008	01	Cleaning Metals Glassware	5-May-05	13-May-10	Metals
S-GB-M-009	01	Hardness by Calculation	27-Oct-05	29-May-09	Metals
S-GB-M-010	00	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry - Automated Tekran 2600	1-Dec-06	30-Jan-09	Metals
S-GB-M-011	01	The Determination of Mercury in Biological Samples by Cold Vapor Atomic Absorption Spectroscopy (245.6)	28-Dec-06	13-May-10	Metals
S-GB-M-013	00	Soil Fraction Preparation for Lead Analysis	13-Mar-06	13-May-10	Metals
S-GB-M-014	00	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010B	28-Feb-07	13-May-10	Metals
S-GB-I-028	02	Soil Fraction Preparation for Lead Analysis	21-Mar-06	3-Jan-08	Metals
S-ALL-GB-I-015	01	Measurement of pH in Water, Soil, and Waste	12-Dec-06	26-May-09	Wetchem
S-GB-I-001	06	Total Sulfide, Iodometric Titration	26-Jan-05	5-Feb-10	Wetchem
S-GB-I-002	05	Flash Point (Pensky-Martens Closed Cup Method For Ignitability	27-Jan-05	18-Nov-10	Wetchem
S-GB-I-004	03	Acidity	27-Jan-05	16-Dec-09	Wetchem
S-GB-I-009	03	Ion Chromatography- Sulfate, Nitrate, Flouride (EPA 300.0)	7-Feb-05	29-Dec-09	Wetchem
S-GB-I-010	02	Wet Chemistry Glassware Cleaning	5-Feb-05	15-Dec-09	Wetchem
S-GB-I-013	02	Free Liquids (Paint Filter)	13-Jul-05	12-Mar-10	Wetchem
S-GB-I-014	02	Alkalinity	18-Jul-05	26-May-09	Wetchem
S-GB-I-015	02	Oxidation - Reduction Potential (Eh) Measurement	29-Jul-05	9-Mar-10	Wetchem
S-GB-I-016	02	Specific Gravity	25-Aug-05	9-Mar-10	Wetchem
S-GB-I-017	02	Ferrous Iron	30-Aug-05	9-Mar-10	Wetchem
S-GB-I-019	03	Fecal Coliform Determination Using the Membrane Filter Technique	13-Sep-05	7-Jun-10	Wetchem
S-GB-I-020	03	Color Determination in Aqueous Samples	15-Sep-05	9-Mar-10	Wetchem
S-GB-I-023	03	Heterotropic Plate Count	17-Nov-05	13-May-10	Wetchem
S-GB-I-024	03	Colisure Presence/Absence Test for Detection and Identification of Coliform Bacteria and *Escherichia coli* in Drinking Waters	28-Nov-05	13-May-10	Wetchem
S-GB-I-027	02	Dissolved Oxygen	1-Mar-06	9-Mar-10	Wetchem
S-GB-I-030	02	Turbidity (Nephelometric)	10-Apr-06	18-Nov-10	Wetchem

SOP Number	Rev.	SOP Name	Date of Publication	Signature Date	Department
S-GB-I-037	03	The Determination of Total Organic Carbon Using the Walkley-Black Procedure	8-Aug-05	7-Jan-11	Wetchem
S-GB-I-042	02	The Determination of Total Organic Carbon Using the Apollo 9000 Instrument for MDEQ RRD Operational Memorandum No. 2	4-Jan-07	22-Nov-10	Wetchem
S-GB-I-044	02	Biochemical Oxygen Demand	15-Feb-07	7-Jan-11	Wetchem
S-GB-I-045	03	Chromium, Hexavalent-Colorimetric	15-Feb-07	7-Jan-11	Wetchem
S-GB-I-047	01	Total Kjeldahl Nitrogen using Block Digestion and Analyzed by Lachat 8000 Flow Injection following EPA Method 351.2	17-Feb-07	29-Dec-08	Wetchem
S-GB-I-048	01	Ammonia using Micro-Distillation and Analyzed by Lachat 8000 Flow Injection following EPA Method 350.1	17-Feb-07	29-Dec-08	Wetchem
S-GB-I-051	01	Nitrate and Nitrite Analyzed by Lachat 8000 Flow Injection	20-Feb-07	5-Sep-08	Wetchem
S-GB-I-052	03	Chemical Oxygen Demand, Colorimetric, Manual (Chemetric Vials)	21-Feb-07	7-Jan-11	Wetchem
S-GB-I-053	01	Acid Volatile Sulfide / Simultaneously Extracted Metals	4-Apr-07	16-Nov-09	Wetchem
S-GB-I-059	02	The Determination of Total Organic Carbon Using the Apollo 9000 Instrument	21-May-09	18-Nov-10	Wetchem
S-GB-I-060	00	Ammonia using Micro-Distillation and Analyzed by SmartChem	1-Jun-09	21-Jan-10	Wetchem
S-GB-I-061	01	Total Phosphorus using Block Digestion and Analyzed by SmartChem	1-Jun-09	16-Sep-10	Wetchem
S-GB-I-063	01	The Determination of Total Organic Carbon Using the Teledyne Tekmar Fusion Instrument	28-Dec-09	18-Nov-10	Wetchem
S-GB-I-064	00	Cyanide by SmartChem	18-Jan-10	21-Jan-10	Wetchem
S-GB-I-066	00	Measurement of Specific Conductance in Water	12-Dec-06	18-Nov-10	Wetchem
S-GB-I-067	00	TCLP - Toxicity Characteristic Leaching Procedure, SPLP - Synthetic Precipitation Leaching Procedure and ASTM - ASTM D 3987-85	5-Jul-10	10-Jul-10	Wetchem
S-GB-I-068	00	Measurement of Volatile Solids and Solids in Water	24-Aug-07	18-Nov-10	Wetchem
S-GB-I-069	00	Alkalinity by Titration using the Radiometer TIM840 - SM 2320B	17-Dec-10	1-Mar-11	Wetchem
S-GB-O-005	01	Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO	29-Apr-05	5-Dec-07	Gas
S-GB-O-005	02	Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO	29-Apr-05	20-Nov-08	Gas
GB-O-006	00	Modified Method for Determination of Gasoline Range Organics		27-Apr-05	Gas
S-GB-O-006	01	Modified Method for Determination of Gasoline Range Organics	27-Apr-05	4-Oct-07	Gas
S-GB-O-006	02	Modified Method for Determination of Gasoline Range Organics	27-Apr-05	20-Nov-08	Gas
S-GB-O-008	01	Total Petroleum Hydrocarbons - Gasoline by Gas Chromatography Using Flame-ionization Detectors	15-Jun-05	28-Nov-07	Gas
S-GB-O-008	02	Total Petroleum Hydrocarbons - Gasoline by Gas Chromatography Using Flame-ionization Detectors	15-Jun-05	21-Nov-08	Gas
S-GB-O-008	03	Total Petroleum Hydrocarbons - Gasoline by Gas Chromatography Using Flame-ionization Detectors	15-Jun-05	8-Jun-10	Gas
S-GB-O-009	01	Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors	27-Apr-05	4-Oct-07	Gas
S-GB-O-009	02	Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors	27-Apr-05	21-Nov-08	Gas
S-GB-O-010	01	Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics	29-Apr-05	28-Nov-07	Gas

SOP Number	Rev.	SOP Name	Date of Publication	Signature Date	Department
S-GB-O-010	02	Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics	29-Apr-05	22-Dec-08	Gas
S-GB-O-017	01	Analysis of Dissolved Methane, Ethane, and Ethene in Ground Water by Static Headspace and Gas Chromatography	27-Apr-05	7-Dec-07	Gas
S-GB-O-017	02	Analysis of Dissolved Methane, Ethane, and Ethene in Ground Water by Static Headspace and Gas Chromatography	27-Apr-05	29-Dec-08	Gas
S-GB-O-005	03	Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO	29-Apr-05	8-Jun-10	Gas
S-GB-O-006	03	Modified Method for Determination of Gasoline Range Organics	27-Apr-05	8-Jun-10	Gas
S-GB-O-008	04	Total Petroleum Hydrocarbons - Gasoline by Gas Chromatography Using Flame-ionization Detectors	15-Jun-05	15-Sep-10	Gas
S-GB-O-009	03	Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors	27-Apr-05	8-Jun-10	Gas
S-GB-O-010	03	Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics	29-Apr-05	8-Jun-10	Gas
S-GB-O-017	03	Analysis of Dissolved Methane, Ethane, and Ethene in Ground Water by Static Headspace and Gas Chromatography	27-Apr-05	8-Jun-10	Gas
SVOA SECTION					
S-GB-O-015	03	Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics	11-May-05	7-Aug-09	SVOA
S-GB-O-018	02	Determination of DRO Sample Weight and Methylene Chloride Addition	13-Apr-05	13-Apr-10	SVOA
S-GB-O-019	03	WI Modified Method for Determination of Diesel Range Organics	13-Apr-05	5-Apr-10	SVOA
S-GB-O-023	04	Total Petroleum Hydrocarbons	25-May-05	20-Nov-08	SVOA
S-GB-O-023	05	Total Petroleum Hydrocarbons - Diesel	25-May-05		SVOA
S-GB-O-025	02	Alcohols & Glycols by Direct Injection GC/FID	14-Dec-05	8-Jul-10	SVOA
S-GB-O-026	04	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by 8082	23-May-06	20-Apr-10	SVOA
S-GB-O-027	03	Analysis of Organochlorine Pesticides by Gas Chromatography by 8081	23-May-06	11-May-09	SVOA
S-GB-O-028	03	Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes	24-May-06	16-Apr-10	SVOA
S-GB-O-031	02	Extraction of Biological Samples for Organochlorine Pesticides/PCBs	20-Jan-05	20-Apr-10	SVOA
S-GB-O-032	02	Gel Permeation Chromatography	26-Jan-05	20-Apr-10	SVOA
S-GB-O-033	01	Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis	8-Feb-05	6-May-10	SVOA
S-GB-O-034	02	Sulfuric Acid Cleanup	3-Feb-05	13-Apr-10	SVOA
S-GB-O-035	02	Mercury Cleanup for the Removal of Sulfur from PCB and Toxaphene Samples	2-Feb-05	13-Apr-10	SVOA
S-GB-O-036	02	Florisil Cleanup for PCB and Toxaphene Samples	26-Jan-05	13-Apr-10	SVOA
S-GB-O-037	02	Florisil Cartridge Cleanup from PCB and Toxaphene Samples	26-Jan-05	13-Apr-10	SVOA
S-GB-O-038	03	Silica Gel Cleanup for Organic Analysis	3-Feb-05	10-Jul-10	SVOA
S-GB-O-039	02	Copper Cleanup for the Removal of Sulfur from PCB and Toxaphene Samples	23-Aug-05	13-Apr-10	SVOA
S-GB-O-040	03	Extraction of Wipes and Oil for PCB Analysis	11-Jul-08	19-Apr-10	SVOA
S-GB-O-041	03	Extraction of PCBs Using the Automated Soxhlet 3541	23-Aug-07	20-Apr-10	SVOA
S-GB-O-043	02	Extraction of Toxaphene Using the Automated Soxhlet 3541	3-May-07	20-Apr-10	SVOA
S-GB-O-044	01	Determination of Low Level PAHs by GC/MS-SIM in Solid and Biological Matrices:8270SIMPAH	5-Feb-05	24-Jan-08	SVOA
S-GB-O-045	03	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-May-10	SVOA

SOP Number	Rev.	SOP Name	Date of Publication	Signature Date	Department
S-GB-O-047	02	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography following 8082A	22-Sep-08	15-Sep-10	SVOA
S-GB-O-048	00	Analysis of Fox River Polychlorinated Biphenyls (PCBs) by Gas Chromatography	4-Jun-09	4-Jun-09	SVOA
S-GB-O-049	03	Determination of Semi-Volatile Organics by GC/MS (8270)	28-Nov-06	9-Jul-10	
S-GB-O-050	00	Determination of Semi-Volatile Organics by gc/ms (Selective Ion Monitoring)	28-Nov-06	9-Jul-10	SVOA
S-GB-O-052	00	Extraction of PCBs In Tissue Using the Automated Soxhlet	11-Nov-09	20-Nov-09	SVOA
S-GB-O-053	00	Separatory Funnel Extraction by 3510C	16-Nov-06	12-Jan-10	SVOA
S-GB-O-054	00	Ultrasonic Extraction by 3550B	16-Nov-06	18-Mar-10	SVOA
S-GB-O-057	00	Analysis of Toxaphene by Gas Chromotography Using St.John's River Waste Management Department (SJRWMD) Protocol	10-Jul-10	10-Jul-10	SVOA
S-GB-O-058	00	Analysis of Toxaphene by Gas Chromotography Using Hercules 97 and Total Area Under the Curve (TAUC)	7-Jul-10	7-Jul-10	SVOA
S-GB-O-001	04	Sample Screening Volatile Organics Prior to Preparation	24-Aug-05	9-Apr-10	VOA
S-GB-O-012	03	Cleaning of Syringes Used in the Analysis of Volatile Organics	12-Apr-05	13-May-10	VOA
S-GB-O-056	02	Determination of Volatile Organics by GC/MS 8260	14-Jul-06	15-Sep-10	VOA
S-ALL-IT-001	01	System Security and Integrity	26-Jan-05	15-Apr-05	IT
S-ALL-IT-002	02	Server Back-up	4-Jun-09	12-Oct-09	IT
T-ALL-IT-001	04	EPIC Pro 01: Basic System Functions	30-Oct-08	30-Oct-08	IT
T-ALL-IT-002	05	EPIC Pro 02: Client Setup	30-Oct-08	30-Oct-08	IT
T-ALL-IT-004	03	EPIC Pro: PMs / Sales II	30-Oct-08	30-Oct-08	IT
T-ALL-IT-005	04	EPIC Pro: Login	30-Oct-08	30-Oct-08	IT
T-ALL-IT-006	04	EPIC PRO: Lab Prep	30-Oct-08	30-Oct-08	IT
T-ALL-IT-007	03	EPIC Pro: Analyst / Lab Management	30-Oct-08	30-Oct-08	IT
T-ALL-IT-008	05	EPIC Pro: PM - Additional Knowledge	30-Oct-08	30-Oct-08	IT
T-ALL-IT-009	01	EPIC Pro: Detection, Reporting and Control Limits	14-Oct-04	30-Oct-08	IT
T-ALL-IT-010	02	EPIC Pro: Standard Traceability	30-Oct-08	30-Oct-08	IT
S-ALL-Q-001	09	Preparation of Standard Operating Procedures	5-May-04	15-Sep-10	QA
S-ALL-Q-002	02	Document Management	30-Dec-04	5-Jan-09	QA
S-ALL-Q-003	04	Document Numbering	12-Apr-04	16-Sep-10	QA
S-ALL-Q-004	05	Method Detection Limit Studies	21-Feb-05	22-Sep-09	QA
S-ALL-Q-007	02	EPIC Pro: Acode Validation	15-Feb-05	16-Sep-10	QA
S-ALL-Q-008	01	EPIC Pro: Acode Addition/Modification	7-Jun-10	7-Jun-10	QA
S-ALL-Q-009	03	Laboratory Documentation	22-Dec-04	18-Nov-10	QA
S-ALL-Q-010	03	Proficiency Testing Program	18-Oct-06	19-Mar-10	QA
S-ALL-Q-011	03	Audits and Inspections	30-Dec-04	16-Sep-10	QA
S-ALL-Q-012	02	Corrective Action / Preventative Action Process	17-Aug-05	7-Jun-10	QA
S-ALL-Q-013	02	Support Equipment	17-Jan-05	18-Nov-10	
S-ALL-Q-014	02	Quality system Review	5-May-04	7-Jun-10	QA
S-ALL-Q-016	04	Manual Integration	15-Mar-05	18-Nov-10	QA
S-ALL-Q-018	03	Monitoring Storage Units	20-Apr-05	18-Nov-10	QA
S-ALL-Q-020	03	Orientation and Training Procedures	30-Jun-05	23-Sep-09	QA
S-ALL-Q-021	04	Sample Homogenization and Sub-Sampling	16-Nov-06	18-Nov-10	QA
S-ALL-Q-022	02	3P Program Continuous Process Improvement	19-May-05	15-Mar-10	QA
S-ALL-Q-025	04	Standard and Reagent Management and Traceability	9-Feb-11	9-Feb-11	QA
S-ALL-Q-026	01	Software Validation	9-Jun-09	22-Sep-09	QA
S-ALL-Q-027	01	Evaluation and Qualification of Vendors	10-Apr-08	24-Aug-09	QA
S-ALL-Q-028	01	Use and Operation of Lab Track System	22-May-07	16-Jul-10	QA
S-ALL-Q-029	01	MintMiner Data File Review	20-Nov-07	19-Mar-10	QA
S-ALL-Q-030	03	Operation of Data Checker for Epic Pro	5-Aug-10	16-Sep-10	QA
S-ALL-Q-032	00	Control Charting and Trend Analysis	9-Feb-11	9-Feb-11	
S-ALL-Q-033	00	MCL Violation Reporting	11-May-09	22-Sep-09	QA
S-ALL-Q-034	00	Anonymous Hotline Procedure	5-Aug-10	16-Sep-10	QA
S-ALL-Q-037	00	Data Review Process	9-Feb-11	9-Feb-11	QA
S-GB-Q-001	02	Employee Master Signature Log	10-Jan-06	19-Oct-09	QA
S-GB-Q-002	02	Training Record Files Maintained by the QAC	10-Jan-06	19-Oct-09	QA
S-GB-Q-003	01	Data Reduction, Validation, and Reporting	5-Jan-09	19-Oct-09	QA
S-GB-Q-004	02	Laboratory Notebooks and Logbooks	17-Jan-06	19-Oct-09	QA

SOP Number	Rev.	SOP Name	Date of Publication	Signature Date	Department
S-GB-Q-005	02	Precision and Accuracy Measurement and Evaluation	23-Mar-06	19-Oct-09	QA
S-GB-Q-006	02	Data Archiving	27-Apr-06	19-Oct-09	QA
S-GB-Q-007	03	Method of Syringe Technique	9-May-06	7-Dec-09	QA
S-GB-Q-008	03	Preventative, routine, and non-routine maintenance	9-May-06	19-Oct-09	QA
S-GB-Q-009	02	Common Laboratory Calculations and Statistical Evaluation of Data	11-May-06	19-Oct-09	QA
S-GB-Q-010	02	Estimation of Measurement Uncertainty	15-Feb-07	19-Oct-09	QA
S-GB-Q-011	00	Method Validation and Modification Studies	9-Oct-09	8-Jun-10	QA
S-GB-Q-012	00	Purchasing of Laboratory Supplies	3-Apr-06	15-Mar-10	QA
S-GB-Q-013	00	Receipt and Storage of Laboratory Supplies	21-Mar-06	15-Mar-10	QA
S-GB-Q-014	00	Control Charting and Trend Analysis	23-Jun-10	15-Sep-10	QA
S-ALL-S-001	02	Hazard Assessment	1-Jan-05	12-Mar-10	Safety
S-GB-S-001	03	Regulated Soil Handling (Green Bay Location)	5-Jan-06	18-Nov-10	Safety
S-GB-S-002	02	Control of Hazardous Energy Program - Lockout/Tagout	10-Jan-06	15-Mar-10	Safety
S-GB-S-004	02	Rescue Alert System Operation	16-Nov-05	15-Mar-10	Safety
S-GB-S-005	00	Waste Management Training Requirements	11-Feb-09	2-Apr-09	Safety
S-GB-S-006	00	Waste Handling and Management	29-Jul-05	15-Mar-10	Safety
S-GB-E-001	01	Use and Maintenance of the NANOpure Infinity Water Purification System	28-Mar-06	6-Oct-08	Equipment
S-GB-E-002	01	Operation of Waste Disposal Equipment	21-Sep-07	6-Oct-08	Equipment
S-GB-L-001	04	Biological Tissue, Plant, and Synthetic Material Preparation	20-Jan-05	2-Feb-11	Laboratory
S-GB-L-002	02	Small Rodent Handling and Homogenization	20-Jan-05	7-Jul-10	Laboratory
S-GB-L-003	03	The Determination of Lipids in Tissues, Fats, and Plants	20-Jan-05	15-Nov-10	Laboratory
S-GB-L-004	02	Determination of Percent Solids in Tissue Samples	3-Feb-05	26-May-09	Laboratory
S-GB-L-005	00	Reagent Water Quality	10-Jan-08	10-Jan-08	Laboratory

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

GREEN BAY LABORATORY

Accrediting Authority	Program Category	Accrediting Agency	Certification #	Expiration Date
Florida (NELAP)	Biological Tissue	Dept of Health, Bureau of Laboratories	E87948	6/30/2011
Florida (NELAP)	Hazardous Waste - Solid	Dept of Health, Bureau of Laboratories	E87948	6/30/2011
Florida (NELAP)	Waste Water	Dept of Health, Bureau of Laboratories	E87948	6/30/2011
Georgia	Waste Water -NELAP stipulation	Environmental Protection Division	E87948	6/30/2011
Georgia	Waste Water - NELAP stipulation	Environmental Protection Division	E87948	6/30/2011
Illinois (NELAP)	Hazardous Waste - Solid	Illinois EPA	200050	8/3/2011
Illinois (NELAP)	Waste Water	Illinois EPA	200050	8/3/2011
Kentucky	UST	Environmental and Public Protection Cabinet	82	9/17/2011
Louisiana (NELAP)	Hazardous Waste - Solid	Department of Environmental Quality	04168	6/30/2011
Louisiana (NELAP)	Waste Water	Department of Environmental Quality	04168	6/30/2011
Louisiana (NELAP)	Biological Tissue	Department of Environmental Quality	04168	6/30/2011
Minnesota	Hazardous Waste	Dept of Health	055-999-334	8/31/2011
Minnesota	Waste Water	Dept of Health	055-999-334	8/31/2011
Minnesota	UST	Department of Health	055-999-334	8/31/2011
New York (NELAP)	Solid - Hazardous Waste - NELAP	Dept of Health	11888	4/1/2011
New York (NELAP)	Waste Water - NELAP	Dept of Health	11888	4/1/2011
North Carolina	Waste Water	Dept of Environment, Health & Natural Resources	503	12/31/2011
North Dakota	Hazardous Waste	Dept of Health Chemistry Division	R-150	6/30/2011
North Dakota	Waste Water	Dept of Health Chemistry Division	R-150	6/30/2011
South Carolina	Hazardous Waste	Dept of Hlth & Environmental Control	83006001	8/31/2011
South Carolina	Waste Water	Dept of Hlth & Environmental Control	83006001	8/31/2011
US Dept of Agriculture	Foreign Soil Permit	Dept of Argiculture	S-76505	3/31/2011
Wisconsin	Drinking Water	Dept of Natural Resources	405132750	8/31/2011
Wisconsin	Drinking Water	Dept of Agriculture, Trade & Consumer Protection	105-444	12/31/2011
Wisconsin	Hazardous Waste	Dept of Natural Resources	405132750	8/31/2011
Wisconsin	Waste Water	Dept of Natural Resources	405132750	8/31/2011

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

Pace Analytical™ www.pacelabs.com

CHAIN-OF-CUSTODY / Analytical Request Document

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

Page: of

Section A Required Client Information:
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.

ITEM #	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS			
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										

ADDITIONAL COMMENTS

SAMPLER NAME AND SIGNATURE
PRINT Name of SAMPLER:
SIGNATURE of SAMPLER:
DATE Signed (MM/DD/YY):

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

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

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
2, 3, 7, 8-TCDD	1613B	Soil	8oz Glass	None	90/40 Days
2, 3, 7, 8-TCDD	1613B	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	90/40 Days
2, 3, 7, 8-TCDD	8290	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Acidity	SM2310B	Water	Plastic/Glass	≤6°C	14 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	≤6°C	14 Days
Alpha Emitting Radium Isotopes	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Anions by IC, including Br, Cl, F, NO_2, NO_3, SO_4	300.0/300.1/ SM4110B	Water	Plastic/Glass	≤6°C	Br, Cl, F, SO_4 (28 Days) NO_2, NO_3 (48 Hours)
Anions by IC, including Br, Cl, F, NO_2, NO_3, SO_4	300.0/9056	Soil	Plastic/Glass	≤6°C	Br, Cl, F, SO_4 (28 Days) NO_2, NO_3 (48 Hours)
Aromatic and Halogenated Volatiles	8021	Soil	5035 vial kit	See 5035 note*	14 days
Aromatic and Halogenated Volatiles	601/602/8021	Water	40mL vials	pH<2 HCl; ≤6°C; $Na_2S_2O_3$ if Cl present	14 Days
Acid Volatile Sulfide	Draft EPA 1629	Soil	8oz Glass	≤6°C	14 Days
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤6°C; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Soil	8oz Glass	≤6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals and Acids	8270	Tissue	Plastic/Glass	≤-10°C	1 Year if frozen
Base/Neutrals, Acids & Pesticides	525.1/525.2	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/30 Days
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤6°C	48 hours
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	14 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag	None	48 Hours
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Chloride	SM4500Cl/9250/	Water	Plastic/Glass	None	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
	9251/9252				
Chlorinated Herbicides	8151	Soil	8oz Glass Jar	≤6°C	7/40 Days
Chlorinated Herbicides	8151	Water	1L Amber Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Chlorinated Herbicides	515.1	Water	1L Amber Glass	≤6°C; $Na_2S_2O_3$ if Cl present	14/28 Days
Chorine, Residual	SM4500Cl	Water	Plastic/Glass	None	15 minutes
COD	SM5220C/ 410.3/410.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Color	SM2120B,C,E	Water	Plastic/Glass	≤6°C	48 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	6 Months
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN/9010/ 9012/335.4	Water		pH>12 NaOH; ≤6°C; ascorbic acid if Cl present	14 Days, 24 Hours if Sulfide present
Diesel Range Organics- TPH DRO	8015	Soil	8oz Glass Jar	≤6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Glass	≤6°C	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	Plastic/Glass	≤-10°C	1 Year if frozen
Diesel Range Organics (WI)	WI MOD DRO	Soil	2oz Glass Jar, MeCl	≤6°C	10/47 Days
Diesel Range Organics (WI)	WI MOD DRO	Water	1L Glass, HCl	≤6°C	7/40 Days
Dioxins & Furans	TO-9	Air	PUF	None	30/45 Days
EDB & DBCP	504.1/8011	Water	40mL vials	≤6°C; $Na_2S_2O_3$ if Cl present	14 Days
Explosives	8330/8332	Water	1L Glass	≤6°C	7/40 Days
Explosives	8330/8332	Soil	8oz Glass Jar	≤6°C	14/40 Days
Fecal Coliform	SM9222D	Water	100mL Plastic	≤6°C	6 Hours
Fecal Coliform	SM9222D	Soil	100mL Plastic	≤6°C	6 Hours
Ferrous Iron	SN3500Fe-D, Hach 8146	Water	Glass	None	Immediate
Flashpoint/Ignitability	1010/1030	Water	Plastic/Glass	None	28 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Soil	5035 vial kit	See 5035 note*	14 days
Gasoline Range Organics (WI)	WI MOD GRO	Water	40mL vials	pH<2 HCl,	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				≤6°C	
Gasoline Range Organics (WI)	WI MOD GRO	Soil	40mL 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
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤6°C	14/7 Days
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Heterotrophic Plate Count (MPC)	EPA 9215B	Water	100mL Plastic	≤6°C	24 Hours
Hexavalent Chromium	7196/218.6/ SM3500Cr	Water	Plastic/Glass	≤6°C	24 Hours
Hydrogen Halide & Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Lipid	Pace Lipid	Tissue	Plastic/Glass	≤-10°C	1 Year if frozen
Low Level Mercury	EPA 1631E	Water	Glass	BrCl	90 days (if preserved and oxidized)
Low Level Mercury	EPA 1631E	Soil	Glass	≤6°C	28 Days
Low Level Mercury	EPA 1631E	Tissue	Plastic/Glass	≤-10°C	28 Days if frozen
Mercury	7471	Soil	8oz Glass Jar	≤6°C	28 days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	≤-10°C	28 Days if frozen
Metals	7300/7303	Air	Filters	None	6 Months
Metals (and other ICP/ICPMS elements)	6010/6020	Soil	8oz Glass Jar	None	6 months
Metals (and other ICP/ICPMS elements)	6010/6020/200.7/ 200.8	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Metals (and other ICP/ICPMS elements)	6020	Tissue	Plastic/Glass	≤-10°C	6 Months if frozen
Methane, Ethane, Ethene	EPA Mod 8015	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag	None	48 Hours
Methanol, Ethanol	EPA 8015	Water	40mL vials	≤6°C	14 Days
Methanol, Ethanol	EPA 8015	Soil	2oz Glass	≤6°C	14 Days
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Nitrogen, Kjeldahl	SM4500-Norg; 351.1/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/ 352.1	Water	Plastic/Glass	≤6°C	48 Hours
Nitrogen, Nitrate & Nitrite	SM4500-NO3/ 353.2	Water	Plastic/Glass	pH<2 H_2SO_4;	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				≤6°C	
Nitrogen, Nitrite	SM4500-NO2/ 353.2	Water	Plastic/Glass	≤6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/ 351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag	None	48 Hours
Odor	SM2150B	Water	Glass	≤6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/ 9070	Water	Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Organochlorine Pesticides & PCBs	TO-4	Air	PUF	None	7/40 Days
Organochlorine Pesticides & PCBs	8081/8082/608	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Organochlorine Pesticides & PCBs	8081/8082	Soil	8oz Glass Jar	≤6°C	14/40 Days
Organochlorine Pesticides & PCBs	8081/8082	Tissue	Plastic/Glass	≤-10°C	1 Year if frozen/40 Days
Organophosphorous Pesticides	8141	Soil	8oz Glass Jar	≤6°C	14/40 Days
Organophosphorous Pesticides	8141	Water	1L Amber Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particulates	PM-10	Air	Filters	None	6 Months
Permanent Gases	EPA 3C	Air	Summa Canister	None	14 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag	None	48 Hours
pH	SM4500H+B/9040/ 9041/150.2	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	15 minutes
Phenol, Total	420.1/420.4/9065/ 9066	Water	Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; ≤6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤6°C	28 Days
Phosphorus, Total	EPA 365.4	Soil	Plastic/Glass	≤6°C	28 Days
Polynuclear Aromatic Hydrocarbons	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons	8270 SIM	Soil	8oz Glass Jar	≤6°C	14/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Polynuclear Aromatic Hydrocarbons	8270 SIM	Water	1L Glass	≤6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Polynuclear Aromatic Hydrocarbons	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 Radon Emanation Technique	903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤6°C	7 Days
Solids, Total (FOC)	ASTM D2974	Soil	Plastic/Glass	≤6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤6°C	7 Days
Solids, Total Suspended	SM2540D	Water	Plastic/Glass	≤6°C	7 Days
Solids, Total Volatile	SM2540E	Water	Plastic/Glass	≤6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤6°C	28 Days
Stationary Source Dioxins & Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	6 Months, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	6 Months, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	6 Months
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	6 Months
Sulfate	SM4500SO4/9036/ 9038/375.2/ASTMD516	Water	Plastic/Glass	≤6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants	SM5540C	Water	Plastic/Glass	≤6°C	48 Hours
Total Organic Carbon (TOC)	SM5310B,C,D/ 9060	Water	Glass	pH<2 H_2SO_4 or HCl; ≤6°C	28 Days
Total Organic Halogen (TOX)	SM5320/9020/ 9021	Water	Glass; no headspace	≤6°C	14 Days
Tritium	906.0	Water	Glass	pH<2 HNO_3	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤6°C	48 Hours
Uranium Radiochemical Method	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HNO_3	180 days
Volatiles	TO-14	Air	Summa Canister	None	30 Days
Volatiles	TO-14	Air	Tedlar Bag	None	48 Hours
Volatiles	TO-15	Air	Summa Canister	None	30 Days
Volatiles	8260	Soil	5035 vial kit	See 5035	14 days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				note*	
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤6°C; $Na_2S_2O_3$ if Cl present	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤6°C; $Na_2S_2O_3$ if Cl present	14 Days (7 unpreserved)
Volatiles	524.1/524.2	Water	40mL vials	pH<2 HCl; ≤6°C; $Na_2S_2O_3$ if Cl present	14 Days
Alaska DRO	AK102	Soil	8oz Glass	≤6°C	14/40 Days
Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤6°C	14/40 Days
Alaska RRO	AK103	Soil	8oz Glass	≤6°C	14/40 Days
Alaska GRO	AK101	Soil	5035 vial kit	See 5035 note*	14 Days
Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤6°C	14 Days

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

ATTACHMENT IX- NELAC

CERTIFICATE

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.

EFFECTIVE July 01, 2010 THROUGH June 30, 2011

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E87948-15-07/01/2010
Supersedes all previously issued certificates

Charlie Crist
Governor

FLORIDA DEPARTMENT OF HEALTH

Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-15, expiration date June 30, 2011. 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	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-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
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2010** **Expiration Date: 6/30/2011**

Charlie Crist
Governor

FLORIDA DEPARTMENT OF HEALTH

Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-15, expiration date June 30, 2011. 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 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
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-15, expiration date June 30, 2011. 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-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
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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Pace Analytical Services, Inc - Green Bay #1
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Green Bay, WI 54302

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005

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E87948
Pace Analytical Services, Inc - Green Bay #1
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Green Bay, WI 54302

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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
Carbazole	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Laboratory Scope of Accreditation

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Green Bay, WI 54302

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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
Conductivity	EPA 9050	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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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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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Mercury	EPA 1631	Metals	NELAP	5/30/2007
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
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	4/1/2005

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State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-15, expiration date June 30, 2011. 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 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 150.1	General Chemistry	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
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

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Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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 (20th/21st Ed.)	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	5/30/2007
Total organic carbon	SM 5310 C	General Chemistry	NELAP	5/30/2007
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-Dichloropropylene	EPA 624	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropylene	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
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	5/15/2009

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Green Bay, WI 54302

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Turbidity	SM 2130 B	General Chemistry	NELAP	4/1/2005
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

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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
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	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-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
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005

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FLORIDA DEPARTMENT OF HEALTH

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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: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	5/15/2009

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E87948
Pace Analytical Services, Inc - Green Bay #1
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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009

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Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-15, expiration date June 30, 2011. 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
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
Iodomethane (Methyl iodide)	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: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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 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
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Green Bay, WI 54302

Matrix: **Solid and Chemical Materials**

Analyte	Method/Tech	Category	Certification Type	Effective Date
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, 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
Thallium	EPA 6020	Metals	NELAP	4/1/2005
Tin	EPA 6010	Metals	NELAP	4/1/2005
Titanium	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
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-Dichloropropylene	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
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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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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Ana M. Viamonte Ros, M.D., M.P.H.
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

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2010** **Expiration Date: 6/30/2011**

Charlie Crist
Governor

FLORIDA DEPARTMENT OF HEALTH

Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87948-15, expiration date June 30, 2011. 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
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 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
n-Nitrosodi-n-propylamine	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: 7/1/2010** **Expiration Date: 6/30/2011**

Charlie Crist
Governor

FLORIDA DEPARTMENT OF HEALTH

Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-15, expiration date June 30, 2011. 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-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: 7/1/2010** **Expiration Date: 6/30/2011**

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-ALL-GB-I-015-REV.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-ALL-GB-I-015-REV.00
SOP TEMPLATE NUMBER:	SOT-ALL-I-015-Rev.00

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 5/26/09

Kate Grams, Laboratory Quality Manager — Date: 5/26/09

Chad Rusch, Department Manager — Date: 05/26/2009

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 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 water rinsing with distilled. 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 every 20 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 ALL-Q-013, "Support Equipment". Record the calibration in the thermometer calibration logbook and label the probe with the date of calibration.

13. Method Performance

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. 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.

14. Pollution Prevention and Waste Management

14.1. Procedures for handling waste generated during this analysis are addressed in ALL-S-002, Waste Handling.

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	1. Restructured document format to new corporate template. 2. 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

ATTACHMENT I
pH Logbook

Pace Analytical

pH (SW 846 9040B, SW 846 9045C, SM 4500-H+B)

Logbook #

Meter: Orion Model 720A (40WET3)	Meter Serial Number: 019247	
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.04 (12May2009)

Review/Date_______________________________

Pace Analytical Services, Inc.
1241 Bellevue Street
Green Bay, WI 54302

Phone: 920 469-2436
Fax: 920 469-8827

STANDARD OPERATING PROCEDURE

MEASUREMENT OF PERCENT MOISTURE IN SOILS AND SOLIDS

Reference Methods: ASTM D 2974-87

LOCAL SOP NUMBER:	S-GB-C-008-Rev.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-C-008-Rev.00
SOP TEMPLATE NUMBER:	SOT-ALL-L-004-rev.00

LOCAL APPROVAL

Nils Melberg, Laboratory General Manager — Date: 11/4/09

Kate Grams, Laboratory Quality Manager — Date: 11/2/09

Laura Buholca Department Manager — Date: 11/4/09

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

1.1 This is Standard Operating Procedure (SOP) describes procedures used to measure percent moisture of soils and solid samples based on ASTM D 2974-87 Standard Test Methods for moisture.

2. Scope and Application

2.1 This method is applicable to most soil and solid samples (including sludges) containing at least 0.1% moisture.

2.2 Percent moisture is typically used to correct results of inorganic and organic parameter analysis to dry weight basis.

2.3 This SOP was prepared for method-compliant use by all Pace Analytical environmental laboratories. Additional lab-specific requirements that may be mandated by individual states or clients are presented in the addendum.

2.4 This procedure is restricted to use by, or under the supervision of, analysts experienced with the used of laboratory balances, desiccators, and ovens. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3. Summary of Methods

3.1 A sample aliquot is weighed before and after heating to dryness at 103-105° C. The weight loss is calculated as % Moisture.

4. Interferences

4.1 Non-representative materials, e.g., leaves and sticks should be removed from the sample prior to measurement.

4.2 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.

5. Safety

5.1 **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. **DO NOT WEAR LATEX OR NITRILE GLOVES WHILE HANDLING HOT TRAYS.**

6. Definitions

Refer to Glossary section of the Pace Quality Manual.

7. Sample Collection, Preservation and Handling

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil/ solid	Wide mouth glass or plastic 4-oz container	N/A	4 ± 2°C	Analyze as soon as possible to minimize microbiological decomposition of organic solids

8. Equipment and Supplies

9.1 Instrumentation

Equipment	Vendor	Model / Version	Laboratory Identification	Description / Comments
Analytical Balance	Mettler Toledo	PB602-S	40BAL9	Electronic with RS-232 output, capable of weighing 0.01g
Drying Oven	Precision Scientific	STG80	Oven #5	Capable of maintaining temperature at 103-105°C
Computer for LimsLink™				Automated sample weight upload into LIMS
LimsLink™ Software				

9.2 Supplies

Supplies	Vendor	Model / Version	Description / Comments
Desiccators	Fisher	Fisher p/n 08-644	Labconco Model
Indicating Desiccant	Fisher	Fisher p/n 07-578-4B	
Non-indicating Desiccant	Fisher	Fisher p/n 07-577-3B	
Disposable Aluminum Weighing Dishes	Fisher	Fisher p/n 08-732	
Spoonula Lab Spoons	Fisher	Fisher p/n 14-375-10	
Trays, plastic or metal	NA	NA	

9. Reagents and Standards

Not applicable

10. Calibration

10.1 Analytical Balance Calibration

10.1.1 Annual Calibration - The balance must be calibrated at least annually by an outside agency and checked daily before each use using Class 1 or 2 weights. Refer to Pace SOP, S-ALL-Q-013 *Support Equipment*.

10.2 Daily Calibration Check

10.2.1 Clean the balance and surrounding area prior to starting the daily calibration check.

10.2.2 Check the sight level on the balance. If it needs adjusting, level the balance.

10.2.3 The weight set ID indicated in the logbook is used as the primary set. If an alternate weight set ID is used, that ID must be recorded in the comment section of the balance calibration logbook for that day.

10.2.4 Tare the balance before weighing the NIST certified weights.

10.2.5 Use forceps or other means to lift each weight (Do not touch the weights with fingertips as the residue may artificially adjust the true value of the weights). Record the date of the calibration check, the true value of the weight, and the actual measured weight in the logbook. Repeat this procedure for the other certified weights. If calibration weights differ from the certified weights by more than specified in the balance calibration logbook, corrective action must be taken (see 11.3).

10.3 Corrective Action

10.3.1 Clean the balance and balance pan. Check the sight level on the balance and adjust if necessary. Re-tare and re-weigh all the certified weights.

10.3.2 The internal calibration function (if available) of the balance may be used as a means of corrective action.

10.3.3 Utilize the internal calibration function and diagnostics. Refer to instrument manual.

10.3.4 Contact the QA office for assistance if the balance does not meet the calibration tolerances.

10.3.5 If the above action does not correct the problem, the balance should be taken out of service and appropriately labeled to avoid improper usage. A service technician should be contacted.

10.3.6 Record any corrective action. Initial and date all entries in the logbook

11. Procedure

11.1 Locate the samples to be analyzed, place on a cart and allow samples to warm to ambient temperature prior to processing.

11.2 Determine the number of aluminum weighing pans required for the number of samples to be analyzed plus one for a duplicate.

11.3 The samples scheduled for analysis are batched in the PMST QUEUE in groups of 20. The QC batch will also include a duplicate for one of the project soil samples.

11.4 After batching samples in EPIC Pro print the work list. This creates an xxx.wld for LimsLink™ to recognize

11.5 Open LimsLink™ to start a new worksheet, click on the running man icon. Select New. Name the worksheet by the QC batch number (Batch #). Click OK to initialize the method. To start the EPIC Pro upload, click on the ▶ green triangle (Play, Start) icon. Select sample info from Epic. Open Click OK.

11.6 Next, click on the worksheet header and select get file data. Scroll down and highlight the xxx.wld file that will be transferred. It will be designated the same number as the batch number. Click OK.

11.7 After file data has been loaded, click on the ■ red square icon to reset instrument list.

11.8 Verify balance calibration - refer to section 11.2 for balance check procedures and corrective actions. Refer to the balance logbook for the acceptance criteria for the designated balance.

11.9 For each sample, label and record the tare weight (to the nearest 0.01 g) for an aluminum-weighing dish in the LimsLink™ worksheet (see **attachment I** – Acquiring weights and Auto posting).

11.10 Using a clean spoonula lab spoon, stir the material in the sample container. Transfer at least 10g of the remaining sample to the tared weighing dish.

11.11 Weigh the sample and dish, recording to the nearest 0.01g (**see attachment I** for recording the weight in LimsLink™).

11.12 Place the samples to dry in the oven overnight at 103-105°C. Check that the oven temperature is recorded on the LimsLink™ benchsheet each morning and is within required specifications before placing samples into oven as per Pace SOP S-ALL-Q-013 *Support Equipment.*

11.13 Overnight is a period of time $\geq$ 8 hours.

11.14 Remove the sample from the oven place in a desiccator to cool. The desiccator should contain mostly non-indicating desiccant with enough indicating desiccant to demonstrate that the desiccant is still active.

11.15 After the sample has cooled, weigh the dried residue to the nearest 0.01g (see **Attachment I** for LimsLink™ posting procedures). If the sample has been oven dried for at least 8 hours, proceed to section *12.17.* If dried less than 8 hours, proceed to the next step.

11.16 Return the samples to the oven for one additional hour. At the end of the hour, remove the samples once again; allow them to cool to room temperature and reweigh. If the weight is within 0.01g or 0.1% of the previous weight, record the weight and proceed to *12.17*. If the weight has changed by more than 0.01g or 0.1%, repeat step *12.16* until a constant weight (<0.01g or 0.1% change) is achieved.

11.17 % Moisture is calculated and in the LimsLink worksheet using the following equation. If an automated system is used to perform this calculation, select a few samples randomly and verify that the answer is being calculated correctly (see **Attachment I** for LimsLink™ related calculations).

$$\% \text{ Moisture} = (W_w - W_d)*100\%/W_w$$

Where: W_w = Wet weight of the sample (Dish + sample weight before drying – dish tare weight)
W_d = Dry weight of the sample (Dish + sample weight after drying – dish tare weight)

12. Quality Control

13.1 **Duplicate Sample** – Measure one duplicate sample with each batch of 20 samples. The Relative Percent Difference (RPD) for duplicate results must be ≤ 10%. If this is not met the entire batch must be re-analyzed. Calculate the RPD as follows:

$$\%RPD = (S1-S2)*100\%/((S1+S2)/2)$$

Where: S1 = %Moisture for Sample
S2 = %Moisture for Sample Duplicate

13.2 **Documentation of Equipment Operation and Calibration** – The balance calibration check and oven temperature should be recorded on the lab datasheet for each sample batch. In addition, the oven temperature should be read each day it contains active samples and the temperature recorded in the oven log. If balance checks and oven temperatures are not within acceptable limits, all effected samples must be re-analyzed.

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) described in S-ALL-Q-020, *Orientation and Training Procedures.* All results must be ± 5% of the mean to qualify the analyst for reporting sample results. Results of DOC studies for each analyst shall be retained in the lab quality assurance office. Each analyst must successfully repeat this study annually to maintain qualification.

14. Pollution Prevention and Waste Management

14.1 Procedures for handling waste generated during this analysis are addressed in ALL-S-002, *Waste Handling,* and the individual laboratory SOP addenda for waste management

14.2 The Pace Chemical Hygiene Plan/Safety Manual contains additional information on pollution prevention.

15. References

15.1 ASTM D 2974-87 "Standard Test Methods for moisture, Ash and Organic Matter of Peat and Other Organic Soils", American Society of Testing and Materials, March/April 1993.

15.2 EPA Contract Laboratory Program SOW for Inorganic Analysis Doc. ILM 1.03 March 1990.

15.3 ASTM D 2216-98 "Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass", American Society of Testing and Materials, 1998.

16. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

Attachment I

Acquiring Weights and Auto-posting Through LimsLink™

1. Acquiring the Weights and Auto-posting

1.1. In LimsLink™, click on the ▶ green triangle (Play, Start) icon and highlight **Tare Mass**. Click **OK.**

1.2. Number the weigh pans in the order of the work list and place them on a tray. Tare scale. Weigh the empty pans in order of the work list, pressing the button between each sample. Press red button to transfer weights in LimsLink™ worksheet.

1.3. Click on the ■ red square icon to reset instrument list.

1.4. Click on the ▶ green triangle (Play, Start) icon and highlight **Wet** Mass. Click **OK**.

1.5. Tare scale. Weigh the wet sample into the pans in order of the work list, pressing the button between each sample. Press red button to transfer weights in LimsLink™ worksheet

1.6. Click on the ■ red square icon to reset instrument list.

1.7. Close work sheet.

1.8. After the samples are dry, remove from oven and cool in desiccator. Return to LimsLink™, click on the running man icon. Highlight the batch # and return to the worksheet.

1.9. ▶ green triangle (Play, Start) icon and highlight **Dry Mass**. Click **OK**.

1.10. Tare scale. Weigh the dry samples in order of the work list, pressing the button between each sample. Finally, press red button to transfer weights in LimsLink™ worksheet.

1.11. After placing samples back into oven and cooling, the constant weight weighing is ready to proceed.

1.12. Go back into LimsLink™, click on ▶ green triangle (Play, Start) icon and highlight **Constant Weight**. Click **OK.**

1.13. Tare scale. Weigh the constant weight dry samples in order of the work list, pressing the button between each sample. Finally, press red button to transfer weights in LimsLink™ worksheet.

1.14. % Moisture is calculated by the LimsLink worksheet using the following equation. Select a few samples randomly and verify that the answer is being calculated correctly:
% Moisture = $(W_w - W_d)*100/W_w$

Where: W_w = Wet weight of the sample (Dish + sample weight before drying – dish tare weight)
W_d = Dry weight of the sample (Dish + sample weight after drying – dish tare weight)

1.15. Highlight data that is going to be reported.

1.16. Select **Options** header and click on **Report 1**.

1.17. Wait for the **parser** and **auto-posting** programs to run. Wait for **parser 2** and **auto-posting** programs to run. **Auto-posting** is run 5 minutes after the **parser 2** runs. **Note**: It normally requires about 10 minutes to complete the data transfer.

1.18. Generate the validation list. Perform the final review.

2. Creating the Runlog

2.1 In LimsLink™, highlight data that will go into the **Runlog**.

2.2 In LimsLink™, click on **Printer** button. Select **Runlog** and click on **Report**.

2.3 Default path: G:\metals\prep\runlog\%moist.txt. Click on **OK**. Click on **Yes** to overwrite the system files.

2.4 After a **%moist.txt** file has been created by LimsLink™, the data will be ready to be transferred to the **Runlog**.

2.5 Open the prep logbook.

2.6 Click on the **percent moisture** icon.

2.7 The **Runlog** sheet will come up:

- Enter **time in**.
- Enter **time out**.
- Enter **oven temperature**.
- **Date**: This is the date on which the **Runlog** page is created. It should be the same as the date the samples are run.
- Click on **Import Samples** button. Data will transfer to the large box in the middle of the screen.
- Edit data and add comments as needed.
- **Signature**: Pick initials from the drop down box.

2.8 If you don't enter your initials, and then **exit** the screen, the data may still be edited. To do this, re-click on **instrument icon** and the data will be there for editing.

2.9 Once you have entered your initials, you will be able to review data by clicking on the **Review** icon. You can see your data, but it cannot be edited. If errors were made, see the QAO.

17. Revisions

Document Number	Reason for Change	Date
SOT-ALL-L-004-rev.00	New Template Document	December 19, 2006
S-GB-C-008-rev.0	Conversion from template to local SOP	March 20, 2008
S-GB-C-008-Rev.01	Updated Signature Page to Periodic Review Removed Responsibilities and Distribution Section Renumbered Document Section 11.12: Incorporated language to record temperature on LimsLink™ benchsheet.	28Oct2009

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STANDARD OPERATING PROCEDURE
FLASH POINT

(PENSKY-MARTENS CLOSED CUP METHOD FOR IGNITABILITY)
(SW 846 1010A)

SOP NUMBER:	S-GB-I-002-REV.05
EFFECTIVE DATE:	Date of final signature
SUPERSEDES:	S-GB-I-002-REV.04

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 11/18/10

Kate Grams, Laboratory Quality Manager — Date: 11/8/10

Chad Rusch, Department Manager — Date: 11/15/2010

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

This method is used to determine the flash point of liquids, surface film forming liquids, and liquids containing non-filterable, suspended solids and solids as delineated in SW 846 1010A.

2. SCOPE AND APPLICATION

This method determines the flashpoint of liquids and soils within the range of 73°-210°F with a detection limit of 73 °F and a sensitivity of 1°F.

3. SUMMARY OF METHOD

The sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the sample cup at regular intervals. The flash point is defined as the lowest temperature at which the flame ignites the vapor above the sample.

4. INTERFERENCES

Not Applicable

5. SAFETY

5.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

5.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

5.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

6. DEFINITIONS

Definitions can be found in Section 10 of the most recent version of the Pace Analytical Services, Inc. Quality Manual.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in plastic or glass bottles and stored at 4 °C±2°C. The maximum holding time is 28 days.

8. EQUIPMENT AND SUPPLIES

8.1 Pensky-Martens Closed Flash Tester

8.2 Thermometer, 20º to 230ºF

8.3 Hurst motor, P/N: 3204-026

8.4 Gas Supply

8.5 Matches or lighter

9. REAGENTS AND STANDARDS

9.1 p-Xylene- Store at room temperature. The manufacturer specifies expiration date, or 5 years from received date whichever comes first.

9.2 Performance evaluation sample. The PE sample is analyzed on a twice yearly basis to cover the requirements of the method.

10. CALIBRATION

Not Applicable

11. PROCEDURE

11.1 PREPARATION

11.1.1 Bring sample to room temperature (73ºF).

11.1.2 Place the Precision Scientific Pensky-Martens Flash Point tester into the hood.

11.1.3 Hook the gas line to the apparatus.

11.1.4 Thoroughly clean and dry all parts of the cup.

11.1.5 Fill the cup to the interior line with the sample to be tested. Place the lid on the cup and put it into the apparatus.

11.1.6 Supply the heat at a slow, steady rate.

11.1.7 Light the flame and adjust it to a length of 4 mm.

11.1.8 Stir the sample continuously at 90 – 120 RPM with an automated stirrer.

11.2 ANALYSIS

11.2.1 Begin applying the test flame at room temperature (73°F).

11.2.2 Apply the test flame by operating the mechanism on the cover of the cup. Allow the flame to be lowered into the vapor space of the cup in 0.5 seconds, left in the lowered position for 1 second and then quickly raised to its high position. Do not stir the sample while applying the test flame.

11.2.3 Continue applying the test flame at temperature readings in multiples of 2°F. Example: Begin at 73°F, then 75°F, 77°F, etc. For aqueous and solid samples continue until a maximum reading of 210°F is reached, then stop and report the flashpoint value as >210° F. If sample flashes at <160°F, a duplicate of the sample is analyzed with the first of the two flashpoints being reported.

11.2.4 Record the flash point as the temperature read on the thermometer at the time the test flame causes a distinct flash in the interior of the cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at application. The actual flash will have occurred when a large flame propagates itself over the surface of the sample. Apply thermometer correction if necessary.

11.2.5 For samples containing large amounts of solid material, the test flame may be applied at intervals of 3°F instead of 2°F. Example: 73°F, 76°F, etc.

11.3 CALCULATIONS

11.3.1 All flashpoint temperatures are corrected for barometric pressure using the following procedure:

11.3.2 Obtain the Ambient Atmospheric Pressure in inches of mercury at the time of analysis from the local weather bureau website. The Ambient Atmospheric Pressure is entered into spreadsheet which calculates the conversion to mm Hg and the flashpoint correction, which is obtained by multiplying this number by the correction factor (CF) of 0.98. The 0.98 Correction factor is determined by using the following equation:

[(760 – (Altitude in feet * 0.026)] / 760 = Correction Factor

Note: Pressure drops by 26 millimeters (mm, about 1 inch) for every 1000 feet above sea level. This is why the altitude is multiplied by 0.026.

11.3.3 Convert the altitude corrected inches of mercury to altitude corrected millimeters of mercury by multiplying by 25.4.

11.3.4 Correct the observed flashpoint to barometric pressure corrected using the following equation:

[(760 - Corrected mm of Hg) * 0.06] + Observed Flashpoint = Barometric Pressure Corrected Flashpoint (°F).

12. QUALITY CONTROL

12.1 Laboratory Control Sample (LCS)

12.1.1 Laboratory Control Sample (LCS) is analyzed prior to analyzing any samples. p-Xylene is used as the LCS. The flash point of p-Xylene is 81.5°F after barometric correction. The acceptance criteria for the LCS are ± 1.5°F.

12.2 Duplicate Analysis

12.2.1 Samples that flash at <160° F have a duplicate performed. Duplicate acceptance criteria is 20% RPD.

12.2.2 If no sample flashes at <160° F in a batch of 20 samples or less, a duplicate must be performed at a 5% rate.

12.3 Thermometer calibration

12.3.1 Calibrate the thermometer used in this procedure on an annual basis. See SOP S-ALL-Q-013, *Support Equipment*. Document the calibration and label the thermometer with date, initials and correction factor, if any.

13. METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

14.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

14.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

15. REFERENCES

15.1 ASTM D93-99b, Standard Test Methods for Flash Point by Pensky-Martens Closed Tester, 09/1999.

15.2 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Method 1010A.

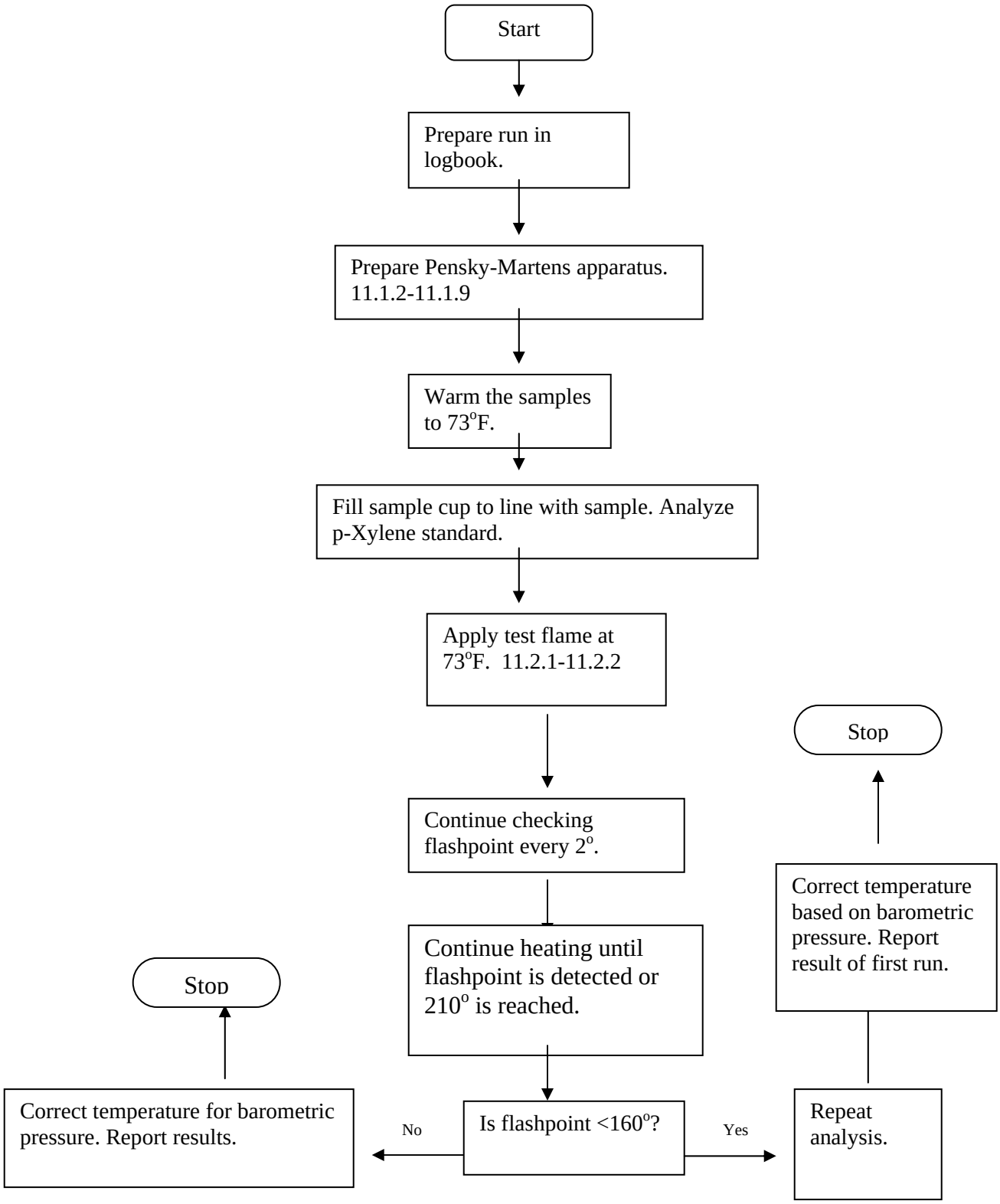
16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ETC.

Flow Chart – Attachment I

17. REVISIONS

Document Number	Reason for Change	Date
GB-I-002-Rev.0	Reformatted SOP from En Chem to Pace Analytical format.	27January2005
GB-I-002-Rev.1	Updated hold time Updated reference	18July2005
S-GB-I-002-Rev.2	Updated signature page Updated Sections 5, 6 14, and 15 with current information. Updated Section 12.2.3 with duplicate reporting requirement.	05Oct2007
S-GB-I-002-REV.03	Updated signature page. Deleted Section 7. Renumbered document. Section 7 – Stated soil samples can be collected in plastic containers. Section 11.3.2 – Section updated. Section 11.3.5 – Section deleted. Section 13 – Updated SOP references Flow Chart was updated	02Dec2008
S-GB-I-002-REV.04	Updated Method information to SW846 1010A.	20May2009
S-GB-I-002-Rev.05	Section 11.1.8: Added to stir samples continuously at 90-120 RPM. Section 11.3.2: Added calculation to UN-correct the barometric pressure from an aneroid barometer.	08Nov2010

FLOW CHART I


Start
Prepare run in logbook.
Prepare Pensky-Martens apparatus. 11.1.2-11.1.9
Warm the samples to 73°F.
Fill sample cup to line with sample. Analyze p-Xylene standard.
Apply test flame at 73°F. 11.2.1-11.2.2
Continue checking flashpoint every 2°.
Continue heating until flashpoint is detected or 210° is reached.
Is flashpoint <160°?
No
Yes
Correct temperature for barometric pressure. Report results.
Stop
Repeat analysis.
Correct temperature based on barometric pressure. Report result of first run.
Stop

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STANDARD OPERATING PROCEDURE

Free Liquids (Paint Filter)

Reference Methods: EPA Manual SW 846, 3rd Edition, Method 9095A

SOP NUMBER:	S-GB-I-013-REV.02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-013-REV.01

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 3/11/10

Kate Grams, Laboratory Quality Manager — Date: 3/11/2010

Chad Rusch, Department Manager — Date: 03/10/2010

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

This method is used to determine the presence of free liquids in a representative sample of solid material or waste according to EPA Method SW-846 9095A.

2. Scope and Application

This method is used to determine the presence of free liquids in a representative sample of solid material or waste.

3. Summary of Method

A predetermined amount of material placed in a paint filter. If any portion of the material passes through and drops from the filter within the 5-minute test period, the material is deemed to contain free liquids.

4. Interferences

Temperature can affect test results if the test is performed below the freezing point of any liquid in the sample. Tests must be performed above the freezing point and can, but are not required to, exceed room temperature of 25° C.

5. Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training

6. Definitions

Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation and Handling

See Table C for a summary.

8. Equipment and Supplies

See Table A and B for summary.

9. Reagents and Standards

Not Applicable.

10. Calibration

Balance calibration is verified daily to ensure accuracy of measurement. See the latest revision of SOP S-ALL-Q-013 *Support Equipment* for specifics on balance calibration.

11. Procedure

11.1 Place paint filter on top of 250-ml beaker. Place beaker on balance and tare the balance to zero grams.

11.2 Measure into the filter 100 grams of sample.

11.3 Allow sample to drain for 5 minutes into beaker.

11.4 If any portion of the test material collects in the beaker within the 5-minute collection period, the material is deemed to contain free liquids. This is reported qualitatively as **FAIL**. If no liquid passes through the filter, then material is deemed to contain no free liquids. This is reported qualitatively as **PASS**.

12. Quality Control

12.1 A sample duplicate will be performed every 20 samples or whichever is greater.

12.2 See Table D for a summary.

13. Method Performance

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per the latest revision of S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.2.1.1 To verify the analyst can perform this test, the trainer will make up four different matrix samples using sand and water for the analyst to perform the paint filter test on. There will be 2 samples that “pass” and 2 that “fail”.

14. Pollution Prevention and Waste Management

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of ALL-S-002, *Waste Handling.*

15. References

EPA Manual SW 846, 3rd Edition, Method 9095A.

16. Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

16.1 Table of Equipment – Table A

16.2 Table of Supplies – Table B

16.3 Table of Sample Collection, Preservation, Shipment, and Storage – Table C

16.4 Table of Quality Control – Table D

16.5 Bench Sheet – Attachment I

16.6 Flowchart – Attachment II

17. Revisions

Document Number	Reason for Change	Date
GB-I-013-rev.0	Reformatted SOP body to Pace Analytical format	13July2005
S-GB-I-013-Rev.01	Updated signature page. Section 1 – Added SW-846 reference Section 6 – Updated definition source Section 7 – Removed Responsibilities and Distribution Section. Renumber Section. Section 10 – Added current SOP reference Section 13 – Updated with current information Section 14 – Updated with current Information Attachment I – Update benchsheet.	08July2008
S-GB-I-013.Rev.02	Updated signature page. Attachment I – Update benchsheet.	10March2010

Table A
EQUIPMENT

Equipment	Manufacturer	Model(s)
Balance	Mettler	PJ400 (or equivalent)

Table B
SUPPLIES

Supplies	Vendor	Catalog #
Conical paint filter Mesh 60	Shop.com	
250-ml beaker		

Table C
SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

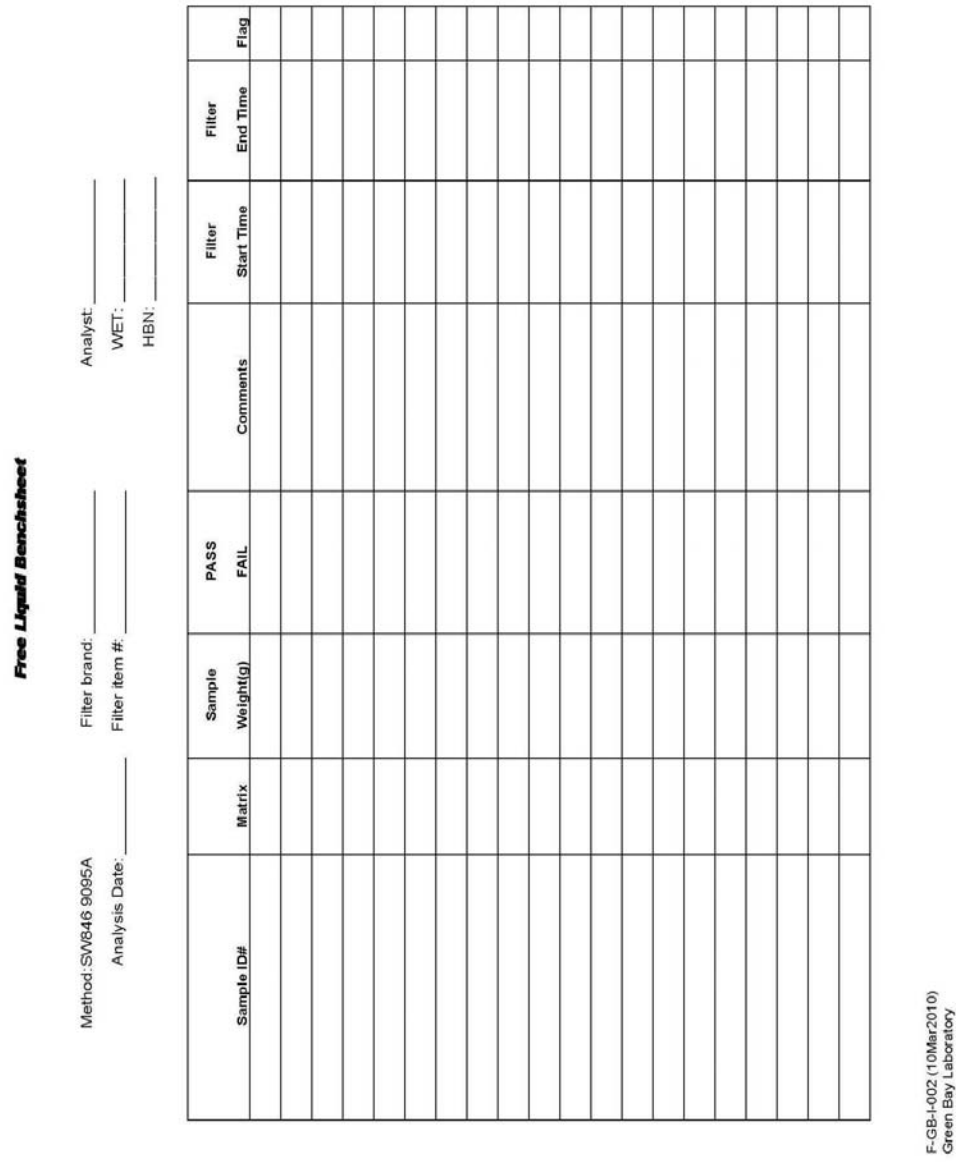
Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Solid Waste	EPA SW846 9095A	Clean Plastic or glass containers	Cool	On ice 4+/- 2° Celsius	Room Temperature

Table D
QUALITY CONTROL

Parameter	Frequency SW846	Frequency EPA citation
Sample Duplicate	1 per batch, up to 20 samples	-

ATTACHMENT I

Free Liquid Bench Sheet



Free Liquid Benchsheet

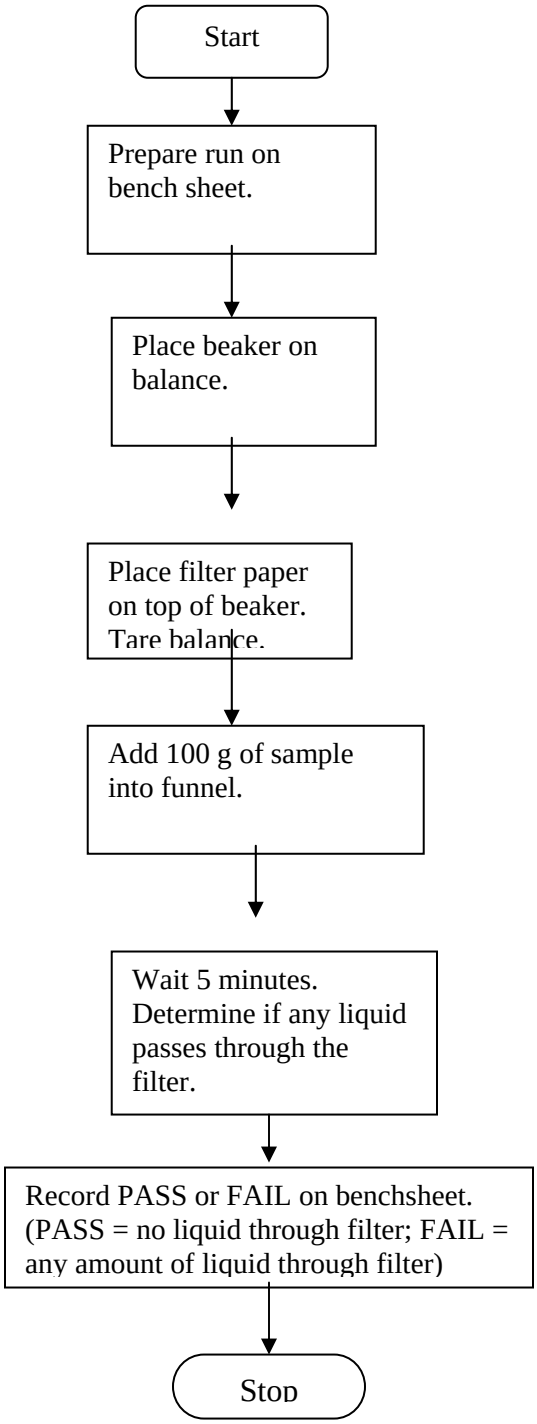
Method:SW846 9095A
Analysis Date:_____
Filter brand:_____
Filter item #:_____
Analyst:_____
WET:_____
HBN:_____

Sample ID#	Matrix	Sample Weight(g)	PASS FAIL	Comments	Filter Start Time	Filter End Time	Flag

F-GB-I-002 (10Mar2010)
Green Bay Laboratory

ATTACHMENT II

FLOWCHART



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STANDARD OPERATING PROCEDURE

The Determination of Total Organic Carbon Using the Walkley-Black Procedure

Reference Methods: Methods of Soil Analysis, Walkley-Black

SOP NUMBER:	S-GB-I-037-Rev.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-037-Rev.02

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 1/07/11

Kate Grams, Laboratory Quality Manager — Date: 12/20/10

Chad Rusch, Department Manager — Date: 12/20/2010

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 Standard Operating Procedure (SOP) is to describe the method used to determine the concentration of Total Organic Carbon (TOC) in soil samples compliant with the Walkley-Black procedure.

2. Scope and Application

This SOP pertains to sediments, soils and sludges. This method is based on Methods of Soil Analysis, Walkley-Black. The applicable range is 400 to 32,000 mg/kg.

3. Summary of Method

An aliquot of a dried and homogenized solid sample is put into a COD vial and caped. The sealed tubes are heated in a hot block at 150°C. After two hours, the tubes are removed, cooled and measured spectrophotometrically at 620 nm.

4. Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Chloride and iron give positive interference. Mercuric sulfate in the digestion tubes complexes the chlorides.

5. Safety

5.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

5.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management.*

5.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

6. Definitions

6.1 Total Organic Carbon (TOC): All carbon atoms covalently bonded in organic molecules. To measure TOC, all the inorganic carbon is removed by the addition of acid; the result is that only organic carbon is left.

6.2 Additional definitions can be found in Section 10 of the Pace Analytical Services, Inc. Quality Manual.

7. Sample Collection, Preservation and Handling

Table 1. SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Matrix	Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Solid	Walkley Black	Clean 4oz glass containers	None 28 days	On ice 4+/- 2° Celsius	4^{0}C

8. Reagents and Standards

8.1 Reagents

8.1.1 15,000mg/L COD Vials

8.1.2 Reagent Water – Deionized water (DI water)

8.2 Standards

8.2.1 Potassium Hydrogen Phthalate ($KHC_8H_4O_4$) – Neat, dry at 105°C for 1 hour.

8.1.2.1 One primary source for calibration and CCVs.

8.1.2.2 Secondary source for ICV and spikes.

8.2.2 Laboratory Control Spike/Laboratory Control Spike Duplicate – Potassium Hydrogen Phthalate at a concentration of 400mg/L (calculates out to16,000 mg/kg assuming 0.05g to 2mL).

8.2.3 4,000mg/L TOC standard from 0.85g of Neat Potassium Hydrogen Phthalate to 100mL with DI water.

8.2.4 800mg/L TOC standard from 2mL of 4,000mg/L TOC standard to 10mL with DI water.

8.2.5 Calibration Curve: Made up of the following six standards, 10, 20, 50, 200, 400, 800 mg/L.

9. Equipment and Supplies

Table 2. EQUIPMENT

Equipment	Manufacturer*	Model(s)*
Top loading Balance	OHAUS	AP 110S
Spectrophotometer	Hach	DR2000
COD reactor block	Hach	45600
Adjustable pipettor	Finnpipette	
Adjustable pipettor	Chemisphere	02111245
Sample cells	Hach	Cat# 13537-02
Drying Oven (103-105°C)	Precision Scientific Group	17
Mortar and Pestle	Coors	60316, 60317
Vented Hood	Hamilton or Equivalent	
100 mL volumetric flasks	Pyrex	5660 100
1000 mL volumetric flask	Pyrex	5640 1L
Wire racks	Fisher	Cat# 14802

* Or equivalent

Table 3. SUPPLIES

Supplies	Manufacturer*	Vendor*	Catalog #*
COD digestion vials	Chemetrics	Fisher	K-7370S
Latex gloves	Diamond Grip	Fisher	11-462-67D
Thermal gloves	Fisher	Fisher	19-013-541
Finntip 5mL tips	Thermolabsystems	VWR	53515-886
Eppendorf 1000 uL Pipette tips	Eppendorf	Fisher	21-372

10. Calibration and Standardization

10.1 Prepare a calibration curve using a minimum of five standards and a blank: see standards. Add 2.0 mL of each curve standard to a digestion tube and digest per section 11.

10.2 Turn on Spectrophotometer as per instrument manual and set to 620 nm. Let warm up at least 20 minutes.

10.3 Wipe the outside of each vial with tissue paper to remove any fingerprints. Place Level 0 calibration standard into the spectrophotometer and zero the instrument.

10.4 Place the standard cells into the spectrophotometer and record standard absorbance at 620 nm into the excel spreadsheet.

10.5 Plot absorbance versus concentration using the WB Spec. Excel spreadsheet. The data is stored under R:/WetChem/Data/WB/year/month/date analyzed.xls. Note: The zero point is included in the regression of the curve.

10.6 A linear regression analysis of the absorbance values against the corresponding concentrations must yield a correlation coefficient of >0.995. The y-intercept must be less than the Pace Reporting Limit.

10.6.1 A new calibration curve should be prepared yearly at minimum or whenever the continuing calibration standard does not pass control criteria. Create ICV, ICB, and RL standards. Run and record as in section 11. The ICV, ICB, and RL standards must be run immediately following creation of a new curve.

11. Procedure

11.1 Sample Preparation.

11.1.1 Physical Preparation

11.1.1.1 Air-dry and grind to a size that the sample will pass through a 0.5-mm sieve.

11.1.1.2 Mix the sample thoroughly before selecting a portion for analysis.

11.1.1.3 Discard any foreign objects such as sticks, leaves, and rocks.

NOTE: Document all sample sizes, standards and reagents used in the digestion on the analytical benchsheet.

11.2 Analytical

11.2.1 Label Chemetrics COD digestion vials

11.2.2 Unseal the vials and carefully weigh out 0.05 g using a calibrated balance. Place the 0.05 g of soil into a vial. Pipette 1.95 mLs of Nanopure water into a vial such that it forms a layer on top of the reagents contained in the vial.

11.2.3 Prepare ICV, ICB, CCV, CCB, MB, LCS, MS, and MSD. See Section 12 for control limits and frequency.

11.2.4 Carefully seal the vial. During digestion, the reagents and sample are raised to a point just below boiling. Improperly sealed vials may leak or break.

11.2.5 Thoroughly mix the contents of the sealed vial by shaking. CAUTION: The vial will get very hot during mixing. It is recommended that vials be mixed either in racks or with the use of insulated gloves. Eye protection MUST be worn.

11.2.6 Place the digestion vials in the reactor block at 150°C ± 2°C for 2 hours.

11.2.7 Cool about ½ hours. Then invert vials to mix.

11.2.8 Cool to room temperature. Allow any suspended precipitate to settle.

11.2.9 Turn on Spectrophotometer as per instrument manual and set to 620 nm. Let warm up at least 20 minutes.

11.3 Wipe the outside of each vial with tissue paper to remove any fingerprints. Place ICB into the spectrophotometer and zero the instrument. Then, place the sample vials into the spectrophotometer and record sample and QC absorbance at 620 nm into the excel spreadsheet.

11.3.1 Using the excel file and calibration equation, calculate the sample results in mg/L TOC, and report the final results in LIMS.

12. Calculations

12.1 $$\frac{\text{(mg/L TOC from the curve) X (Final Volume (mL))}}{\text{(Weight in (g))}} = \text{TOC (mg/Kg)}$$

12.2 TOC (%) = (concentration in mg/kg / 10,000)

12.3 Accuracy:

A laboratory control spike / laboratory control spike duplicate is analyzed for each analytical batch of 20 or fewer samples.

$$\text{LCS, \% Recovery} = \frac{\text{TOC, mg/kg}}{\text{True Value mg/Kg}} \times 100$$

12.4 Precision:

The precision is calculated based on the recovery of the sample / sample duplicate result. A sample duplicate is performed at a frequency of 10% or one per batch which ever is more frequent and must meet laboratory specific limits for precision.

Relative percent difference (RPD) calculation:

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

S = Sample Value
SD = Sample Duplicate Value

13. Quality Control:

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 **Initial Calibration Verification (ICV)**

13.2.1 The ICV must be analyzed immediately after calibration.

13.2.2 Concentration must be within ± 10% of the true value. The true value is 400 mg/L. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified.

13.2.3 The lot number of the KHP used to make the ICV must be different from that of the calibration curve standards.

13.3 **Continuing Calibration Verification (CCV)**

13.3.1 The CCV is analyzed after every 10 samples.

13.3.2 Concentration must be within ± 10% of the true value. The true value is 400 mg/L. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified. If the reset CCV fails recalibrate and reanalyze all samples back to the last acceptable CCV.

13.3.2.1 If the CCV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.4 **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)**

13.4.1 The ICB must be analyzed after the ICV. The CCB must be analyzed after the CCV.

13.4.2 The value must be < PRL. 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.

13.4.2.1 If the sample concentration is greater than ten times the concentration in the ICB or CCB, the samples do not need to be reanalyzed.

13.4.2.2 If the samples are non-detections and the ICB or CCB fails high, the samples do not need to be reanalyzed.

13.5 **Reporting Limit Verification Standard (RLVS)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.6 **Method Blank (MB)**

13.6.1 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.

13.6.2 The absolute value must be < PRL.

13.6.2.1 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

13.6.2.2 Method blank criteria is evaluated and flagged with appropriate data qualifiers to the MDL when MDL reporting is required.

13.6.2.3 Qualify data with the appropriate data qualifier if sample results are less than 10 times the absolute value detected in the blank and are not a non-detection.

13.6.2.4 Additionally, method blank acceptance may be based on project specific criteria or regulatory limits .

13.7 **Laboratory Control Sample (LCS)**

13.7.1 The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples. A Laboratory Control Spike Duplicate (LCSD) must be analyzed at the same frequency.

13.7.2 The recovered concentration must be within default limits of $\pm$ 20%. The true value is 400 mg/L.

13.7.2.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.2.2 If no errors are found and sufficient sample is available, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still out side the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.7.2.3 If sufficient sample volume is not available, report the sample data with appropriate data qualifier on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.7.3 The precision between the LCS and LCSD must a be =/< 20% RPD.

13.7.3.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.3.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.7.4 The lot number of the KHP used to make the CCV is not the same for the LCS.

13.8 **Matrix Spike (MS)** (8.14) **and Matrix Spike Duplicate (MSD)** (8.14)

13.8.1 One pair per up to 10 environmental samples or 10% frequency whichever is more frequent.

13.8.2 Both QC samples must be calculated for accuracy and precision.

13.8.3 The recovered concentration must be within default limits of ± 20%. The true value is 400 mg/L.

13.8.3.1 If four times the concentration of the spike is less than the analyte concentration of the parent, accuracy need not be calculated.

13.8.3.2 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.3.3 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.8.4 The precision between the MS and MSD must a be =/< 20% RPD.

13.8.4.1.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.4.1.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.8.5 The sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD.

13.8.6 The lot number of the KHP used to make the CCV is not the same for the MS and MSD.

13.9 When preparation of a sample exceeds 28 days past the time of collection, notify the project manager before proceeding. If a sample is run past 28 days after collection, flag the result with appropriate data qualifier.

13.10 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with appropriate data qualifier.

13.11 See attachments Table A and Table B for a summary of QC.

14. Method Performance

There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

14.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

14.2 An initial demonstration of capability (IDOC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

14.3 Continuing demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the CDOC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

14.4 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

14.5 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

14.6 Linear Dynamic Range Study - A linear dynamic range study must be conducted at least once. The study is conducted 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.

14.7 Once the linear dynamic range study determination is performed, keep the data, and then every six months verify with a single high point.

15. Pollution Prevention and Waste Management

15.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

15.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of ALL-S-002, *Waste Handling*.

16. References

16.1 Methods of Soil Analysis, 1982 Second Edition, Method 29-3.5.2.1 Walkley-Black Procedure.

16.2 Nelson D W, Sommers L E. A Rapid and Accurate Method for Proceedings of the. Indiana Academy of Science, 1975, 84: 456-462.

16.3 EPA Manual 600 4-79-020, March 1983, Method 410.4, 40CFR Part 136

16.4 PASI Quality Manual, current revision

17. Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

17.1 Table A – Quality Control

17.2 Table B – Analyst/Technician Data Assessment

17.3 Flowchart

17.4 Attachment 1 - Walkley Black Organic Carbon Preparation Log

17.5 Attachment II – Walkley Black Organic Carbon Analytical Log

18. Revisions

Revision Number	Reason for Change	Date
KM-I-004.Rev.00	Converted SOP to PASI format. Added laboratory charted control limits.	8Aug2005
GB-I-037-Rev.00	Converted from Kimberly to Green Bay. 9.1.7 Changed expiration date of Ferrous Sulfate. 11.1.7 Added acceptable range for ferrous sulfate titrant standardization. Clarified 12.1.1.1 and 12.2.1.1.	12Sept2006
S-GB-I-037-REV.01	Updated signature page. Deleted Section 7. Renumbered document. Section 13.3 – Updated Method Blank acceptance criteria Section 14 – Updated SOP references. Updated Attachments	24July2008
S-GB-I-037-Rev.02	Updated body of document to Hach Spec Method Updated Attachments	10Sept2009
S-GB-I-037-Rev.03	Updated SOP refereces for Waste Management SOP to S-GB-S-006 Waste Management and Handling. Table B,1 MB 20X rule changed to 10X.	13Dec2010

Table A. QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**Walkley Black**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. A LCSD is required if MS/MSD is not performed.
Matrix Spike / Matrix Spike Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.
Initial Calibration	Minimum of 5 standards plus blank. Must be performed every time before samples are analyzed.
RL Standard	After the calibration and monthly at minimum.
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. CCV – analyzed after every 10 samples.
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. CCB – analyzed after every CCV pair.

Table B. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Walkley Black If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
RLVS standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 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 concentration of the spike is less than 25% of the concentration of the parent, dilute parent to a level that the subsequent spike concentration will be greater than 25% the parent and so the parent concentration is above the EQL. Rerun the parent, MS, and MSD at this new dilution. If the MS and/or MSD fails at the diluted concentration, appropriately flag the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately flag the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate (precision) data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifiers to reflect the deficiencies.

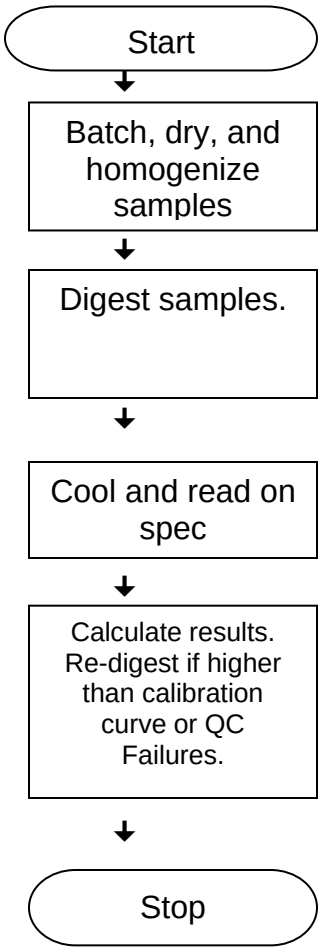
4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB the samples do not have to be reanalyzed.

FLOWCHART


Start
Batch, dry, and homogenize samples
Digest samples.
Cool and read on spec
Calculate results. Re-digest if higher than calibration curve or QC Failures.
Stop

Attachment I: Walkley Black Organic Carbon Preparation Log

Preparation Total Organic Carbon by Walkley Black

Analyst:		**WETA Batch:**	-
Preparation Date:		**HBN:**	-
Oven:	Precision, Model 17, SN:21AR-10		
Oven Temp:			
103°C (+/-2°C)			
In Oven:			
Out of Oven:			

Sample ID:	Sample ID:	Sample ID:
MB	NA	NA
LCS	NA	
LCSD	NA	
NA	NA	
MS	NA	
MSD	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	
NA	NA	

F-GB-I-187-Rev.01 (20Dec2010) Walkley Black Prep Log
Green Bay Laboratory

Attachment II: Walkley Black Organic Carbon Analytical Log

Analysis Total Organic Carbon by Walkley Black

Analyst:	-	**WETA Batch:**	-	**HBN:** -	
Analysis Date:	-	**Digestion Block:**	HACH COD Reactor SN:890700308		
Block Temp:		**Instrument:**	40WTA1	HACH DR 2000/SN:960300039446	
150°C (+/- 5°C)		**Digestion Vial Lot**			
Digestion Started:		**Number:**	10807		
Digestion Finished:		**Balance ID:**	40BAL7		

Standards: Potassium Hydrogen Phthalate (C8H5KO4)
Primary: 800mg/L C -
Secondary: 800mg/L C -

Sample ID:	Initial Weight (g)	Final Vol. (mL)	Absorb @ 620nm	Raw Conc. (mg/L)	Final Conc. (mg/Kg)	Standard TV (mg/L)	% Recovery	Limits	Standard Vol (mL)
CCV1	-	-	-	#VALUE!	NA	400	#VALUE!	90-110%	1.0 Primary
CCB1	-	-	-	#VALUE!	NA	0	#VALUE!	<10	DI Water
MB			-	#VALUE!	#VALUE!	0	#VALUE!	<10	DI Water
LCS			-	#VALUE!	#VALUE!	400	#VALUE!	80-120%	1.0 Secondary
LCSD			-	#VALUE!	#VALUE!	400	#VALUE!	80-120%	1.0 Secondary
NA			-	#VALUE!	#VALUE!	-	-		
MS			-	#VALUE!	#VALUE!	400	#VALUE!	80-120%	1.0 Secondary
MSD			-	#VALUE!	#VALUE!	400	#VALUE!	80-120%	1.0 Secondary
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
CCV2	-	-	-	#VALUE!	NA	400	#VALUE!	90-110%	1.0 Primary
CCB2	-	-	-	#VALUE!	NA	0	#VALUE!	<10	DI Water
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
CCV3	-	-	-	#VALUE!	NA	400	#VALUE!	90-110%	1.0 Primary
CCB3	-	-	-	#VALUE!	NA	0	#VALUE!	<10	DI Water
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
NA			-	#VALUE!	#VALUE!	-	-		
CCV4	-	-	-	#VALUE!	NA	400	#VALUE!	90-110%	1.0 Primary
CCB4	-	-	-	#VALUE!	NA	0	#VALUE!	<10	DI Water

Calculation: (Absorb @620nm - Intercept)/Slope = mg/L TOC
[(mg/L TOC)*Final Vol (mL)]/Initial Weight (g) = mg/Kg TOC

Comments:

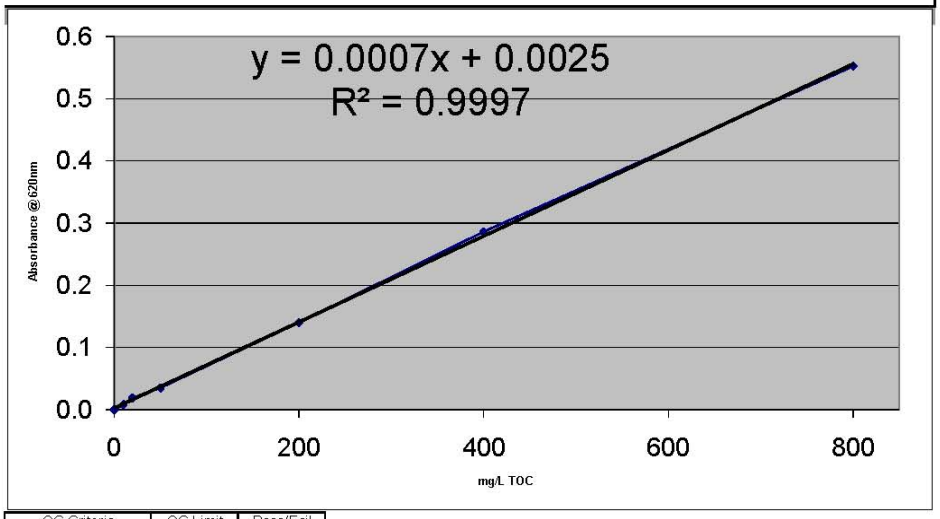
F-GB-I-188-Rev.01 (20Dec2010) Walkley Black Analytical Benchsheet
Green Bay Laboratory

Calibration Total Organic Carbon by Walkley Black

Analyst:			
Calibration Date:		**Digestion Block:**	HACH COD Reactor SN:890700308
Block Temp:		**Instrument:**	40WTA1 HACH DR 2000/SN:960300039446
150°C (+/- 5°C)		**Digestion Vial Lot**	
Digestion Started:		**Number:**	
Digestion Finished:		**Balance ID:**	40BAL7

Standards:	Potassium Hydrogen Phthalate (C8H5KO4)	
Primary:	800mg/L C	-
Secondary:	800mg/L C	-

Standard ID	Conc. mg/L	Std Used	Std. Vol. Used (mL)	DI Water Vol. (mL)	Final Vol. (mL)	Absorb @ 620nm	mg/L Result	% Recovery	
Cal 1	0	DI Water	0.000	2.0	2.0	0.000	-3.6	NA	
Cal 2	10	Primary	0.025	1.975	2.0	0.008	7.9	78.6	Slope
Cal 3	20	Primary	0.075	1.925	2.0	0.019	23.6	117.9	0.0007
Cal 4	50	Primary	0.125	1.875	2.0	0.035	46.4	92.9	
Cal 5	200	Primary	0.5	1.5	2.0	0.140	196.4	98.2	Intercept
Cal 6	400	Primary	1.0	1.0	2.0	0.286	405.0	101.3	0.0025
Cal 7	800	Primary	2.0	0.0	2.0	0.552	785.0	98.1	
ICV	400	Secondary	1.0	1.8	2.0	0.279	395.0	98.8	
ICB	0	DI Water	0.0	2.0	2.0	0.000	-3.6	NA	
RLVS	10	Primary	0.025	1.975	2.0	0.010	10.7	107.1	
CCV	400	Primary	0.200	1.8	2.0	0.279	395.0	98.8	
CCB	0	DI Water	0.0	2.0	2.0	-0.001	-5.0	NA	



QC Criteria	QC Limit	Pass/Fail
R^2	>0.9949	
ICV	+/-10%	Pass
ICB	<RL	Pass
RLVS	+/-40%	Pass
CCV	+/-10%	Pass
CCB	<RL	Pass

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STANDARD OPERATING PROCEDURE

The Determination of Total Organic Carbon Using the Apollo 9000 Instrument

Reference Method: EPA 9060A

SOP NUMBER:	S-GB-I-059-REV.02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-059-REV.01

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 11/18/10

Kate Grams, Laboratory Quality Manager — Date: 11/8/10

Chad Rusch, Department Manager — Date: 11/15/2010

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 Standard Operating Procedure (SOP) is to describe the procedures used to determine the concentration of Total Organic Carbon (TOC) in solid and soil samples using the Apollo 9000 Instrument.

2. SCOPE AND APPLICATION

This SOP is applicable the analysis of Total Organic Carbon (TOC) in soils and solids. The applicable range is MDL to the top standard in the curve. The current Pace Reporting Limit is 250 mg/kg. The current MDL can be found in Epic Pro.

3. SUMMARY OF METHOD

The sample is placed in a combustion furnace where it heats up and reacts with the catalyst that is contained in the furnace. The product of the reaction is CO_2 gas, which is then blown into the non-dispersive infrared detector (NDIR). The NDIR uses electromagnetic radiation or infrared energy to measure the CO_2. This measurement is proportional to the carbon in the sample. In order to analyze for TOC the sample must first have the Inorganic Carbon (IC) removed during the preparation process utilizing acid and heat.

4. INTERFERENCES

4.1 Carbonate and bicarbonate carbon represent interferences under the terms of this test and must be removed or accounted for in the final calculation.

4.2 This SOP is applicable only to those samples that can be adequately ground with mortar and pestle to result in a fairly homogenous sample.

5. SAFETY

5.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

5.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

5.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

6. DEFINITIONS

6.1 Total Carbon (TC): The measurement of all the carbon in the sample, both inorganic and organic.

6.2 Total Organic Carbon (TOC): All carbon atoms covalently bonded in organic molecules. To measure TOC, all the inorganic carbon is removed by addition of acid and purging: The result is that only organic carbon is left.

6.3 Total Inorganic Carbon (TIC): The measurement of the total inorganic carbon is found by subtracting the Total Organic Carbon from the Total Carbon. TIC = TC – TOC.

6.4 Additional definitions can be found in Section 10 of the Pace Analytical Services, Inc. Quality Manual.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

Table 1. SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Matrix	Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Solid	9060A	4 oz Clean glass containers	None 28 days	On ice 4+/- 2° Celsius	4^0C

8. REAGENTS AND STANDARDS

8.1 Reagents

8.1.1 Sulfuric Acid (H_2SO_4) – concentrated

8.1.2 Sulfuric Acid (H_2SO_4) – 10%

To make: Fill 2000mL volumetric flask with 1000mL of DI Water. Measure out 200mL of concentrated Sulfuric Acid and add to volumetric flask. Dilute to volume. Shelf life = 6 months.

8.1.3 Deionized water (DI Water)

8.1.4 Purified Compressed Air

8.2 Standards (Equivalent pre-made standards may be purchased and used)

8.2.1 Potassium hydrogen phthalate (KHP) – 4000 mg/L calibration stock solution

To make: Fill a 100mL volumetric flask with approximately 50mL of DI water. Dissolve 850mg of KHP in the DI water. Dilute to volume. Shelf life = 1 month.

8.2.2 Soil Calibration Standard 1 – 0 mg/Kg

10mL DI water.

8.2.3 Soil Calibration Standard 4 – 1000mg/Kg

Fill a 10mL volumetric flask with approximately 5mL of DI water. Pipette 2.5mL of calibration stock solution into the flask. Dilute to volume. Shelf life = 1 month.

8.2.4 Soil Calibration Standard 2 and Reporting Limit Verification Standard (RLVS) – 250mg/Kg

Fill a 10mL volumetric flask with approximately 5mL of DI water. Pipette 2.5mL of Soil Calibration Standard 4 (1000mg/Kg) into the flask. Dilute to volume. Shelf life = 1 month.

8.2.5 Soil Calibration Standard 3 – 500mg/Kg

Fill a 10mL volumetric flask with approximately 5mL of DI water. Pipette 1.25mL of Soil calibration stock solution into the flask. Dilute to volume. Shelf life = 1 month.

8.2.6 Soil Calibration Standard 5– 2000mg/Kg

Fill a 10mL volumetric flask with approximately 5mL of DI water. Pipette 5.0mL of calibration stock solution into the flask. Dilute to volume. Shelf life = 1 month.

8.2.7 Potassium hydrogen phthalate (KHP) – 4000mg/L ICV stock solution

Fill a 100mL volumetric flask with approximately 50mL of DI water. Dissolve 850mg of KHP in the DI water. Dilute to volume. Shelf life = 1 month.

Note: The KHP used to prepare the ICV stock solution must be from another source than the KHP used to prepare the calibration stock solution.

8.2.8 Soil Initial Calibration Verification (ICV) –1000mg/Kg

Fill a 10mL volumetric flask with approximately 5mL of DI water. Pipette 2.5mL of ICV stock solution into the flask. Dilute to volume. Shelf life = 1 month.

8.2.9 Laboratory Control Spike (LCS) – An independently prepared certified solution of Potassium hydrogen phthalate (KHP) from the calibration curve at a concentration equal to the mid-range calibration standard. Shelf life = 1 month from solution being made in the lab.

8.2.10 Soil Continuing Calibration Verification (CCV) –1000mg/Kg

Fill a 10mL volumetric flask with approximately 5mL of DI water. Pipette 2.5mL of calibration stock solution into the flask. Dilute to volume. Shelf life = 1 month.

9. EQUIPMENT AND SUPPLIES

9.1 Equipment

9.1.1 Tekmar / Dohrmann Apollo 9000 TOC analyzer with NDIR detector

9.1.2 TOC Boat Analyzer – Rosemount/Dohrmann

9.1.3 Calibrated Mechanical Pipettes –Assorted adjustable air displacement pipettes with disposable tips - Eppendorf or equivalent

9.1.4 Analytical syringe – 500uL

9.1.5 Analytical syringe – 50uL

9.1.6 Hot Block

9.1.7 Analytical balance – accurate to 0.0001gram

9.1.8 Mortar & Pestle

9.1.9 Spatula

9.1.10 Volumetric flasks

9.1.11 Oven

9.2 Supplies

9.2.1 Aluminum dishes

9.2.2 40 mL VOA vials

9.2.3 Digestion Tubes

9.2.4 Data printout paper

10. CALIBRATION AND STANDARDIZATION

10.1 Turn on the **TOC Boat Sampler** power (oven) and the **Compressed Air**.

10.2 Enter **TOC/Apollo 9000** software through the **Talk 4.2 Boat** icon.

10.3 *Activating the instrument.* Go to the **Setup** dropdown and select **Instrument**. Go to the **System** portion and select **Ready**. A bubbling sound will be made, signaling the instrument is not hibernating.

10.4 *Setting up a new curve.* Go to **Setup** dropdown and select **Calibration** then **Standards**. This is where the new calibration and the calibration points are set. Through the **Edit** drop down rows can be added, deleted, or copied and pasted. Create the following curve:

Table 2. Calibration Information

Standard ID	Concentration (ppmC)	Method ID	ugC	Comments
0ppmC	0	Boat Sampler	0.0000	
250ppmC	250	Boat Sampler	10.0000	
500ppmC	500	Boat Sampler	20.0000	
1000ppmC	1000	Boat Sampler	40.0000	
2000ppmC	2000	Boat Sampler	80.0000	

The **Cal. Curve ID** will be filled in when **File, Save As** is done. Save the file as the date that the calibration will be done. Click **OK** to exit.

10.5 *Activating the New Curve*. Set the new curve as the active curve by going to the **Setup** dropdown and selecting **Calibration,** then **Set Active,** and **TOC Curves**. From the popup set the **Aux** to the calibration curve just set up. Leave all the other fields set to **Default**. **OK** will close the popup.

10.6 *Running the New Curve*. Click **Run** then **Sample Setup** and change the sample type to **TOC Standard**. In the popup select **Aux** and then click **OK**. In the new popup select the level that is to be run by highlighting it. Number of Repeats should be 1, Method ID is Boat Sampler, Mode is TOC, and the Calibration Curve should reflect the new curve. Click Save/Use. **Click Start** and enter sample weight (40uL for standards and blanks) and follow the prompts to inject the boat. After a calibration point is run a calibration popup will appear showing each point that has been analyzed. From this popup different points can be selected, the curve calculated, and the calibration report printed. This popup is closed by clicking **OK**.

10.7 *Rest of Calibration Points*. Repeat 10. 6 for the rest of the calibration points. For each point **TOC Standard** will need to be selected even though it is already showing. This is done to initiate the next popup.

10.8 When four standards have been read click on menu command **Results**, **Calibration**. Select the calibration curve ID. This is the same as the popup that shows after each calibration point. Locate and check the calibration points just performed, all others should not be checked. Click "**Recalc**" button. This will create a calibration curve. The curve will be 1st order linear. The r-sqr value must be greater than 0.995, if not, recalibrate.

10.9 *Initial Calibration QC*. Change the **Sample Type** to **Sample** and analyze the applicable QC: ICV, ICB, RLVS, CCV, and CCB.

10.10 The calibration report and all analyses should be printed out with all standards traced back to COA and scanned to a file on the desktop labeled the same as the calibration ID.

11. PROCEDURE

11.1 Two methods are used for soil samples, 9060A Quads and 9060A Mods. For 9060A Quads four replicates analyses of the sample are performed with each replicate, the RSD, and the average being reported. For 9060A Mods two replicate analyses of the sample are preformed with each replicate the RPD, and the average being reported.

11.2 No injections can be used if they are above the curve. A smaller amount of sample must be used and rerun.

11.3 Soil Sample Preparation

11.3.1 Take an approximate sub-sample of 10 g of soil, add 10% Sulfuric Acid and place in a digestion block at 70°C. After sample does not react with acid. It is filtered and then baked in an oven at 103°C for a minimum of 18 hours.

11.3.2 Crush and grind the sample with a mortar and pestle to form a homogenous sample.

11.4 Epic Pro/LimsLink.

11.4.1 Samples are batched in Epic Pro.

11.4.2 LimsLink is used to aquire and evaluate data as it is generated.

11.4.3 Sample weights and instrument raw results are recorded in LimsLink.

11.4.4 Instrument QC and replicate precision are evaluated in LimsLink.

11.4.5 Acceptable data is then posted to EPIC Pro.

11.5 Sample Analysis.

11.5.1 Turn on the **TOC Boat Sampler** power (oven) and the **Compressed Air**.

11.5.2 Enter **TOC/Apollo 9000** software through the **Talk 4.2 Boat** icon.

11.5.3 *Activating the instrument.* Go to the **Setup** dropdown and select **Instrument**. Go to the **System** portion and select **Ready**. A bubbling sound will be made, signaling the instrument is not hibernating.

11.5.4 *Each analysis is performed as follows:* Click **Run** then **Sample Setup** and change the **Sample ID** to what is being analyzed. Sample Type should be **Sample**. Number of Repeats should be 1, Method ID is Boat Sampler, Mode is TOC, and the Calibration Curve should reflect the curve being used. Click Save/Use. **Click Start** and enter sample weight (40uL for standards and blanks) and follow the prompts to inject the boat. Once the instrument beeps and prints out the result from the injection, slide the boat out of the oven and wait at least one minute for the boat to cool down.

11.5.5 *CCV Analysis:* Inject 40 µL of the 1000mg/Kg Soil Continuing Calibration Verification (CCV) standard into the boat through the septa using a 50-µL syringe.

11.5.6 *CCB Analysis:* Inject 40µL of DI H_2O (CCB).

11.5.7 *Batch QC:* When running a Method Blank (MB), 40uL of DI water is injected into the boat. A Laboratory Control Spike (LCS) is run by injecting 40uL of the prepared LCS standard. Matrix Spike / Matrix Spike Duplicate (MS/MSD) are run by injecting 20uL of the 4000mg/L calibration stock standard along with a variable weight of the parent sample for each the MS and MSD.

11.5.8 *Samples:* For each field sample in an analytical batch, weigh 0.04g or less into the boat. Run a CCV/CCB every 10 samples and at the end of the run.

11.5.9 While running samples, no injections can be used if they are above the curve. If an injection is above the curve, weigh out a smaller amount of the sample and rerun that injection.

11.5.10 To determine that the sample concentration is less than the reporting limit, at least 0.04 grams must be utilized.

11.5.11 *Continuing Analytical QC:* Run a CCV/CCB every 10 samples and at the end of the run.

11.5.12 *Printing data:* After the run has completed go to the **Results** drop down and select **Samples**. From here select the **File** dropdown and **Clear**. Then select the **File** drop down and Open **Results From Day**. Type in the year, month and day the samples were analyzed and then click **OK**. The samples will then appear. From the **File** dropdown select both **Print Summary Report** and **Print Detail Report**. Click **Exit** and **No** to Save Changes.

11.5.13

11.6 Shutdown

11.6.1 Slide boat into oven.

11.6.2 Turn off the **TOC Boat Sampler** power (oven) and the **Compressed Air**.

11.7 Calculations:

11.7.1 All data produced by the Apollo 9000 must be processed through the appropriate curve.

Injection 1:
Soil:

$$\text{Raw Data Value (mg/L)} \times \frac{0.04\ (\text{ml})}{\text{Injection weight (g)}} \times \frac{1000\ (\text{g})}{1\ (\text{Kg})} \times \frac{1(\text{L})}{1000\ (\text{ml})} = \text{TOC (mg/Kg) (TOC1)}$$

Injection 2:
Soil:

$$\text{Raw Data Value (mg/L)} \times \frac{0.04\ (\text{ml})}{\text{Injection weight (g)}} \times \frac{1000\ (\text{g})}{1\ (\text{Kg})} \times \frac{1\ (\text{L})}{1000\ (\text{ml})} = \text{TOC (mg/Kg) (TOC2)}$$

Injection 3:
Soil:

$$\text{Raw Data Value (mg/L)} \times \frac{0.04\ (\text{ml})}{\text{Injection weight (g)}} \times \frac{1000\ (\text{g})}{1\ (\text{Kg})} \times \frac{1\ (\text{L})}{1000\ (\text{ml})} = \text{TOC (mg/Kg) (TOC3)}$$

Injection 4:
Soil:

$$\text{Raw Data Value (mg/L)} \times \frac{0.04\ (\text{ml})}{\text{Injection weight (g)}} \times \frac{1000\ (\text{g})}{1\ (\text{Kg})} \times \frac{1\ (\text{L})}{1000\ (\text{ml})} = \text{TOC (mg/Kg) (TOC4)}$$

11.7.2 Average sample result (mg/kg)

$$\text{Mod Average result} = \frac{(\text{TOC1}) + (\text{TOC2})}{2}$$

$$\text{Quad Average result} = \frac{(\text{TOC1}) + (\text{TOC2}) + (\text{TOC3}) + (\text{TOC4})}{4}$$

Note that soils are dried to 100% dry weight before analysis – therefore dry weight correction is not needed.

11.7.3 The result in mg/kg is divided by 10,000 to express the result as a percentage (%).

11.7.4 MS/MSD calculation

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

SSR = Spike sample result
SR = Sample result
SA = Spike added

11.7.5 Precision (for MOD replicates)

The Relative Percent Difference is calculated between the two replicates used to calculate the average.

$$\text{Replicate \% RPD} = \frac{|\text{TOC1} - \text{TOC2}|}{(\text{TOC1}+\text{TOC2})/2} \times 100$$

11.7.6 Precision (for Quad replicates)

The Relative Standard Deviation is calculated for the four replicates used to calculate the average.

$$\text{Mean} = (\text{TOC1}+\text{TOC2}+\text{TOC3}+\text{TOC4})/4$$

$$\text{Standard Deviation} = \sqrt{(((\text{TOC1}-\text{Mean})+(\text{TOC2}-\text{Mean})+(\text{TOC3}-\text{Mean})+(\text{TOC4}-\text{Mean}))/3)}$$

$$\text{Relative Standard Deviation} = (\text{Standard Deviation}/\text{Mean})*100$$

11.7.7 Precision (for QC samples):

The precision is calculated based on the % recovery of the matrix spike / matrix spike duplicate (MS/MSD) result.

$$\text{QC Sample \% RPD} = \frac{|\text{MS}-\text{MSD}|}{(\text{MS}+\text{MSD})/2} \times 100$$

12. QUALITY CONTROL

12.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

12.2 **Initial Calibration Verification (ICV)** must be run immediately following the instrument calibration. The ICV must be prepared from stock standard obtained from a different source than the stock standard used to prepare the initial calibration and must be prepared at a level at or near the mid range of the calibration curve. The result of the ICV must fall within the range of 90%-110% - if not rerun or recalibrate.

12.3 **Initial Calibration Blank (ICB**) – An ICB is analyzed after the ICV and must meet the acceptance criteria before samples are analyzed. The absolute value of the ICB result must be less than the Pace Reporting Limit (PRL) – if not rerun or recalibrate.

12.4 **Continuing Calibration Verification (CCV) -** Each analytical run must begin and end with a CCV and a CCV must be run every 10 samples. The CCV must be prepared from the same solution as the mid-point calibration standard and the result must fall within the range of 90%-110%. If outside the range, rerun 1X. If still outside the range, the problem must be corrected before continuing and the affected samples reanalyzed.

12.5 **The Continuing Calibration Blank (CCB**) – A CCB is analyzed after the CCV and must meet the acceptance criteria before samples are analyzed. The absolute value of the CCB result must be less than the PRL. If outside the range, rerun 1 time. If still outside the range, correct the problem and rerun all samples back to the last passing CCB.

12.6 **Method Blank** - A Method Blank (MB) must be prepared and analyzed with every sample batch or every 20 samples whichever is more frequent. The data obtained is used to assess contamination from the laboratory environment. Values that exceed the PRL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis. Values that are between the MDL and PRL are evaluated based on the concentration in the samples compared to the concentration in the blank.

12.7 **Laboratory Control Sample (LCS**) - A LCS prepared from an independent source must be performed with every batch or every 20 environmental samples, which ever is more frequent. The LCS must be evaluated for accuracy and must meet the laboratory generated control limits before samples are analyzed. If no in-house limits are in use, then a range of 80 - 120% will be used. If the LCS recovery does not meet the acceptance criteria corrective action must be taken and the LCS recovery must be acceptable before proceeding with sample analysis. A LCSD is performed when requested by the client or when there is insufficient sample volume to perform a matrix spike / matrix spike duplicate. The LCS/LCSD must be evaluated for precision and must meet the laboratory generated control criteria.

12.8 **Matrix spike/Matrix spike duplicate** - A MS/MSD pair must be prepared and analyzed every 10 samples. The MS must be a duplicate of the aliquot used for sample analysis. Calculate the percent recovery, corrected for concentration measured in the parent sample, and compare to the in-house generated limits. If no in-house limits are in used then a range of 80 - 120% will be used. Calculate the precision between the MS and MSD and compare to the in-house generated limits. If no in-house limits are in use, then an upper limit of 20% will be used.

12.9 **Reporting Limit Verification Standard (RLVS)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

12.10 **MOD Replicate Precision (RPD)** - The Relative Percent Difference is calculated between the two replicates used to calculate the average. If the RPD resulting from the two reported replicates is 40% or greater, two additional replicates are analyzed. The calculated results of each of these two additional replicates are reported in a qualifier for the reported RPD.

12.11 **Quad Replicate Precision (RSD)** - The Relative Standard Deviation is calculated for the four replicates used to calculate the average. If the RSD resulting from the four reported replicates is 40% or greater, a qualifier for the reported RSD is added.

12.12 When preparation of a sample exceeds 28 days past the time of collection, notify the project manager before proceeding. If a sample is run past 28 days after collection, flag the result with appropriate data qualifier.

12.13 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with appropriate data qualifier.

12.14 See attachments Table A and Table B for a summary of QC.

13. METHOD PERFORMANCE

13.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Linear Calibration Range - The linear calibration range must be determined initially and verified whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. If any verification data exceeds the initial values by $\pm$ 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

13.1.5 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

14.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

14.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

15. REFERENCES

15.1 PASI Quality Manual, current revision

15.2 USEPA Test Methods for Evaluating Solid Wastes, SW 846, 3rd Edition, Methods 9060A.

15.3 Apollo 9000 TOC Combustion Analyzer User Manual

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

16.1 Table A: **QUALITY CONTROL**

16.2 Table B: **ANALYST/TECHNICIAN DATA ASSESSMENT**

17. REVISIONS

Revision Number	Reason for Change	Date
S-GB-I-059-Rev.00	First Issue.	09Jan2009
S-GB-I-059-Rev.01	Updated Method Reference to SW846-9060A throughout document.	20May2009
S-GB-I-059-Rev.02	Updated Signature Page Section 8.2: Updated calibration information. Section 11.5.13: Removed Section 11.7: Updated calculations to include multiple replicates and manner to average replicates for final TOC value. Section 12.0: Added additional criteria for RSD of Quadruplicate analysis and RPD criteria for Mod analysis. Throughout document: Updated instances of the language "EQL" with "PRL".	08Nov2010

Table A. QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**TOC**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. A LCSD is required if MS/MSD is not performed.
Matrix Spike / Matrix Spike Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.
Initial Calibration	Minimum of 4 standards plus blank.
RLVS	After the calibration and monthly at minimum.
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. CCV – analyzed after every 10 samples.
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. CCB – analyzed after every CCV pair.

Table B. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**TOC If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
RLVS standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 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 concentration of the spike is less than 25% of the concentration of the parent, dilute parent to a level that the subsequent spike concentration will be greater than 25% the parent and so the parent concentration is above the EQL. Rerun the parent, MS, and MSD at this new dilution. If the MS and/or MSD fails at the diluted concentration, appropriately flag the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately flag the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate precision data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB the samples do not have to be reanalyzed.

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STANDARD OPERATING PROCEDURE

The Determination of Total Organic Carbon Using the Teledyne Tekmar Fusion

Reference Methods: SM 5310 C and SW846 9060A

SOP NUMBER:	S-GB-I-063-Rev.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-063-Rev.00

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 11/18/10

Kate Grams, Laboratory Quality Manager — Date: 11/8/10

Chad Rusch, Department Manager — Date: 11/15/2010

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 Standard Operating Procedure (SOP) is to describe the procedures used to determine the concentration of Total Organic Carbon (TOC), Total Carbon (TC), and Total Inorganic Carbon (TIC) in aqueous samples using the Teledyne Tekmar Fusion Instrument. This SOP follows analytical methods SM 5310C and SW 846-9060.

2. SCOPE AND APPLICATION

This SOP pertains to drinking waters, ground waters, surface waters, domestic and industrial waste samples. This SOP includes analysis using quadruplicate repetitions for SW 846-9060 and duplicate repetitions for SM 5310C.

3. SUMMARY OF METHOD

When running water samples, the sample is injected into a reactor filled with a persulfate solution where it is oxidized by means of ultra-violet light. The product of the reaction is CO_2 gas, which is then blown into the non-dispersive infrared detector (NDIR). The NDIR uses infrared energy to measure the CO_2. This measurement is proportional to the carbon in the sample. In order to analyze for TOC the water sample must first have the Inorganic Carbon (IC) removed by the addition of acid and being purged with nitrogen. TC analysis is done with out the acid addition and nitrogen purge.

4. INTERFERENCES

4.1 Carbonate and bicarbonate carbon represent interferences under the terms of this test and must be removed or accounted for in the final calculation.

4.2 When dealing with a water matrix, this SOP is applicable only to homogenous samples that can be injected into the apparatus reproducibly by means of an auto-sampler syringe.

5. SAFETY

5.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

5.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

5.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

6. DEFINITIONS

6.1 Total Carbon (TC): The measurement of all the carbon in the sample, both inorganic and organic.

6.2 Total Organic Carbon (TOC): All carbon atoms covalently bonded in organic molecules. To measure TOC, all the inorganic carbon is removed by addition of acid and purging: The result is that only organic carbon is left.

6.3 Total Inorganic Carbon (TIC): The measurement of the total inorganic carbon is found by subtracting the Total Organic Carbon from the Total Carbon. TIC = TC – TOC.

6.4 Additional definitions can be found in Section 10 of the Pace Analytical Services, Inc. Quality Manual.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Water samples are collected in amber glass 40mL VOA vials or 4oz amber glass jars, which are acidified with H_2SO_4 to a pH of <2. Samples are shipped to the lab on ice and stored at 4°C until time of analysis. Analysis must occur within 28 days of sample collection. Samples to be analyzed for Total Carbon must be received unpreserved in amber glass 40mL VOA vials or 4 oz amber glass jars.

8. REAGENTS AND STANDARDS

8.1 Reagents

8.1.1 Phosphoric Acid (H_3PO_4) – 85%

8.1.2 Phosphoric Acid (H_3PO_4) – 21%

To make: Fill 1000mL volumetric flask with 500mL of DI Water. Pour 250mL phosphoric acid into the volumetric. Transfer to 1000mL bottle and label. Shelf life = 1 year.

8.1.3 Deionized water (DI water)

8.1.4 Purified Compressed Nitrogen

8.1.5 Sodium Persulfate – Neat

8.1.6 Sodium Persulfate Solution, 10.0% w/v – Dissolve 100 g of sodium persulfate in a one liter volumetric flask, add 500 ml DI water followed by 25 ml 85% phosphoric acid. Dilute to volume with deionized water.

8.2 Standards

8.2.1 Two sources of neat Potassium hydrogen phthalate (KHP)

8.2.1.1 A **Primary Source** to be used for C**alibration**, **CCV**s and **RLVS** standards. Expiration date – 5 years from date of receipt, not to exceed manufacturers expiration date.

8.2.1.2 A **Secondary Source** to be used for **ICV, LCS**, and **MS/MSD** standards and spikes. Expiration Date – 5 years from date of receipt, not to exceed manufacturers expiration date.

8.2.2 See Table A for Stock, Intermediate, and Working Standards, instructions and concentrations. **Shelf Life = 1 Month**

9. EQUIPMENT AND SUPPLIES

9.1 **Equipment**

9.1.1 Teledyne Tekmar Fusion TOC analyzer with NDIR detector

9.1.2 Calibrated Mechanical Pipettes –Assorted adjustable air displacement pipettes with disposable tips - Eppendorf or equivalent.

9.1.3 Volumetric flasks

10. CALIBRATION AND STANDARDIZATION

10.1 Turn on the Teledyne Tekmar Fusion TOC analyzer power and the Compressed Nitrogen.

10.2 Enter Teledyne Tekmar Fusion software through the Fusion icon. Double click on the instrument in the dialog box, this will open the full screen instrument control.

10.3 **Activating the instrument**. Go to the Instrument dropdown and select Ready Mode. This will turn on the UV lamp and ready the instrument for use.

10.4 **Setting up a new curve**. Go to NEW dropdown and select Calibration then TOC. This is where the new calibration curve is named. Through the Edit drop down rows can be

10.5 **Activating the New Curve**. Set the new curve as the active curve by going to the Setup dropdown and selecting Calibration, then Set Active, and TOC Curves. From the popup set the Aux to the calibration curve just set up. Leave all the other fields set to Default. OK will close the popup.

10.6 **Running the New Curve**. Click Run then Sample Setup and change the sample type to TOC Standard. In the popup select Aux and then click OK. In the new popup select the level that is to be run by highlighting it. Number of Repeats should be 1, Method ID is based on ppm range and date made, Mode is TOC, and the Calibration Curve should reflect the new curve. Click Save/Use. Click Start and enter sample weight (40uL for standards and blanks) and follow the prompts to inject the boat. After a calibration point is run a calibration popup will appear showing each point that has been analyzed. From this popup different points can be selected, the curve calculated, and the calibration report printed. This popup is closed by clicking OK.

10.7 Rest of Calibration Points. Repeat 10.4.6 for the rest of the calibration points. For each point TOC Standard will need to be selected even though it is already showing. This is done to initiate the next popup.

10.8 When four standards have been read click on menu command Results, Calibration. Select the calibration curve ID. This is the same as the popup that shows after each calibration point. Locate and check the calibration points just performed, all others should not be checked. This will create a calibration curve. The curve will be 1st order linear. The r-squared value must be greater than 0.995, if not, recalibrate.

10.9 Initial Calibration QC. Change the Sample Type to Sample and analyze the applicable QC: ICV, ICB, RLVS, CCV, and CCB.

10.10 The calibration report and all analyses should be printed out with all standards traced back to COA and scanned to a file on the desktop labeled the same as the calibration ID.

11. PROCEDURE

11.1 There are two different methods by which to analyze TOC: SW846 9060A and SM 5310C.

11.1.1 When referencing Method SW846 9060A, quadruplicate analysis is required for all samples. Each sample will be composed of 4 individual injections. Report all four individual injection readings and the average of the four readings.

11.1.2 When referencing Method SM 5310C, two injections of the sample within 10% RPD is a valid analysis. The first injection reading that is within 10% RPD of the next injection reading is reported. If the first two injection readings are at or below the PQL, precision is NOT calculated and the first injection reading is reported. If no injection reading is within 10% RPD of the next injection reading and the injection readings are above the PQL, the sample must be rerun. This is used for water matrix only.

11.1.3 No injections can be used if they are above the curve. The sample must be diluted and rerun.

11.2 Water Sample Preparation

11.2.1 Prepare the water sample as follows:

11.8.4.1 Shake sample vigorously to suspend the sediment.

11.8.4.2 Pipette 1.00mL of sample into a clean 40mL auto-sampler vial and dilute to 20mL with 19.0mL of lab water.

11.8.4.3 The Teledyne Tekmar Fusion will add 1mL of 21% Phosphoric Acid to all samples and QC including MB, LCS, and MS/MSD and purge with Nitrogen. For TC analysis the Teledyne Tekmar Fusion will not add 1mL of 21% Phosphoric Acid and not purge with Nitrogen.

11.8.4.4 Prepare Method Blank (MB) by pipetting 20.0mLs DI water into clean 40mL auto-sampler vial.

11.8.4.5 Prepare the Laboratory Control Spike (LCS) by pipetting 1.0mL of 100mg/L Secondary Intermediate Standard into clean 40mL auto-sampler vial and dilute to 20mL with 19.0mL of lab water.

11.8.4.6 Prepare the Matrix Spike (MS), Matrix Spike Duplicate (MSD) by pipetting 1.0mL of 100mg/L Secondary Intermediate Standard and 1.00mL of sample into clean 40mL auto-sampler vial and dilute to 20mL with 18.0mL of lab water.

11.3 Water Sample Analysis.

11.3.1 Turn on the compressed nitrogen tank then turn on the Teledyne Tekmar Fusion power switch on the instrument.

11.3.2 Log onto Fusion icon on desktop

11.3.3 Once Fusion window is open click on Ready icon on top toolbar to turn on instrument lamp.

11.3.4 Build sample schedule by selecting New and then Schedule.

11.3.5 Schedule begins with line 1 clean, line 2 blank, followed by the batch samples with appropriate CCV, CCB, and RLVS samples included. Run a CCV/CCB every 10 samples and at the end of the run.

11.3.6 Sample ID follows the following format “sample number_Batch number_X dilution”. Click on Method ID cell and then select the appropriate method being used in the batch, method will attach its calibration automatically. This is where TC and TOC is differentiated.

11.3.7 Select number of reps to be run, CCV, CCB, RLVS run via single reps, samples are 4 reps for 9060 and 2 reps for 5310C.

11.3.8 Once all samples have been added to schedule click “save and start” on upper toolbar. Save schedule as “WETA Batch # HBN # TOC Date”

11.3.9 Schedule will shut off automatically once the end of the schedule has been reached.

11.4 Shutdown

11.4.1 Turn off the Teledyne Tekmar Fusion power switch, upper right front of instrument.

11.4.2 Turn off the compressed nitrogen at the cylinder valve.

11.5 Epic Pro/LimsLink

11.5.1 Samples are batched in Epic Pro.

11.5.2 Samples are pulled into a new LimsLink file created for the batches in the analysis.

11.5.3 The sample dilutions are recorded in LimsLink.

11.5.4 When the analytical run has been completed, acceptable data is then posted to Epic Pro.

11.6 Calculations:

11.6.1 The instrument provides raw data results in ppm (mg/L) for aqueous samples. Add data produced by the Teledyne Tekmar Fusion must be processed through the appropriate curve.

11.6.2 SW846 9060A Sample Calculations:

Injection 1:

Raw Data Value mg/L X Dilution = TOC (mg/L) (TOC1)

Injection 2:

Raw Data Value mg/L X Dilution = TOC (mg/L) (TOC2)

Injection 3 (Quad Only):

Raw Data Value mg/L X Dilution = TOC (mg/L) (TOC3)

Injection 4 (Quad Only):

Raw Data Value mg/L X Dilution = TOC (mg/L) (TOC4)

11.6.3 SW846 9060A average Sample result (quadruplicate)

$$\text{Quad Average Result} = \frac{(TOC1) + (TOC2) + (TOC3) + (TOC4)}{4 \{\text{number of injections}\}}$$

11.6.4 For samples analyzed in Quadruplicate (SW846 9060A) each injection is calculated using the appropriate injection equation according to 11.6.2. The average final result is acquired using 11.6.3. The final average result from 11.6.4 is reported as well as each individual injection's final result.

11.6.5 SW846 9060A : The accuracy is calculated based on the average and not the individual injections.

11.6.6 SM 5310C: Repetition acceptance criteria

Relative percent difference (RPD) must be less than 10%

$$\%\ RPD = \frac{I\ Rep.\ 1 - Rep.\ 2I}{(Rep.\ 1 + Rep.\ 2)/2} \times 100$$

Rep. 1 = raw amount of first replicate
Rep. 2 = raw amount of second replicate

11.6.7 SM 5310C : The first rep that is within the acceptance criteria stated in 11.6.6 is reported as follows:

Raw Data Value mg/L X Dilution = TOC (mg/L)

11.6.8 MS/MSD calculation for both SW846 9060A and SM 5310C:

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

SSR = Spike sample result
SR = Sample result
SA = Spike added

For SW846 9060A: recovery is only calculated on the final average – not each injection.

11.6.9 Precision (for QC samples) for both SW846 9060A and SM 5310C:

The precision is calculated based on the % recovery of the matrix spike / matrix spike duplicate (MS/MSD) result.

Relative percent difference (RPD) calculation:

$$\% \text{ RPD} = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

For SW846 9060A: precision is only calculated on the final average – not each injection.

12. QUALITY CONTROL

12.1 Refer to the most current version of the Pace Quality Manual Appendix I Quality Control Calculations and SOP GB-Q-009 Common Laboratory Calculations and Statistical Evaluation of Data for equations and calculation details.

12.2 Initial Calibration Verification (ICV) must be run immediately following the instrument calibration. The ICV must be prepared from stock standard obtained from a different source than the stock standard used to prepare the initial calibration and must be prepared at a level at or near the mid range of the calibration curve. The result of the ICV must fall within the range of 90%-110% - if not rerun or recalibrate.

12.3 Initial Calibration Blank (ICB) – An ICB is analyzed after the ICV and must meet the acceptance criteria before samples are analyzed. The absolute value of the ICB result must be less than the laboratory PQL – if not rerun or recalibrate.

12.4 Continuing Calibration Verification (CCV) - Each analytical run must begin and end with a CCV and a CCV must be run every 10 samples. The CCV must be prepared from the same solution as the mid-point calibration standard and the result must fall within the range of 90%-110%. If outside the range, rerun 1X. If still outside the range, the problem must be corrected before continuing and the affected samples reanalyzed.

12.5 The Continuing Calibration Blank (CCB) – A CCB is analyzed after the CCV and must meet the acceptance criteria before samples are analyzed. The absolute value of the CCB result must be less than the laboratory PQL. If outside the range, rerun 1 time. If still outside the range, correct the problem and rerun all samples back to the last passing CCB.

12.6 Method Blank - A Method Blank (MB) must be prepared and analyzed with every sample batch or every 10 samples whichever is more frequent. The data obtained is used to assess contamination from the laboratory environment. Values that exceed the PQL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis. Values that are between the MDL and PQL are evaluated based on the concentration in the samples compared to the concentration in the blank.

12.7 Laboratory Control Sample (LCS) - A LCS prepared from an independent source must be performed with every batch. The LCS must be evaluated for accuracy and must meet the laboratory generated control limits before samples are analyzed. If no in-house limits are in use, then a range of 80 - 120% will be used. If the LCS recovery does not meet the acceptance criteria corrective action must be taken and the LCS recovery must be acceptable before proceeding with sample analysis. A LCSD is performed when requested by the client or when there is insufficient sample volume to perform a matrix spike / matrix spike duplicate. The LCS/LCSD must be evaluated for precision and must meet the laboratory generated control criteria.

12.8 Matrix spike/Matrix spike duplicate - A MS/MSD pair must be prepared and analyzed every 10 samples. The MS must be a duplicate of the aliquot used for sample analysis. Calculate the percent recovery, corrected for concentration measured in the parent sample, and compare to the in-house generated limits. If no in-house limits are in used then a range of 80 - 120% will be used. Calculate the precision between the MS and MSD and compare to the in-house generated limits. If no in-house limits are in use, then an upper limit of 20% will be used.

12.9 Reporting Limit Verification Standard (RLVS) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

12.10 When preparation of a sample exceeds 28 days past the time of collection, notify the project manager before proceeding. If a sample is run past 28 days after collection, flag the result with appropriate data qualifier.

12.11 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with appropriate data qualifier.

12.12 See attachments Table B and Table C for a summary of QC.

13. METHOD PERFORMANCE

13.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, Orientation and Training Procedures. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.2.1 Analysis of four (4) replicates of reagent water spiked with 5.00mL of 100 mg/L Secondary Intermediate Standard to 20 mL final volume with DI Water. The recovery is to be within the current water LCS QC limits for the known concentrations and 20% RSD for all replicates.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, Method Detection Limit Studies, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Linear Calibration Range - The linear calibration range must be determined initially and verified whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. If any verification data exceeds the initial values by $\pm$ 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

13.1.5 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, PE/PT Program, to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

14.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, Waste Handling and Management.

14.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

15. REFERENCES

15.1 40CFR Part 136 National Institute of Standards and Technology (NIST)

15.2 Standard Methods for the Examination of Water and Wastes, on-line Edition – Method 5310 C -00.

15.3 USEPA Test Methods for Evaluating Solid Wastes, SW 846, 3rd Edition, Methods 9060A.

15.4 PASI Quality Manual, current revision

15.5 Teledyne Tekmar Fusion TOC analyzer User Manual

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

16.1 Table A**: STANDARD**S

16.2 Table B: **QUALITY CONTROL**

16.3 Table C: **ANALYST/TECHNICIAN DATA ASSESSMENT**

17. REVISIONS

Revision Number	Reason for Change	Date
S-GB-I-063-Rev.00	First Issue	28Dec2009
S-GB-I-063-Rev.01	Updated Signature Page	08Nov2010

Table A: STANDARDS:

Standard Name	Final Conc. (mg/L)	Amount of Std Added	Conc. of Std	Name of Std Used	Reagent Used	Final Volume (mL)
Stock and Intermediate Standards						
4000mg/L Primary Stock Standard	4000	4250 mg	Neat	Primary KHP Neat	DI Water	500
4000mg/L Secondary Stock Standard	4000	4250 mg	Neat	Secondary KHP Neat	DI Water	500
100mg/L Primary Intermediate Standard	100	5.0 mL	4000 mg/L	4000mg/L Primary Stock Standard	DI Water	200
100mg/L Secondary Intermediate Standard	100	5.0 mL	4000 mg/L	4000mg/L Secondary Stock Standard	DI Water	200
Calibration and QC Standards						
Calibration Std 1	0.00	0.00 mL	-	-	DI Water	100
Calibration Std 2	0.10	0.10 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
Calibration Std 3	0.25	0.25 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
Calibration Std 4	0.50	0.50 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
Calibration Std 5	1.00	1.00 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
Calibration Std 6	2.00	2.00 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
Calibration Std 7	5.00	5.00 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
Calibration Std 8	10.0	10.0 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
ICV	5.00	1.00 mL	100 mg/L	100mg/L Secondary Intermediate Standard	DI Water	20
CCV	5.00	5.00 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
ICB/CCB	0.00	0.00 mL	-	-	DI Water	100
RLVS	0.10	0.10 mL	100 mg/L	100mg/L Primary Intermediate Std.	DI Water	100
MB	0.00	0.00 mL	-	-	DI Water	20
LCS	5.00	1.00 mL	100 mg/L	100mg/L Secondary Intermediate Standard	DI Water	20
MS/MSD	5.00	1.00 mL	100 mg/L	100mg/L Secondary Intermediate Standard	Analytical Sample	20

Table B. QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**TOC**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples. A LCSD is required if MS/MSD is not performed.
Matrix Spike / Matrix Spike Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.
Initial Calibration	Minimum of 5 standards plus blank. Must be performed every time before samples are analyzed.
RLVS	After the calibration and monthly at minimum.
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. CCV – analyzed after every 10 samples.
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. CCB – analyzed after every CCV pair.

Table C. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**TOC If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
RLVS standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 20 times the method blank contamination level is 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 concentration of the spike is less than 25% of the concentration of the parent, dilute parent to a level that the subsequent spike concentration will be greater than 25% the parent and so the parent concentration is above the PQL. Rerun the parent, MS, and MSD at this new dilution. If the MS and/or MSD fails at the diluted concentration, appropriately flag the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately flag the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate precision data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB the samples do not have to be reanalyzed.

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STANDARD OPERATING PROCEDURE

Total Cyanide using Micro-Distillation and SmartChem

Reference Method: SW846 9012A and EPA 335.4

SOP NUMBER:	S-GB-I-064-REV.00
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	First Issue

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 1/21/10

Kate Grams, Laboratory Quality Manager — Date: 1/20/10

Chad Rusch, Department Manager — Date: 01/19/2010

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

This Standard Operating Procedure (SOP) describes the analyses of Total Cyanide using Micro-Distillation and Analyzed by SmartChem Discrete analyzer using methods SW846 9012A and EPA Method 335.4.

2. Scope and Application

2.1 This SOP is applicable the analysis of Cyanide in drinking, ground, surface, and saline waters, domestic and industrial wastes, soils, and TCLP, SPLP, ASTM extracts. The applicable range is MDL to the top standard in the curve. The current Pace Reporting Limit is 20 ug/L. The current MDL can be found in EpicPro.

2.2 This SOP utilizes the SmartChem Discrete analyzer.

3. Summary of Method

3.1 By means of a passive miniature distillation device, MICRO DIST, cyanide in the sample is released by digesting and acidifying cyanide complexes, and converting them to hydrocyanic acid (HCN). The cyanide ion is trapped in a 1.0 M sodium hydroxide absorbing solution, which is diluted to 0.25M solution during distillation. By means of discrete analysis, the 0.25M Noah distillate is converted to cyanogens chloride by reaction with chloramines-T, pyridine and barbituric acid to give a red-colored complex. The absorbance of this complex is measured at 570 nm by measuring the peak area resulting from the sample. The peak area is proportional to the concentration of the cyanide in the sample.

3.2 All samples must be distilled before analysis.

4. Interferences

4.1 Most non-volatile interferences are eliminated or minimized by the distillation procedure. Some of the known interferences are aldehyde, nitrate-nitrite, and oxidizing agents, such as chlorine, thiosulfate, and sulfide.

4.2 Oxidizing agents such as chlorine decompose most cyanide. Test a drop of the sample with potassium iodine-starch paper (KI-starch paper) at time of collection; a blue color indicates need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color changes; then add an additional 0.06 g ascorbic acid per liter of sample.

4.3 False positive results may be obtained by samples that contain Nitrate/or nitrite. Sulfamic acid is added to all samples prior to distillation to eliminate this interference.

4.4 Sulfides adversely affect the procedure by producing hydrogen sulfide during distillation. If a drop of sample on lead acetate paper indicated the presence of sulfide, treat the stabilized sample (pH >12) with bismuth nitrate. Repeat until a drop on the lead acetate paper does not darken. Filter sample to remove precipitate and use filtrate as sample to be distilled.

5. Safety

5.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

5.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of ALL-S-002, *Waste Handling*.

5.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention

6. Definitions

Definitions can be found in Section 10 of the most recent version of the Pace Analytical Services, Inc. Quality Manual.

7. Sample Collection, Preservation and Handling

Table 1. Sample Collection, Preservation and Handling

Matrix	Prep Method	Container(s)	Preservation Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	SW-846 9012A EPA 335.4	250 ml plastic containers	pH >12; NaOH 14 days	On ice 4+/- 2° Celsius	4°C
Solid	SW-846 9012A	glass or plastic containers	None 14 days	On ice 4+/- 2° Celsius	4°C

Reagents and Standards

Table 2. REAGENTS

Reagent	Alias	Concentration	Directions found in:
Potassium Cyanide	Stock Standard	Neat	
Potassium Cyanide	Second Source Stock Standard	Neat	
Potassium Hydroxide		Neat	
Sodium Hydroxide		Neat	
Noah – 0.25 N	Carrier Solution	0.25 N	Table 4
Deionized Water			
Potassium Phosphate, Monobasic, (KH_2PO_4)		Neat	
Sodium dihydrogenphosphate Buffer	Buffer Solution	1 M	Table 4
Chloramine-T hydrate		Neat	
Chloramine-T Reagent	Color Reagent		Table 4
Barbituric Acid		Neat	
Pyridine		Neat	
Hydrochloric Acid		12 M	
Pyridine-Barbituric Acid Reagent	Pyridine-Barbituric Acid Reagent		Table 4
p-dimethylaminobenzalrhodanine	Indicating solution		Table 4
Standard Silver Nitrate Solution		0.0192 N	
Magnesium Chloride Hexahydrate		Neat	
Sulfuric Acid		Concentrated	
Sulfuric Acid/Magnesium Chloride Solution	Releasing Agent	7.11 M H_2SO_4 0.79 M MgCl	Table 4
Nitrate Bismuth		Neat	
Lead Acetate Paper		Neat	
Sulfamic Acid		Neat	
Sea Sand (Washed)*		Neat	

***** Washed with 50% sulfuric acid (v/v) solution and then dried.

Table 3. STANDARDS

Standard	Concentration	Direction found in Section:	Alias
Potassium Cyanide	1000 mg/L	Table 5	Stock Standard
Potassium Cyanide Must be different Lot # than Stock	1000 mg/L	Table 5	Second Source Stock Standard
Level 1 Calibration Standard	0.40 mg/L	Table 5	Cal Std. 1
Level 2 Calibration Standard	0.20 mg/L	Table 5	Cal Std. 2
Level 3 Calibration Standard	0.10 mg/L	Table 5	Cal Std. 3
Level 4 Calibration Standard	0.05 mg/L	Table 5	Cal Std. 4
Level 5 Calibration Standard and RLVS Standard	0.02 mg/L	Table 5	Cal Std. 5
Level 6 Calibration Standard	0.00 mg/L	Table 5	Cal Std. 6
Initial Calibration Verification – 1	0.10 mg/L	Table 5	ICV – 1
Initial Calibration Verification – 2	0.05 mg/L	Table 5	ICV – 2
Continuing Calibration Verification	0.20 mg/L	Table 5	CCV
Working Standard (1)	10 mg/L	Table 5	Working Standard (1)
Secondary Working Standard (1)	100 mg/L	Table 5	Secondary Working Standard (1)
Secondary Working Standard (2)	1.2 mg/L	Table 5	Secondary Working Standard (2)
Matrix Spike/Matrix Spike Duplicate – Liquid	0.10 mg/L	Table 5	MS/MSD-Liquid
Matrix Spike/Matrix Spike Duplicate – Solid	0.10 mg/L	Table 5	MS/MSD-Solid
Laboratory Control Spike/Laboratory Control Spike Duplicate - Liquid	0.10 mg/L	Table 5	LCS/LCSD-Liquid
Laboratory Control Spike/Laboratory Control Spike Duplicate - Solid	0.10 mg/L	Table 5	LCS/LCSD-Solid

Table 4. PREPARATION OF REAGENTS

Standard	Alias	Final Concentration	Directions	Final Volume
NaOH – 0.25 N	Carrier Solution	0.25 N	Weigh 10 g of neat NaOH into ~ 500 ml DI water in 1 L plastic container. Dilute to volume. Prepare fresh monthly.	1000 ml
Sodium dihydrogenphosphate Buffer	Buffer Solution	1.0 M	Weigh 69 g of neat NaH_2PO_4 into ~ 500 ml DI water in 1 L volumetric flask. Add 2.0 mL of concentrated Probe Rinse Solution. Dilute to volume. Shelf life = 1 month.	500 ml
Chloramine-T Reagent	Color Reagent		Weigh 0.4 g of neat Chloramine-T hydrate into ~ 50 ml DI water in 100 ml volumetric flask. Dilute to volume. PREPARE DAILY!	100 ml
Pyridine-Barbituric Acid Reagent	Pyridine-Barbituric Acid Reagent		MAKE UNDER HOOD!!!!!!! Weigh 7.5 g barbituric acid into a 500 mL beaker Add 100 ml DI. Add 37.5 ml pyridine, mix to dissolve Add 7.5 ml conc. HCL. Transfer to 500 mL volumetric flask and dilute to volume. Shelf life = 6 months.	500 ml
p-dimethylaminobenzalrhodanine	Indicator Solution		Weigh 20 mg of neat p-dimethylaminobenzalrhodanine into a 100 ml volumetric flask. Dilute into 100 ml acetone. Shelf life = 1 year.	100 ml
Sulfuric Acid/Magnesium Chloride Solution	Releasing Agent	7.11 M H_2SO_4 0.79 M MgCl	Weigh 110.8 g DI water in beaker. Add 32.2 g Magnesium Chloride Hexahydrate ($MgCl_2.6H_2O$). Slowly add 139 g concentrated H_2SO_4. Transfer to automatic pipette container. Cool. Shelf life = 1 year.	200 g

Table 5. PREPARATION OF STANDARDS

Standard	Alias	Final Concentration	Directions	Final Volume
Potassium Cyanide	Stock Standard.	1000 mg/L	POISON!!!!! Weigh 2.51 g of neat KCN and 2 g of neat KOH into ~900 ml DI water in 1 L volumetric flask. Dilute to volume. Shelf life = 3 month. Standardize against AgNO3. If concentration of stock standard is between 995 – 1005 mg/L then use 1ml of stock solution.	1000 ml
Potassium Cyanide	Second Source Stock Standard	1000 mg/L	POISON!!!!! Weigh 2.51 g of neat second source KCN and 2 g of neat KOH into ~900 ml DI water in 1 L volumetric flask. Dilute to volume. Shelf life = 3 month. Standardize against AgNO3. If concentration of stock standard is between 995 – 1005 mg/L then use 1ml of stock solution.	1000 ml
Working Standard (1)	Working Standard (1)	10 mg/L	Pipette 1.0 ml stock standard into 100 ml volumetric flask. Dilute to volume with 0.25N NaoH. PREPARE DAILY!	100 ml
Working Standard (2)	Working Standard (2)	1.2 mg/L	Pipette 1.2 ml working standard (1) into 100 ml volumetric flask. Dilute to volume with DI. PREPARE DAILY!	100 ml
Secondary Working Standard (1)	Secondary Working Standard (1)	100 mg/L	Pipette 10 mL second source stock standard into 100 ml volumetric flask. Dilute to volume with DI. PREPARE DAILY!	100 ml
Secondary Working Standard (2)	Secondary Working Standard (2)	1.2 mg/L	Pipette 1.2 ml secondary working standard (1) into 100 ml volumetric flask. Dilute to volume with DI. PREPARE DAILY!	100 ml
Level 1 Calibration standard	Cal. Std. 1	0.40 mg/L	Pipette 4.0 mls of working standard (1) into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare monthly.	100 ml
Level 2 Calibration Standard	Cal. Std. 2	0.20 mg/L	Pipette 2.0 mls of working standard (1) into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare monthly.	100ml
Level 3 Calibration Standard	Cal. Std. 3	0.10 mg/L	Pipette 1.0 mls of working standard (1) into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare monthly.	100 ml
Level 4 Calibration Standard	Cal. Std. 3	0.050 mg/L	Pipette 0.50 mls of working standard (1) into 100 ml volumetric flask. Dilute to volume with 0.25N NaoH. Prepare monthly.	100 ml
Level 5 Calibration Standard and RLVS Standard	Cal. Std. 5	0.020 mg/L	Pipette 0.20 mls of working standard (1) into 100 ml volumetric flask. Dilute to volume with 0.25N NaoH. Prepare monthly.	100 ml
Level 6 Calibration Standard	Cal. Std. 6	0.000 mg/L	Use 0.25N NaOH only.	100 ml
Continuing Calibration Verification	CCV – 1	0.20 mg/L	Pipette 2.0 mls of working standard (1) solution into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare monthly.	100 ml
Initial Calibration Verification –1	ICV – 1	0.10 mg/L	Pipette 0.50 mls of secondary working standard (2) solution into sample tube. Dilute to volume with DI. Prepare daily.	6 ml
Initial Calibration Verification –2	ICV – 2	0.050 mg/L	Pipette 0.25 mls of secondary working standard (2) solution into sample tube. Dilute to volume with DI. Prepare daily.	6 ml
Matrix Spike/Matrix Spike Duplicate – Liquid	MS/MSD - Liquid	0.10 mg/L	Pipette 0.50 mls of working standard (2) into sample tubes. Dilute to volume with sample. Prepare daily.	6 ml
Matrix Spike/Matrix Spike Duplicate – Solid	MS/MSD-Solid	0.10 mg/L	Pipette 0.50 mls of working standard (2) into sample tubes. Add solid sample. Dilute to volume with DI. Prepare daily.	6 ml
Laboratory Control Spike/Laboratory Control Spike Duplicate – Liquid	LCS/LCSD-Liquid	0.10 mg/L	Pipette 0.50 mls of working standard (2) into sample tubes. Dilute to volume with DI. Prepare daily.	6 ml
Laboratory Control Spike/Laboratory Control Spike Duplicate – Solid	LCS/LCSD-Solid	0.10 mg/L	Pipette 0.50 mls of working standard (2) into sample tubes. Dilute to volume with DI. Prepare daily.	6 ml

8. Equipment and Supplies

Table 6. EQUIPMENT

Equipment	Manufacturer	Model(s)/Catalog number •
SmartChem	SmartChem	SmartChem
Micro-Distillation	Lachat	Micro Distillation
Top loading Analytical Balance	Mettler	BA310P
1000 µL Pipette	Eppendorf	05-402-76 (Fisher Cat#)
500 µL Pipette	Eppendorf	05-402-72 (Fisher Cat#)
250 µL Pipette	Eppendorf	05-402-69 (Fisher Cat#)
Repipetors (0.5 - 5.0mL)	Barnstead/Thermolyne	13-687-33 (Fisher Cat#)
Vented Hood	Hamilton	
Spoonula (Lab Spoon)	FisherBrand or equivalent	14-375-10 (Fisher Cat#)
Gloves – Heat Resistant		
Cyanide Manifold	Lachat	10204-00-1-A
Interference filter – 570 nm	Lachat	

- Or equivalent

Table 7. Supplies

Supplies	Manufacturer	Catalog number •
Distillation tubes	Lachat	A17001
Culture Tubes 13X100	Fisher	71500 13100
Volumetric Flasks	Fisher	Various
Parafilm	Fisher	
Sea Sand	Fisher	S25-3
• Or equivalent		

9. Calibration and Standardization

9.1 The diluent bottle for this method is filled with the 0.25N NaOH solution.

9.2 Prepare a blank and 5 to 7 standards covering the desired analytical range. Prepare standards by diluting suitable volumes of standard solutions to 100 ml with appropriate sample matrix diluents. In the SmartChem's auto standard preparation mode, the diluents used to prepare the standards is drawn from the diluents bottle.

NOTE: The SmartChem can automatically dilute standards and samples, as required, to bring high concentration standards and samples into the working range of the method. Refer to the SmartChem Operation Manual, Chapter 3, for instructions how to configure the SmartChem Method Parameter File.

9.3 Place manually prepared standards in the standard rack in order of increasing concentration and perform the analysis.

10. Procedure

10.1 Distillation Procedure

NOTE: Document all sample volumes, standards and reagents used in the distillation on the prep log.

This section of the SOP details the steps in distilling the samples before analysis. Samples must be checked for sulfides before distilling. If sulfides are present and the samples treated with bismuth nitrate, the entire calibration curve must be similarly treated and distilled. Refer to section 4.4 for procedure. In the following procedure, **D** and **M** refer to the marks on the collector tube. **D** means "distillation" end, and **M** means "measuring" end.

10.1.1 Set the controller to 120°C, allow the heater to warm up. The operating temperature of the distillation block needs to be verified annually.

10.1.2 Label tubes with the **M** end up, place as many properly labeled collector tubes as you have samples into the collector tube rack.

10.1.3 For liquid samples, place a drop of sample on lead acetate paper. If the paper darkens this indicate the presence of sulfide. Treat the stabilized sample (pH >12) with bismuth Nitrate. Repeat until a drop on the lead acetate paper does not darken. Filter sample to remove precipitate and use filtrate as sample to be distilled. Place 6 ml of sample into each properly labeled sample tube using a calibrated automatic pipette. Add ~ 0.1 g sulfamic acid.

NOTE: If samples contain and are treated for sulfide, the entire calibration curve must also be similarly treated and distilled along with the samples.

10.1.4 For soil samples, weigh out 0.20 g of soil or sludge to the nearest 100th on a calibrated balance and dilute to 6 ml with deionized water tube with a calibrated automatic pipette into each properly labeled sample tube. Add ~ 0.1 g sulfamic acid.

10.1.5 For liquid samples, prepare one method blank by adding 6 mls of DI water to properly labeled sample tube using a calibrated automatic pipette. Add ~ 0.1 g sulfamic acid.

10.1.6 For soil samples, prepare one method blank by weighing out 0.20 g of washed sea sand to the nearest 100th on a calibrated balance and dilute to 6 ml with deionized water tube with a calibrated automatic pipette into a properly labeled sample tube. Add ~ 0.1 g sulfamic acid.

10.1.7 For liquid samples, prepare one LCS (and one LCSD if needed/requested) by pipetting 6.0 ml of deionized water, ~ 0.1 g sulfamic acid, and 0.50 ml of 1.2 mg/L CN standard into two properly labeled sample tubes using a calibrated automatic pipette. See Table 5.

10.1.8 For soil samples, prepare one LCS (and one LCSD if needed/requested) by weighing out 0.20 g of washed sea sand to the nearest 100th on a calibrated balance. Bring to 6 ml volume with deionized water. Add ~ 0.1 g sulfamic acid and 0.50 ml of 1.2 mg/L CN standard using a calibrated automatic pipette into each properly labeled sample tube. See Table 5.

10.1.9 For liquid samples, select one parent sample and prepare one MS/MSD pair per 10 samples by adding 6.0 ml of sample, ~ 0.1 g sulfamic acid, and 0.50 ml of 1.2 mg/L CN standard into two properly labeled sample tubes using a calibrated automatic pipette. See Table 5.

10.1.10 For soil samples, select one parent sample and prepare one MS/MSD pair by weighing out 0.20 g of sample to the nearest 100th on a calibrated balance. Bring to 6 ml volume with deionized water. Add ~ 0.1 g sulfamic acid and 0.50 ml of 1.2 mg/L CN standard using a calibrated automatic pipette into each properly labeled sample tube. See Table 5.

10.1.11 Using a calibrated pipette, add 0.75 ml of 7.111 M sulfuric acid / 0.79 M magnesium chloride solution to the sample tube using the supplied automatic pipette.

10.1.12 Immediately push the **D** end of a Cyanide-1 collector tube over the open end of each sample tube to start the seal.

10.1.13 Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing, the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.

10.1.14 The pressing motion should be a smooth constant pressure, which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.

10.1.15 Put on heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.

10.1.16 Set the timer for 30 minutes.

10.1.17 When 30 minutes is up, remove the first tube, use heat-resistant gloves, from the block and immediately pull its sample tube using a downward twisting motion as opposed to a sideways motion. You must remove the sample tube within 4 seconds of removing it for the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution.

10.1.18 Invert each collector tube and place it into the collector tube rack, now with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.

10.1.19 Hold collector tube horizontally and rinse its walls with the distillate in order to homogenize it. Roll tube in order to collect all droplets from the walls of the tube. Return the tube to an upright position so the **D** end is up.

10.1.20 With the **D** end up, break the collector tube in half by pulling the **D** end hard toward yourself to break it, the twisting and tearing off the **D** end. Discard the **D** end.

10.1.21 In the **M** end of the tube dilute to the 6.0 ml mark with DI water. This results in the original sample volume, but now in 0.25 M NaOH. Pour in culture tube.

10.1.22 Parafilm if sample determination is not done immediately.

10.2 Basic System Operation - Analytical

This portion of the SOP is designed to allow the user to set up and run a method, print a sample report, then shut down using the more basic software functions. For a more detailed explanation of the many other options, the user should refer to the SmartChem Software Reference Manual. It is assumed that a method program has already been created for the chemistries you wish to run.

10.2.1 Turn on computer with ON/OFF button. Turn on printer. The power to the monitor, autosampler and reaction module is controlled with a multi-outlet noise filter and surge suppresser located behind the reaction module. Turn the red switch to “ON”.

10.2.2 Log into the Network with your Login name and password. Windows program will be activated. Program Manager screen will appear. Click twice on SmartChem Instruments. To encourage GALP (Good Automated Laboratory Practice), the operator "Log-in" every time with name and password. Enter your assigned name and password in the correct case in the spaces indicated and then click on the **'OK'** button.

10.2.3 SmartChem Start-up procedure.

10.1.3.1 Go to Sample Entry

10.1.3.2 Double click on the Method Run.

10.1.3.3 Enter the # of samples in the Accept Samples Box.

10.1.3.4 Click on Accept Samples.

10.1.3.5 Enter samples ID's.

10.1.3.6 Review the Run Planner and click Save (Red disk in upper right corner.)

10.1.3.7 Go to System Monitor.

10.1.3.8 Click on the Run Planner (the page in the upper left hand corner) that was created.

10.1.3.9 Verify that this is the correct run.

10.1.3.10 Fill standard, check standard and blank vials with proper solutions. Fill sample tubes with proper samples.

10.1.3.11 Check the probe rinse, DI water, and Cleaning Solution bottles. **NOTE:** When filling these solutions, pour slowly to minimize foaming. If filling is required while a run is in progress, do not lift siphon tube above the liquid surface. Insert a long stem funnel through the open port in the reservoir cap and fill the container slowly with the respective solution to minimize foaming.

10.1.3.12 Click Start.

10.1.3.13 Click OK to run a water baseline (WBL). Cancel to skip the WBL. A WBL should be run at least once each day.

10.1.4 Samples that have results greater than the highest standard in the calibration curve need to be diluted appropriately so the result falls within the calibration curve, and reanalyzed. Document all dilutions appropriately.

10.1.5 Document all standards and reagents used in analysis in raw data. Sign and date raw data.

NOTE: Any daily or periodic maintenance must be recorded in the instrument daily logbook.

10.2 SHUTDOWN:

10.2.3 Click on **EXIT** icon

10.2.4 Turn off computer, printer and switch on surge suppresser.

10.3 Calculations:

10.3.3 The instrument provides calculated sample results in ug/L. Calculations are only necessary if a dilution was used.

Liquid Calculation

Raw Data Value (ug/L) x Dilution Factor / 1000 = Cyanide (mg/L)

Soil Calculation

$$\frac{\text{Raw Data Value (ug/L) X 0.006 L X Dilution Factor}}{\text{(0.00020 Kg x \% solids in decimal form x 1000)}} = \text{Cyanide(mg/Kg)}$$

11 Quality Control

11.1 Refer to the most current version of SOP GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

11.2 **Initial Calibration Verification (ICV)**

11.2.1 The ICV must be analyzed before samples.

11.2.2 The ICV is distilled with the batch.

11.2.3 Concentration must be within $\pm$ 10% of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified.

11.2.4 If the ICV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

11.2.5 The lot number of the Potassium Cyanide Standard used to make the ICV must be different from that of the calibration curve standards.

11.3 **Continuing Calibration Verification (CCV)**

11.3.1 The CCV is analyzed after every 10 samples.

11.3.2 The CCV is not distilled with the batch.

11.3.3 Concentration must be within $\pm$ 10% of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified. If the reset CCV fails, recalibrate and reanalyze all samples back to the last acceptable CCV.

11.3.4 If the CCV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

11.3.5 The lot number of the Potassium Cyanide Standard used to make the CCV must be different from that of the ICV.

11.4 **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)**

11.4.1 The ICB must be analyzed after the ICV and before samples. The CCB must be analyzed after the CCV and before samples.

11.4.2 The ICB and CCB are not distilled with the batch.

11.4.3 The absolute value must be < PRL. When measurements are above the PRL, terminate analysis, correct the problem, and the calibration re-verified. If the reset of the I/CCB fails, recalibrate and reanalyze all analytical samples analyzed since the last compliant calibration blank.

11.4.4 If the sample concentration is greater than ten times the concentration in the ICB or CCB, the samples do not need to be reanalyzed.

11.5 **Reporting Limit Verification Standard (RLVS)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

11.6 **Method Blank (MB)**

11.6.1 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. Must be distilled with batch.

11.6.2 The absolute value must be < PRL.

11.6.2.1 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.

11.6.2.2 If the analyte is detected in the method blank, method blank criteria is evaluated and flagged with an appropriate qualifier to the MDL when MDL reporting is required: qualify with an appropriate qualifier sample results 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.

11.7 **Laboratory Control Sample (LCS)**

11.7.1 The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples. A Laboratory Control Spike Duplicate (LCSD) must be analyzed if there is insufficient sample volume to perform a matrix spike/matrix spike duplicate or if the client requests one. Must be distilled with batch.

11.7.2 Concentration must be within $\pm$ 10% of the true value for waters. For soils, the concentration must be within calculated in –house limits or default limits of $\pm$ 20%.

11.7.2.1 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.

11.7.2.2 If no errors are found and sufficient sample is available, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still out side 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.

11.7.2.3 If sufficient sample volume is not available, report the sample data with an appropriate (L) qualifier flag on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

11.7.3 When an LCSD is performed, the precision between the LCS and LCSD must a be =/< 20% RPD.

11.7.3.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

11.7.3.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

11.8 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

11.8.1 One pair per up to 10 environmental samples or 10% frequency whichever is more frequent. Must be distilled with batch.

11.8.2 Both QC samples must be calculated for accuracy and precision.

11.8.3 Concentration must be within $\pm$ 10% of the true value for waters. For soils, the concentration must be within calculated in –house limits or default limits of $\pm$ 20%.

11.8.3.1 If the four times the concentration of the spike is less than the level of the parent, accuracy need not be calculated.

11.8.3.2 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

11.8.3.3 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

11.8.4 The precision between the MS and MSD must a be =/< 20% RPD.

11.8.4.1 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.

11.8.4.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

11.8.5 The parent sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD.

11.8.6 The lot number of the Potassium Cyanide used to make the CCV is different for the MS/MSD.

11.9 The appropriate leach blank must be distilled and analyzed along with the appropriate samples as leached on the same leach date. The absolute value must be < PRL. When measurements are above the PRL, terminate analysis, investigate and correct the problem, verify the calibration, and reanalyze all analytical samples analyzed.

11.10 When preparation of a sample exceeds 14 days past the time of collection, notify the project manager before proceeding. If a sample is run past 14 days after collection, flag the result with an appropriate data qualifier.

11.11 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with an appropriate data qualifier.

11.12 See attachments Table A and Table B for a summary of QC.

12 Method Performance

12.2 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

12.2.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

12.2.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

12.2.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

12.2.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

13 Pollution Prevention and Waste Management

13.2 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

13.3 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

13.4 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of ALL-S-002, *Waste Handling*.

13.5 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

14 References

14.2 SmartChem 200 Method 280-400E (Rev: April 2007)

14.3 Micro Dist Method Cyanide-1, Lachat Instruments, October 12, 2000

14.4 SW846 9012A, Revision 1.0, December 1996

14.5 EPA 600 R-93-100, Revised August 1993, Method 335.4

14.6 Standard Methods for the Examination of Water and Wastewater, 18th Edition, p. 4-18, Methods 4500-CN^- A, B, C, and D (1992)

15 Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

15.2 Table A: **QUALITY CONTOL**

15.3 Table B: **ANALYST/TECHNICIAN DATA ASSESSMENT**

15.4 CN bench sheet example

15.5 CN prep sheet example

15.6 Flowchart

16 Revisions

Revision Number	Reason for Change	Date
S-GB-I-064.Rev.0	First Issue.	19Jan2010

Table A. QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**EPA 335.4 SW846 9012A**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent. Must be distilled with batch.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. A LCSD is required if MS/MSD is not performed. Must be distilled with batch.
Matrix Spike / Matrix Spike Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent. Must be distilled with batch.
Initial Calibration	Minimum of 5 standards plus blank. Must be performed every time before samples are analyzed.
RLVS Standard	After the calibration
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. **The ICV is distilled with batch.** CCV – analyzed after every 10 samples. **The CCV is not distilled with batch.**
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. **The ICB is not distilled with batch.** CCB – analyzed after every CCV pair. **The CCB is not distilled with batch.**
Leach Blank	The appropriate leach blank must be distilled and analyzed along with the appropriate samples as leached on the same leach date.

Table B. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**If these conditions are not achieved ⇒**
Method Blank and Leach Blanks	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
RLVS standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 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 concentration of the spike is less than 25% of the concentration of the parent, dilute parent to a level that the subsequent spike concentration will be greater than 25% the parent and so the parent concentration is above the EQL. Rerun the parent, MS, and MSD at this new dilution. If the MS and/or MSD fails at the diluted concentration, use appropriate qualifier to flag the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, use appropriate qualifier to flag the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate precision data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB the samples do not have to be reanalyzed.

CN Prep Sheet Example

Pace Analytical Services, Inc. Distillation Log

Prep WS#: 17852
Prep Date: 1/25/07
Run Date: 1/25/07
Analyst: DAW

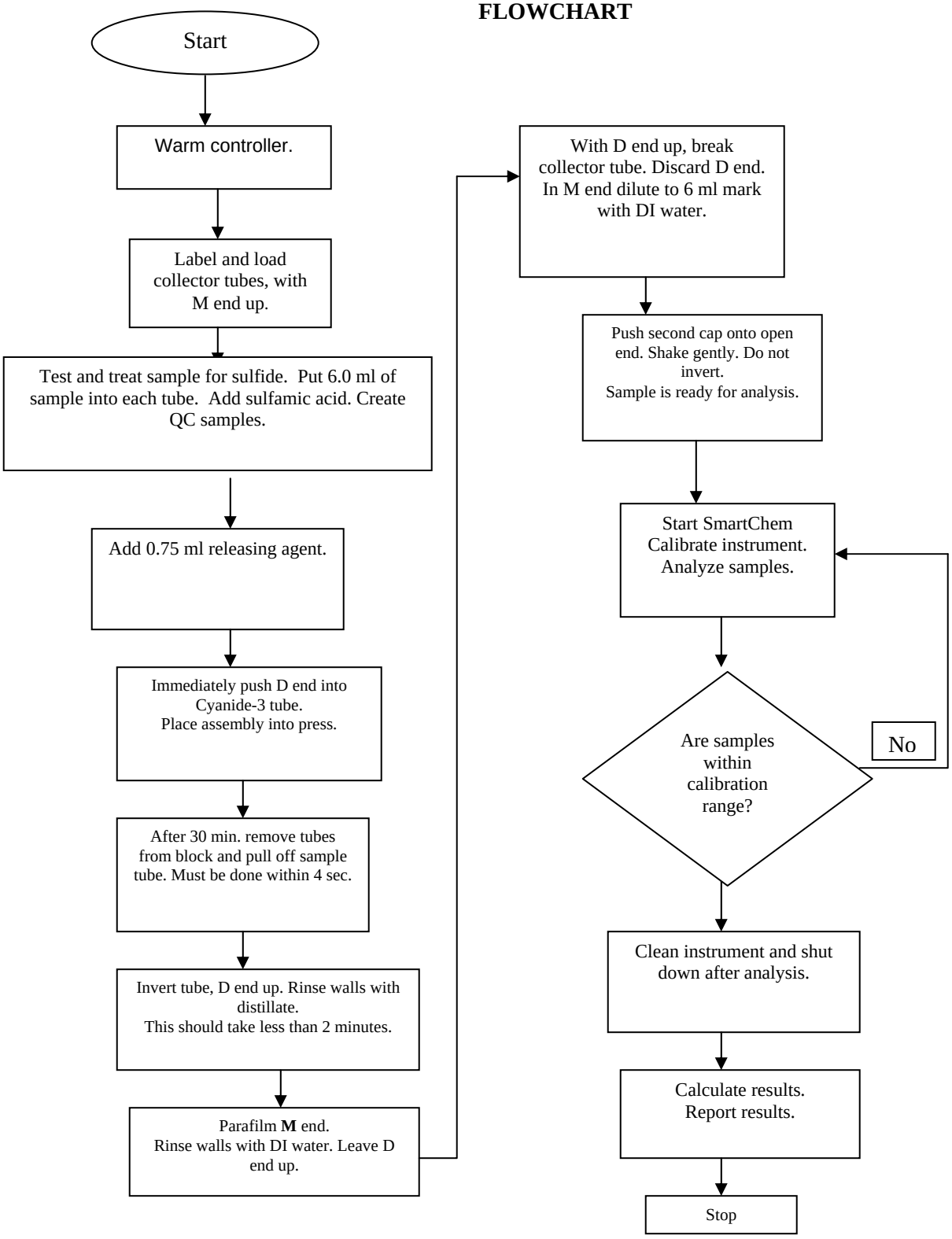
Total Cyanide
EPA 335.4

	Matrix	Sample #	Pre-Dilution	Initial Vol/ Final Vol	Spike Log Number		Matrix	Sample #	Pre-Dilution	Initial Vol/ Final Vol	Spike Log Number
1	W	ICV 100		6ml/6ml		31	D	880326-002MSD		6ml/6ml	
2	W	ICV 50		6ml/6ml		32					
3	W	ICB		6ml/6ml		33					
4	W	WCG2101-077MB		6ml/6ml		34					
5	W	WCG2101-077MBLCS		6ml/6ml		35					
6	W	880226-015		6ml/6ml		36					
7	W	880226-015MS		6ml/6ml		37					
8	W	880226-015MSD		6ml/6ml		38					
9	W	880237-002		6ml/6ml		39					
10	W	880237-004		6ml/6ml		40					
11	W	880253-002		6ml/6ml		41					
12	W	880321-004		6ml/6ml		42					
13	W	880321-006		6ml/6ml		43					
14	W	880321-011		6ml/6ml		44					
15	W	880353-001		6ml/6ml		45					
16	W	880353-002		6ml/6ml		46					
17	W	880353-003		6ml/6ml		47					
18	W	880353-004		6ml/6ml		48					
19	W	880353-005		6ml/6ml		49					
20	W	880353-006		6ml/6ml		50					
21	W	880373-002		6ml/6ml		51					
22	W	880373-005		6ml/6ml		52					
23	W	880373-006		6ml/6ml		53					
24	W	880373-007		6ml/6ml		54					
25	W	880397-001		6ml/6ml		55					
26	W	880397-001MS		6ml/6ml		56					
27	W	880397-001MSD		6ml/6ml		57					
28	D	880326-001		6ml/6ml		58					
29	D	880326-002		6ml/6ml		59					
30	D	880326-002MS		6ml/6ml		60					

There could be three kinds of dilutions in this run: pre, prep, analytical. Pre-dilutions are done before the sample is prepped. Prep-dilutions are done during the prep step. Analytical-dilutions are done at the instrument. The Pre-dilutions are noted on the prep log. The Prep-dilutions are noted in the Initial/Final Volumes on the Prep Log. On the raw data, the dilution noted is the Pre-dilution X the Analytical-dilution. The raw data dilution does not include the Prep-dilution.

Comments: _____

FLOWCHART


Start
Warm controller.
Label and load collector tubes, with M end up.
Test and treat sample for sulfide. Put 6.0 ml of sample into each tube. Add sulfamic acid. Create QC samples.
Add 0.75 ml releasing agent.
Immediately push D end into Cyanide-3 tube.
Place assembly into press.
After 30 min. remove tubes from block and pull off sample tube. Must be done within 4 sec.
Invert tube, D end up. Rinse walls with distillate.
This should take less than 2 minutes.
Parafilm **M** end.
Rinse walls with DI water. Leave D end up.
With D end up, break collector tube. Discard D end. In M end dilute to 6 ml mark with DI water.
Push second cap onto open end. Shake gently. Do not invert.
Sample is ready for analysis.
Start SmartChem
Calibrate instrument.
Analyze samples.
Are samples within calibration range?
No
Clean instrument and shut down after analysis.
Calculate results.
Report results.
Stop

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STANDARD OPERATING PROCEDURE

TCLP - Toxicity Characteristic Leaching Procedure EPA SW846 1311
SPLP – Synthetic Precipitation Leaching Procedure EPA SW846 1312
ASTM - ASTM D 3987-85

Reference Methods: EPA SW846 1311, EPA SW846 1312, ASTM D 3987-85

SOP NUMBER:	S-GB-I-067-REV.00
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-025-REV.02, S-GB-I-057-REV.00, S-GB-I-058-REV.00

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 7/8/10

Kate Grams, Laboratory Quality Manager — Date: 7/7/10

Chad Rusch, Department Manager — Date: 7/7/10

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 Standard Operating Procedure (SOP) is to provide a laboratory specific procedure for extracting organics and inorganic compounds from samples using and meeting the requirements specified in ASTM D 3987-85 and also EPA SW846 1311(TCLP) and EPA SW846 1312(SPLP). This SOP covers bottle extraction and zero headspace extraction (ZHE).

2. Scope and Application

2.1 The policies and procedures contained in this SOP are applicable to all personnel involved in the preparation and analysis of samples using the leach methods within this facility.

2.2 The leach methods are designed to determine the mobility of inorganic and organic contaminants present in liquid, solid, and multi-phase wastes.

2.3 Liquid wastes (below 0.5% dry solid material), after filtration through a 0.6 to 0.8 micron glass filter, are defined as the TCLP or SPLP extract. ASTM liquid samples are tumbled at a 1:20 ratio with reagent water.

2.4 For TCLP and SPLP wastes containing greater than 0.5% solids, the liquid portion, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted (tumbled) with an amount of extraction fluid equal to 20 times the weight of the solid phase. All ASTM samples are tumbled at a 1:20 ratio with reagent water.

2.5 Bottle extractors are used for non-volatile analytes and a special ZHE extraction device is used to tumble samples for volatile analysis.

2.6 For multi-phase samples being leached by TCLP and SPLP, the initial liquid phase and the solid phase extracted water are combined and analyzed as one sample. If the wastes are not compatible, they are analyzed separately and results are mathematically combined to give a volume-weighted average concentration. ASTM samples are homogenized and tumbled with out being separated.

3. Summary of Method

An aliquot of waste sample is tumbled in a sealed container containing extraction fluid to leach out compounds of interest from the sample. The extract then becomes the sample to analyze by a determinative method (volatiles, semi-volatiles, metals, wet chemistry).

4. Interferences

No significant interferences are listed for the preparation of the TCLP extracts. Potential interferences during analyses are discussed in the individual determinative method SOP's.

5. Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training.

5.3 Analysts should take necessary safety precautions when handling chemicals and samples. Proper personal protective equipment may include safety gloves, lab coats, and safety glasses or goggles. Analysts should be familiar with the MSDS sheets for all chemicals and reagents they use for this procedure and the location of the MSDS sheets within the laboratory. Any questions or concerns should be taken to the laboratory Chemical Hygiene/Safety Officer.

6. Definitions

Refer to the most current version section 10 of the Pace Quality Manual.

7. Sample Collection, Preservation and Handling

7.1 Samples are stored separately from all standards, reagents and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

7.2 Liquid samples are collected in 1 Liter amber bottles with Teflon lined lids. Four to six liters may be needed for analysis. Preservations are not added to samples. After collection, samples are cooled to 4°- 6° C or lower, immediately after collection.

7.3 Solid samples are collected in 4 ounce wide-mouth bottles. At least 200g may be needed to perform the procedure. After collection, samples are cooled to 4°- 6° C or lower, immediately after collection.

7.4 Leach extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH <2, unless precipitation occurs. Extract should be preserved for other analytes according to the guidance given in the individual analysis method. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere to prevent losses.

7.5 TCLP, SPLP, ASTM extraction must be performed within the following times:

Sample Maximum Holding Times – Days				
	From: Field Collection **To:** TCLP Extraction	**From:** Leach Extraction **To:** Preparative Extraction/digest	**From:** Preparative Extraction/digest **To:** Determinative Analysis	**Total Elapsed Time**
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals	180	NA	180	360
Volatiles	14	NA	14	28
Cyanide	14	NA	14	28
Total Phenol (Wet Chem)	28	NA	28	56

7.6 Preservatives shall not be added to samples before tumbling.

7.7 TCLP extracts for metallic analyte determinations must be acidified with nitric acid to a pH of < 2.

7.8 Extracts must be preserved and stored for analytes according to the guidance given in the individual analysis methods.

8. Equipment and Supplies - See Tables A and B for summary

9. Reagents and Standards - See Tables C and D for summary

9.1 **TCLP - Extraction fluid:** The pH of these fluids should be checked and recorded before each use; if the pH is outside of listed specifications or if impurities are found, the fluid should be discarded and remade. Shelf life = 1 week.

9.1.1 **Extraction fluid #1**: In 20 L carboy, add 20 L of DI water. Slowly add 114 ml glacial HOAc. Slowly add 130 ml 10 N NaOH. Cap carboy and mix well. The pH of this fluid must be 4.93 +/- 0.05 standard units. If the pH is not within 4.93 +/- 0.05 standard units, the fluid must be discarded and remade.

9.1.2 **Extraction fluid #2**: In 12 L carboy, add 10 L DI water. Slowly add 67.4 ml glacial HOAc dilute to volume. The pH of this fluid must be 2.88 +/- 0.05 standard units. If the pH is not within 2.88 +/- 0.05 standard units, the fluid must be discarded and remade.

9.2 **SPLP - Extraction fluid:** The pH of these fluids should be checked and recorded before each use; if the pH is outside of listed specifications or if impurities are found, the fluid should be discarded and remade. Shelf life = 1 week.

9.2.1 **Extraction fluid #1**: In 20 L car buoy, add DI water. Add 60/40 weight percent mixture of sulfuric and nitric acids to DI water until pH is 4.2 ± 0.05 standard units. If the pH is not within 4.2 ± 0.05 standard units, the fluid must be discarded and remade. The fluid is used to determine the leach ability of soil from a site that is east of the Mississippi River, and the leach ability of wastes and wastewaters.

9.2.2 **Extraction fluid #2**: In 20 L car buoy, add DI water. Add 60/40 weight percent mixture of sulfuric and nitric acids to DI water until pH is 5.0 ± 0.05 standard units. If the pH is not within 5.0 ± 0.05 standard units, the fluid must be discarded and remade. The fluid is used to determine the leach ability of soil from a site that is west of the Mississippi River.

9.2.3 **Extraction fluid #3**: Reagent water. The pH of this fluid must be within ASTM Type II water of 5.5 SU to 7.5 SU. This fluid is reagent water and is used to determine cyanide and volatiles leach ability.

9.3 **ASTM – Extraction fluid is DI reagent water**

9.4 Analytical Standards are not applicable to this SOP.

10. Calibration

10.1 pH Meter shall be calibrated and verified as per directions in the most current version of SOP S-ALL-I-015 *Measurement of pH in Water, Soil, and Waste*.

10.2 The balance calibration shall be verified as per the most current version of SOP S-ALL-Q-013 *Support Equipment*.

11. ASTM Procedure

11.1 ASTM Sample Preparation

11.1.1 Percent moisture is determined on each sample by Sample Management staff as per SOP S-GB-C-008, *Measurement of Percent Moisture in Soils and Solids*.

11.1.2 For free-flowing particulate solid wastes, obtain a sample of the appropriate size required by thoroughly mixing, then quartering the sample as per SOP S-ALL-Q-21, *Sample Homogenization and Sub-Sampling*. A minimum sample size of 70 grams is recommended for each extraction; a sample size of 100 grams is commonly used in this method.

11.1.3 For field-cored solid wastes or castings, cut a representative section weighing approximately 100 grams for each testing. Shape the sample so the leaching solution will cover the material.

11.1.4 For fluid solid samples, mix thoroughly in a manner that does not incorporate air to assure uniformity before withdrawing a 100 gram sample for testing.

11.2 ASTM Extraction Procedure:

11.2.1 Weigh out 100 grams of solid waste into a 2-liter extraction bottle. Record the weight to the nearest 0.5 gram on the data sheet. If weights other than 100 grams are used, note on the data sheet.

11.2.2 Add to the container a volume of reagent water (in mL) to 20 times the weight in grams of the sample. Use this dilution ratio for each sample. (Example: 100 grams sample = 2000 mL water).

11.2.3 Record the sample description, particle size, sample weight used for extraction, and amount of reagent water used in the Extraction Bench sheet Logbook.

11.2.4 Rotate at 29 +/- 2 rpm in rotating leaching apparatus continuously for 18 +/- 0.25 hours at 18 to 27ºC. Record the start and stop temperature in logbook. If the temperature is outside of specifications during the tumbling process, the temperature needs to be bought within control, and the entire batch needs to be re-prepared. Record start and stop time in logbook

11.2.5 Open container and observe sample. Record any physical changes in the sample and leaching solution.

11.2.6 Let the sample settle for 5 minutes. Separate the bulk of the aqueous phase from the solid phase by decantation, or filtration through a course filter paper.

11.2.7 Vacuum filter the liquid through s 0.45-um filter into a 2-liter flask.

11.2.8 Measure the pH (record as final pH) of the extract immediately and preserve the volume of extract needed for the testing program as per Section 7.0. Record pH to the nearest 0.01 – pH units.

11.2.9 Refrigerate the extracts as soon as possible as 0º to $\leq$6ºC.

11.3 ASTM ZHE Vessel Preparation if Volatile Analysis is Required.

11.3.1 When the blank vessel is completely assembled, it needs to be pressurized to eliminate all headspace before transferring the buffer solution. The vessel is connected to nitrogen by using the quick-disconnect Swagelok fitting found at the bottom of the vessel. When the quick-disconnect Swagelok is fitted, a distinct "click" should be heard. The two-way valve on the vessel's top plate is opened, and the vent relief valve on the vessel's bottom plate is closed. Approximately 10-20 psi of pressure is applied to push the piston completely to the top of the vessel, causing the vessel to "jump" slightly. The pressure is released in the vessel by venting the relief valve on the vessel's bottom plate.

11.3.2 Fill the transfer vessel to its capacity with DI water. The vessel's top plate is put on with the two-way valve opened. All the pressure release valves should be closed at this point. The transfer line is then attached tightly to the transfer vessel, and connected to the nitrogen tank. Pressure is applied, being careful not to exceed 50 psi. (Pressure greater than 50 psi may cause the glass fiber filter to break).

11.3.3 Slowly open the two-way valve, allowing the DI water to flow through the transfer line, thus eliminating the air that is in the line. Close the valve when the solution has reached the end of the transfer line, and loosely connect it to the blank vessel. Open both valves (two-way and vent relief) on the blank vessel, and open the two-way valve on the transfer vessel. Tighten the transfer line's connection to the blank vessel once you see the solution begin to leak. This prevents the addition of headspace to the blank vessel.

11.3.4 When there is no more flow through the transfer line, close the two-way valve on the blank vessel. Release the pressure on the transfer vessel. Pressurize to 40 psi and invert the blank vessel three times. Open the two-way valve slowly to allow any headspace to escape. As soon as liquid begins to elute without bubbles, close the top valve and shut off the nitrogen.

11.4 ASTM Solid Wastes Sample Preparation if Volatile Analysis is Required.

11.4.1 The sample is weighed out to 25 ± 0.5 grams, and is added to an empty ZHE vessel. The weight of the sample and the vessel number are recorded in the ZHE Extraction Bench sheet logbook. The top plate of the vessel is closed to an even seal, and the same procedure used for the blank in step 11.3 is followed.

11.4.2 Add the DI water to the ZHE sample vessel via the transfer vessel according to the procedure described in the ZHE blank section. The sample vessel is now ready to be tumbled after being pressurized to 5-10 psi.

Note: The total number of vessels per tumbler is eight. Assemble empty (dummy vessels) to fill any vacant spots in the tumbler for odd sample batches. Remove the legs from the vessels, if necessary, before tumbling. If the tumbler makes any bumping or banging noises when turned on, stop the tumbling and re-adjust the vessels.

Note: When preparing actual samples, always vent into a hood. At a pressure of 5-10 psi, all samples and the blank are placed on the tumbler and rotated for 18 hours +/- 0.25 hours at 29 ± 2 RPM and 18º to 27ºC. Record initiated and completed times and pressures; and temperatures in the ZHE logbook.

11.5 ASTM Extraction, Filtration, and Collection

11.5.1 After the samples have tumbled for 18 $\pm$ 0.25 hours, stop the tumbler and enter the leaching completed time and temperature in the ZHE extraction bench sheet logbook. The legs are re-attached to the ZHE vessels if necessary.

Note: Check the pressure gauges on the ZHE extractors and record in the ZHE logbook. If the ending pressure is less than 5-10 psi, the integrity of the vessel may have been breeched and analytes may have been lost. Check for damaged gaskets or weak or worn fittings. If the ending pressure is less than 5-10 psi, re-prepare and re-tumble affected samples.

11.5.2 Attach a Luer fitted 50 mL syringe to the top of each vessel. Connect each vessel to the Swagelok fitting from the bottom to the nitrogen tank and apply 20-30 psi of pressure. Slowly open the two-way valve on the top plate of the vessel to release the extract into the barrel of the syringe. Close the two-way valve and remove the syringe. Attach a 25 mm, 0.45um nylon filter to the Luer fitted 50 mL syringe. Slowly transfer the extract into a 40 mL glass vial preserved with HCl. Fill three vials per sample.

Note: Add pressure slowly when necessary. There should be no gurgling noises, indicating an influx of air, as volatiles may be lost and the sample would then be invalid. Also, if the filter in the vessel breaks open, the sample becomes invalid. If either of these occurs, the entire extraction for the sample must be repeated.

12. TCLP and SPLP Procedure

12.1 Before leaching for TCLP and SPLP, perform these evaluations on a representative waste sample of at least 100 grams. If there is not at least 100 grams, get client consent before proceeding. **These test samples won't be extracted**, but are used for the preliminary evaluation.

- **Determination of matrix** of the waste sample
- **Determination of percent solids** of the waste sample (Done in Multi-Phase Worksheet if obviously not 100% solids)
- Determination of whether the waste sample contains insignificant solids and therefore, is its own extract after filtration, this is only done on liquid or **multi-phase** samples (< 0.5% (insignificant solids) or ≥ 0.5%.)
- Determination of if there is a need for **particle size reduction**
- Determination of which **extraction fluid** is to be used for the extraction of the waste

12.2 TCLP and SPLP Determination of the matrix of the waste

12.2.1 The matrix of the waste must be determined, recorded, and reported to the analysts receiving the TCLP extracts for matrix spiking purposes. A matrix spike shall be performed for each matrix type.

12.2.2 The matrices are defined as:

12.2.2.1 Aqueous – Matrix that contains less than 0.5% solids.

12.2.2.2 Soil – Matrix consisting of natural ground.

12.2.2.3 Sludge – Matrix consisting of bio solids from a wastewater treatment facility.

12.2.2.4 Oil/Solvent – Matrix consisting of petroleum compounds immiscible with water

12.2.2.5 Other – Matrices that are none of the above, this matrix can include plant material, waste recycling/shredder material, concrete, etc. A matrix spike must be performed on each material determined as "Other".

12.2.3 The extraction analyst must record the matrix for each sample as defined above in the TCLP/SPLP Extraction Bench Sheet Logbook. A copy of this record must accompany the sample extract to the analysts for matrix spiking purposes.

12.3 TCLP and SPLP Percent Solids of Waste – Use Multi-Phase Worksheet if not 100% Solids

12.3.1 **If the waste sample obviously will not yield free liquid during pressure filtration, it is treated as 100% solids. See Particle Size determination in section 12.4.**

12.3.2 If waste is a liquid or a multi-phase sample, separate the phases by filtration as follows:

12.3.2.1 Record the weight of the glass fiber filter and the container that will receive the filtrate. Assemble the filter and filtering assembly.

12.3.2.2 Weigh out representative sample of waste, 100-gram minimum and record weight to 0.1 grams. Quantitatively transfer entire waste sample to filter holder. Spread evenly over surface of filter. Sample should be allowed to reach room temperature, as a refrigerated sample would yield less liquid.

NOTE: If some waste material adheres to container and cannot be transferred into filter holder (>1 percent of sample weight), subtract this residue weight from the original sample weight.

12.3.2.3 Gradually apply pressure of 1-10 psi until air moves through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After 2 minutes at a maximum of 50 psi, the filtration is stopped.

NOTE: Instantaneous application of high pressure can degrade the glass filter and may cause plugging.

12.3.2.4 The material on the filter is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes or paint wastes, contain material that appears to be liquid, but may not filter. The material within the filtration device is defined as a solid. Do not replace the original filter with a fresh one during this determination.

12.3.2.5 Weigh the filled, filtrate container. Determine the weight of the liquid phase by subtracting the original weight of the filtrate container from the total weight of the filled, filtrate container.

12.3.2.6 Determine the weight of the solid phase by subtracting weight of the liquid phase from the total weight of the original sample aliquot.

12.3.2.7 Determine the percent solids using the following formula:

$$\textbf{Percent solids} = \frac{\textbf{weight of solid phase}}{\textbf{total weight of sample}} \times 100$$

12.3.2.8 Evaluation of Percent Solids

12.3.2.8.1 If the percent solid is greater than or equal to 0.5%:

- Proceed to step 12.3.2.9 (determination of percent DRY solids) if the dry weight is low (<2%) and the filter is wet because some of the filtrate is trapped in the filter.
- Proceed to 12.4 (particle size reduction determination) if there is significant percent solid (>2.0%).

12.3.2.8.2 If the percent solid is less than 0.5%, the liquid phase (filtrate) is the TCLP extract: no extraction is needed. Proceed to step 12.6 to filter the sample.

12.3.2.9 Determination of percent DRY solids.

12.3.2.9.1 Dry the filter and solid phase at $100^{o} \pm 20^{o}C$ until two successive weightings yield the same value within ± 1 percent. Record the final weight in logbook.

12.3.2.9.2 Calculate the percent dry solids as follows:

$$\textbf{Percent DRY solids} = \frac{\textbf{(Wt of dry waste + filter) - Wt of filter)}}{\textbf{Initial Wt of waste}} \times \textbf{100}$$

12.3.2.10 Evaluation of percent DRY solids

- If the percent DRY solid is less than 0.5%, the liquid phase (filtrate) is the TCLP extract: no extraction is needed. Proceed to step 12.6 to filter the sample.
- If the percent DRY solids are greater than or equal to 0.5% Proceed to 12.4 (particle size reduction determination)

12.4 TCLP and SPLP Particle Size Determination

12.4.1 Particle size reduction is required unless the solid portion of the sample is capable of passing through a minimum 9.5mm sieve. If it will not pass through, the entire sample must be ground, crushed or cut until it will pass through. Use a fresh representative waste sample for this determination.

12.5 TCLP and SPLP Determination of Appropriate Extraction Fluid

12.5.1 TCLP

12.5.1.1 Extraction Fluid #1 is always used for Volatiles analysis. Do not perform the extraction fluid determination if only ZHE extraction is to be performed. Use a fresh portion of the solid phase of the waste for this evaluation.

12.5.1.2 Weigh out a small sub sample of solid phase. If necessary, reduce the particle size to approximately 1 mm in diameter or less and transfer 5.0 grams into a 150 ml beaker.

12.5.1.3 Add 96.5 mL DI water to the beaker, cover with a watch glass and stir vigorously for 5 minutes using a magnetic stirrer.

12.5.1.4 Measure and record the pH of the sample. If the pH is <5.0 use extraction fluid #1.

12.5.1.5 If the pH is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watch glass, heat to 50°C, and hold at 50°C for 10 minutes.

12.5.1.6 Let solution cool to room temperature.

12.5.1.7 Measure and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

12.5.2 SPLP

12.5.2.1 For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site west of the Mississippi River, extraction fluid #2 should be used.

12.5.2.2 For wastes and wastewater, extraction fluid #1 should be used.

12.5.2.3 For cyanide-containing wastes and/or soils, extraction fluid #3 (reagent water) must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

12.6 TCLP and SPLP Extraction Procedure

12.6.1 If the waste will obviously yield no liquid during filtration, weigh out a representative sub-sample, 100-gram minimum, record weight to nearest 0.1 gram.

12.6.2 If the waste is a liquid or a multi-phase sample, separate the phases as follows:

12.6.2.1 Pre-weigh the container that will receive the filtrate. Assemble the filtering apparatus.

12.6.2.2 If metals analysis is to be performed: Acid-wash the filter with 1 N HNO_3. Do not acid wash filters if wet chemistry or semivolatiles are to be analyzed. Therefore, if you need to leach for metals and wet chemistry or semivolatiles, you'll need to prepare two separate aliquots.

12.6.2.3 Weigh out a representative sub-sample, 100-gram minimum, and record the weight to nearest 0.1 gram.

NOTE: If the sample contains <0.5 percent dry solids, the liquid phase is filtered and considered the extract. Enough sample volume should be filtered so that all necessary analyses can be performed on the filtrate. Likewise, use enough sample with >0.5 percent solids to generate enough solids for a representative extraction.

12.6.2.4 To aid filtration, allow slurries to stand to permit the solids to settle. The liquid phase may be filtered before the solid phase. (Centrifugation may be used to separate wastes that settle slowly.)

12.6.2.5 Quantitatively transfer entire waste sample to filter holder. Spread evenly over surface of filter. Sample should be allowed to reach room temperature if a refrigerated sample would yield less liquid.

NOTE: If some waste material adheres to container and cannot be transferred into filter holder, (>1 percent of sample weight), subtract this residue weight from the original sample weight.

12.6.2.6 Gradually apply pressure of 1-10 psi until air moves through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

NOTE: Instantaneous application of high pressure can degrade the glass filter and may cause plugging. When air begins to move through filter or when the liquid flow ceases at 50 psi, stop the filtration.

12.6.2.7 The material in the filter holder is the solid phase of the waste, and the filtrate is the liquid phase. Make sure there is enough solid phase of the sample to perform the extraction. The liquid phase may be analyzed separately or stored at 4°C and added after extraction to the sample to be rotated depending upon the percent solids of the sample in section.

12.6.2.8 Determine the weight of the solid phase by subtracting the weight of the liquid phase from the sample weight.

12.7 TCLP and SPLP Determine the amount of extraction fluid by using the calculation:

$$\text{Weight of extraction fluid} = \frac{(20)\ X\ (\%\ \text{solids})\ X\ (\text{weight of waste filtered})}{100}$$

12.7.1 Since the density of the buffer solution is very near 1 gram/ml, it is assumed that 500 grams of buffer solution is equivalent to 500 ml of buffer solution.

12.7.2 Check pH of extraction fluids

12.7.3 Slowly add the amount of appropriate extraction fluid to the extraction bottle. Close the bottle tightly and secure in agitation device.

12.7.4 Record the room temperature at the start of the sample rotation. The temperature of the room where the extraction takes place shall be maintained at 23 ± 2°C. The room temperature is monitored continuously during rotation using the Tempscribe and the room thermometer. The room temperature is acceptable if it is within the range of 21-25°C. Record the start and stop temperature in logbook and staple the Tempscribe paper into the logbook.

12.7.5 Rotate at 30 ± 2 rpm for 18 ± 2 hours. Record start and stop time in logbook. The rotation speed of the tumbler must be monitored. On a weekly basis, determine the speed of the tumbler by counting the number of rotations in one minute and record the speed in the monitoring logbook.

NOTE: Pressure may build up within the extraction bottle as agitation continues (e.g. calcium carbonate-containing wastes may evolve gasses such as carbon dioxide). To relieve excess pressure, the bottle may be periodically opened and vented into a hood (e.g. after 15 minutes, 30 minutes, 1 hour, etc.).

12.7.6 Following the 18 ± 2 hours extraction, filter the material in the extraction bottle through a new glass fiber filter. The glass fiber filter may be replaced to aid in filtration. If metals are to be analyzed, the filters should be acid rinsed with 1N HNO_3.

Gradually apply pressure of 1-10 psi until air moves through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

NOTE: Instantaneous application of high pressure can degrade the glass filter and may cause plugging. When air begins to move through filter or when the liquid flow ceases at 50 psi, stop the filtration.

12.7.7 Record the pH of the extract to the nearest 0.01-pH units.

12.7.8 Prepare the Leach extract as follows:

12.7.8.1 The metals extract must be acidified with HNO_3 to pH <2. Metals and Wet Chemistry prep staff digests one Matrix Spike per waste stream. All other aliquots must be stored at 4°C until analysis.

12.7.8.2 If the waste contains no initial liquid phase, the filtered liquid material from 12.7.6 is considered the TCLP extract.

12.7.8.3 For multi-phase samples:

12.7.8.3.1 If separate phases are compatible combine the filtered fluid with the "free liquid" obtained in step 12.6.2.7. This combined liquid is defined as the TCLP extract.

12.7.8.3.2 If the initial phase (free liquid) is not compatible with the filtered liquid from step 12.6.2.7, the liquids may be analyzed separately and the results combined mathematically as follows:

$$\text{Final analyte concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

V_1 = Volume of the first phase, in L
C_2 = Concentration of the analyte in the first phase, mg/L.
V_2 = Volume of the second phase, in L.
C2 = Concentration of the analyte in the second phase, in mg/L.

12.8 TCLP and SPLP ZHE Vessel Preparation

12.8.1 For **volatile analysis**, the ZHE is used (extracts from the ZHE cannot be used for non-volatile analysis). Because the ZHE typically has a maximum capacity of 500mL, and because the amount of extraction fluid must be 20 times the sample weight, the maximum weight of a sample that can be used is 25 grams. A smaller sample may be used but the extraction fluid must still be 20 times the weight of sample used.

12.8.2 Do not allow the waste sample, the liquid phase (if any), or the extract to be exposed for any more time than is necessary.

12.8.3 When the ZHE vessel is completely assembled, it needs to be pressurized to eliminate all headspace before transferring the buffer solution. The vessel is connected to nitrogen by using the quick-disconnect Swagelok fitting found at the bottom of the vessel. When the quick-disconnect Swagelok is fitted, a distinct "click" should be heard. The two-way valve on the vessel's top plate is opened, and the vent relief valve on the vessel's bottom plate is closed. Approximately 10-20 psi of pressure is applied to push the piston completely to the top of the vessel, causing the vessel to "jump" slightly. The pressure is released in the vessel by venting the relief valve on the vessel's bottom plate. Make sure to shut off the Nitrogen source before venting.

12.8.4 Fill the transfer vessel to its capacity with buffer solution. The vessel's top plate is put on with the two-way valve opened. All the pressure release valves should be closed at this point. The transfer line is then attached tightly to the transfer vessel, and connected to the nitrogen tank. Pressure is applied, being careful not to exceed 10 psi.

12.8.5 Slowly open the two-way valve, allowing the buffer solution (fluid #1) to flow through the transfer line, thus eliminating the air that is in the line. Close the valve when the solution has reached the end of the transfer line, and loosely connect it to the ZHE vessel. Open both valves (two-way and vent relief) on the ZHE vessel, and open the two-way valve on the transfer vessel. Tighten the transfer line's connection to the ZHE vessel once you see the solution begin to leak. This prevents the addition of headspace to the ZHE vessel.

12.8.6 When there is no more flow through the transfer line, close the two-way valve on the ZHE vessel. Release the pressure on the transfer vessel. Pressurize to 10 psi and invert the ZHE vessel three times. Open the two-way valve slowly to allow any headspace to escape. As soon as liquid begins to elute without bubbles, close the top valve and shut off the nitrogen.

12.9 TCLP and SPLP Solid Wastes Sample Preparation (ZHE)

12.9.1 After the sample has been processed through steps 12.1 through 12.6, and there is no liquid in the sample, the sample is said to contain 100% solids, by definition. The 100% solids sample requires 500 grams of buffer solution. (Since the density of the buffer solution is very near 1 gram/mL, it is assumed that 500 grams of buffer solution is equivalent to 500 mL of buffer solution).

12.9.2 The sample is weighed out to 25 ± 0.5 grams, and is added to an empty ZHE vessel. The weight of the sample and the vessel number are recorded in the ZHE logbook. The top plate of the vessel is closed to an even seal, and the same procedure used for the blank in step 12.9.1 is followed.

12.10 TCLP and SPLP Liquid Wastes Sample Preparation (ZHE)

12.10.1 For liquid samples, weigh the glass fiber filter, and record the weight in the ZHE Extraction Bench Sheet logbook before the ZHE vessel is assembled. Pour the sample into the vessel and close the top plate tightly. Attach a Luer fitting to the two-way valve on the top plate. Connect a small length of tygon tubing to the Luer fitting.

12.10.2 Pressurize the vessel to 10-20 psi and allow the sample to filter through. Collect the sample in a 40 mL HCL vial. Slowly increase the pressure, by 10-psi increments, up to 50 psi, and hold for 2 minutes. Allow the fluid to collect between pressure increases.

12.10.3 After filtration: Very carefully remove the filter so as not to tear it! Dry the filter in an oven at 80-120° C for 24 hours. When the weight does not vary by more than one percent over a one-hour period, record the last value obtained. Use it to determine the percent dry solids. If the percent dry solids is <0.5%, the fluid eluted into the Tedlar bag is the ZHE extract and no buffer solution is required.

Note: A sample containing particulate matter that immediately clogs the filter during the filtration process, while obviously mostly liquid, is considered to be 100% solids.

12.11 TCLP and SPLP Multi-phase Liquids or Solids Saturated With Liquid

12.11.1 According to SW846 Method 5030 (7.3.3.2.1): "The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula."

12.11.2 Place 25 ± 0.5 grams of sample into a ZHE vessel. Pressurize the vessel 10-20 psi. Any liquid that elutes is collected into a tarred beaker. Slowly increase the pressure, by 10-psi increments, up to 50 psi. Allow the fluid to collect into the beaker between pressure increases. After two minutes at 50 psi, weigh the beaker and record the weight in the ZHE Extraction Bench Sheet logbook.

12.11.3 For multi-phasic wastes, the fluid collected in the beaker is weighed and subtracted from the total sample weight to determine the percent solids.

$$\frac{W_s - W_f}{W_s} \times 100 = \% \text{ Solids}$$

Where:

W_s = Weight of the sample
W_f = Weight of the eluted fluid

If no liquid elutes out of the vessel, the sample is considered to be 100% solids. The set-up can be used--as it is--for the ZHE extraction. Follow the steps in 12.9.

12.11.4 After collecting the fluid portion of the sample, set up the ZHE apparatus - a second time with a sample size computed according to the following formula

$$\frac{25}{\%\ \text{Solids}} \times 100 = \text{Weight of Sample to be extracted (in grams)}$$

For example, if the sample weight equals 25.0 g, with 5.0 g of fluid eluted, the % solids equal 80%. The amount of sample to be extracted would be 31.2 g.

12.11.5 Collect the fluid via pressure filtration, as described in step 11.11, into a tarred Tedlar bag. Record the weight of the fluid in the ZHE Extraction Bench Sheet logbook. Store the fluid 4°C until analysis. Add the buffer solution to the vessel after the fluid has been eluted. Calculate the amount of buffer solution needed is according to the following formula:

$$\frac{20 \times \%\ \text{Solids} \times \text{Weight of Sample}}{100} = \text{Weight of buffer solution}$$

For example, if 31.2 g of sample were added to the vessel, with a % solids value of 80%, the weight of buffer solution needed would be 499 g. Again, since the density of the buffer solution is very near 1 gram/mL, it is assumed that 499 grams of buffer solution is equivalent to 499 mL of buffer solution.)

12.11.6 Add the buffer solution (fluid #1) to the ZHE sample vessel via the transfer vessel according to the procedure described in the ZHE blank section. The sample vessel is now ready to be tumbled.

Note: The total number of vessels per tumbler is eight. Assemble empty (dummy vessels) to fill any vacant spots in the tumbler for odd sample batches. Remove the legs from the vessels, if necessary, before tumbling. If the tumbler makes any bumping or banging noises when turned on, stop the tumbling and re-adjust the vessels.

Note: When preparing actual samples, always vent into a hood. At a pressure of 10 psi, all samples and the blank are placed on the tumbler and rotated for 18 hours (± 2 hours) at 30 ± 2 RPM. Record initiated and completed times and temperatures in the ZHE Extraction Bench Sheet logbook.

12.12 TCLP and SPLP Extraction, Filtration, and Collection

12.12.1 After the samples have tumbled for 18 ± 2 hours, stop the tumbler and enter the leaching completed time, final pressure and temperature in the ZHE Extraction Bench Sheet logbook. The legs are re-attached to the ZHE vessels if necessary.

Note: Check the pressure gauges on the ZHE extractors and record in the ZHE logbook. If the ending pressure is less than 10 psi, the integrity of the vessel may have been breeched and analytes may have been lost. Check for damaged gaskets or weak or worn fittings and redo the ZHE extraction

Attach a Luer fitted 50 mL syringe to the top of each vessel. Connect each vessel to the Swagelok fitting from the bottom to the nitrogen tank and apply 10 psi of pressure. Slowly open the two-way valve on the top plate of the vessel to release the extract into the barrel of the syringe. Close the two-way valve and remove the syringe. Slowly transfer the extract into a 40 mL glass vial preserved with HCl. Fill three vials per sample. Gradually apply pressure of 1-10 psi until sample extract passes through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

Note: Instantaneous application of high pressure can degrade the glass filter and may cause plugging. When the liquid flow ceases at 50 psi, stop the filtration.

Note: Add pressure slowly when necessary. There should be no gurgling noises, indicating an influx of air, as volatiles may be lost and the sample would then be invalid. Also, if the filter in the vessel breaks open, the sample becomes invalid. If either of these occurs, the entire extraction for the sample must be repeated.

12.12.2 For multi-phase samples:

The liquid obtained prior to extraction (from a multi-phase sample or solid sample saturated with liquid) must either:

- If miscible, be combined with the extract and analyzed as one, or
- If non-miscible, be analyzed separately from the extract, mathematically combining the results using the following volume-weighted average formula:

$$\frac{V_1 C_1 + V_2 C_2}{V_1 + V_2} = \text{combined result}$$

Where:
V_1 = the volume of the liquid phase.
V_2 = the volume of the ZHE extract.
C_1 = the concentration of the analyte in the liquid phase.
C_2 = the concentration of the analyte in the ZHE extract.

12.12.3 Store the extracts at 4°C and analyze according to the specific analytical methods used.

13.0 Quality Control

13.1 A method blank comprised of each Extraction fluid used in the set is analyzed with each extracted batch not to exceed 20 samples.

13.2 Matrix spikes are not performed at the time of extraction but must be performed at the time of analysis. Spiking is performed as one spike per waste type (e.g., sludge, soil, aqueous, oil/solvent, etc.).

13.3 Check rotation of the tumbling apparatus weekly and record in logbook. Must be 30±2 RPM. A supervisor should be alerted if the rotation speed is unacceptable and corrective actions must be taken to make the speed acceptable.

13.4 Check start and stop room temperatures. The temperature of the room where the extraction takes place shall be maintained at 23 ± 2°C. If outside this temperature, the leach procedure must be redone.

13.5 The ZHE extractors should be checked periodically for leaks by either pressuring to 50psi and then re-checking the pressure later to see if it stayed the same, or pressurize the ZHE and submerge it in water and look for escaping air bubbles.

13.6 Blanks should be rotated among all vessels. Record vessels used for blanks in the Bottle Blank Location logbook (see attachment).

14 Method Performance

14.1 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.

14.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

14.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager. Note: IDoC for TCLP extraction is documented by demonstration of the trainee to the trainer.

14.1.3 MDLs are not applicable.

14.1.4 PEs are not applicable.

15 Pollution Prevention and Waste Management

15.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

15.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

16 References

"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Method 1311; 3rd edition; December 1996.

Annual Book of ASTM Standards, Volume 11.04, Method D 3987-85 (approved October 21, 1985, Published March 1986), Reapproved 2004.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, November 2000, Method 1312

17 Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

Table A – Summary of Equipment

Table B – Summary of Supplies

Table C – Summary of Reagents

Table D – Preparation of Reagents

Attachment I: Bottle Blank Location logbook page

Attachment II: ZHE Blank Location logbook page

Attachment III TCLP-SPLP Extraction Bench Sheet

Attachment IV ZHE Extraction Bench Sheet

Attachment V: Flow Charts

18 Revisions

Document Number	Reason for Change	Date
GB-I-067-Rev. 00	First Issue Merged SOPs: S-GB-I-025 *Toxicity Characteristic Leaching Procedure (TCLP),* S-GB-I-057, *SPLP – Synthetic Preparation Leaching Procedure,* and S-GB-I-058, *ASTM – Shake Extraction of Solid Waste with Water.*	05July2010

Table A
EQUIPMENT

Equipment	Manufacturer	Model(s)
Millipore Rotary Agitators	Millipore or equivalent*	YT31 ORA HW*
Rotary Agitator Bottles	Millipore or equivalent*	YT30 09G BT*
ZHE Vessels		
Hazardous Waste Filtration Vessel	Millipore or equivalent	YT30 090 HW*
Funnel	-	-
Beakers	VWR	13912-207
pH Meter	Corning*	PH 245*
Balance	Ohaus*	Precision Standard*
Syringes	GasTight*	1050*
Watch glass	-	-
Forceps		
Magnetic Stirrer	Corning*	PC 353*
Stir Bars	-	-
Spoonula (Lab Spoon)	FisherBrand*	14-375-10 (Fisher Cat#)*
100 mL Graduated Cylinder	-	-
2000 mL Graduated Cylinder	-	-
Carboys	-	20 liter and 12 liter
Vacuum Pump	-	-
Tempscribe Recorder	-	-
Thermometer	-	-
Stirring Hotplate	Corning*	Stirrer hotplate*
1L Volumetric Flask	-	-
Heated Water Bath	-	-

*- Or Equivalent

Table B
SUPPLIES

Supplies	Vendor	Catalog #
142 mm TCLP filters	Fisher Scientific*	09804-142G*
90 mm TCLP filter	Fisher Scientific*	09-804-142J*
40 mL Glass VOA vials		
Tempscribe Recorder Paper		
Tedlar Bags	SKC	231-01-TCLP
Compressed Nitrogen Gas	AirGas*	

*- Or Equivalent

Table C
REAGENTS

Reagent	Alias	Concentration	Directions found in:
Reagent Water	ASTM Type II	5.5SU - 7.5SU	-
Sodium Hydroxide		10 N	Table D
Nitric Acid		1 N	Table D
Sulfuric Acid			
Glacial Acetic Acid	HOAc	Concentrated	
Hydrochloric Acid		1 N	Table D
TCLP Extraction Fluid #1	TCLP Fluid #1	pH 4.93 ± 0.05	Table D
TCLP Extraction Fluid #2	TCLP Fluid #2	pH 2.88 ± 0.05	Table D
Sulfuric Acid/Nitric Acid	NA	60/40 weight percent mixture	Table D
SPLP Extraction Fluid #1	SPLP Fluid #1	pH 4.2 ± 0.05	Table D
SPLP Extraction Fluid #2	SPLP Fluid #2	pH 5.0 ± 0.05	Table D

Table D
PREPARATION OF REAGENTS

Reagent	Alias	Concentration	Directions	Shelf Life
Sodium Hydroxide		10 N	Weigh 400 g NaOH into ~400 ml DI water in a 1-L volumetric flask. Dilute to volume.	1 year
Nitric Acid		1 N		
Glacial Acetic Acid		Concentrated		
Hydrochloric Acid		1 N		
TCLP Extraction Fluid #1	TCLP Fluid #1	pH 4.93 ± 0.05	In 23 L carboy add 20 L of DI water. Add 228 ml glacial HOAc. Add 256 ml 10 N NaOH. Mix well. PH must be 4.93 ± 0.05.	1 week
TCLP Extraction Fluid #2	TCLP Fluid #2	pH 2.88 ± 0.05	In 12 L carboy add 10 L DI water. Add 68.4 ml glacial HOAc dilute to volume. pH must be 2.88 ± 0.05.	1 week
Sulfuric Acid/Nitric Acid	NA	60/40 weight percent mixture	Mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.	6 months
SPLP Extraction Fluid #1	SPLP Fluid #1	pH 4.20 ± 0.05	Add 60/40 weight percent mixture of sulfuric and nitric acids to DI water until the pH is 4.2 ± 0.05.	1 week
SPLP Extraction Fluid #2	SPLP Fluid #2	pH 5.00 ± 0.05	Add 60/40 weight percent mixture of sulfuric and nitric acids to DI water until the pH is 5.0 ± 0.05	1 week

Attachment I: Bottle Blank Location logbook page

Logbook #

TCLP Blank Vessel Rotation Log

	Date	Analyst	Vessel # 1	Vessel # 2	Vessel # 3	Vessel # 4	Vessel # 5	Vessel # 6	Vessel # 7	Vessel # 8	Vessel # 9	Vessel # 10	Vessel # 11	Vessel # 12	Vessel # 13	Vessel # 14	Vessel # 15	Vessel # 16	Vessel # 17	Vessel # 18	Vessel # 19
1																					
2																					
3																					
4																					
5																					
6																					
7																					
8																					
9																					
10																					
11																					
12																					
13																					
14																					
15																					
16																					
17																					
18																					
19																					
20																					

B = Used for Blank
1 = Used for Sample

F-GB-I-093-Rev.02 (05July2010)
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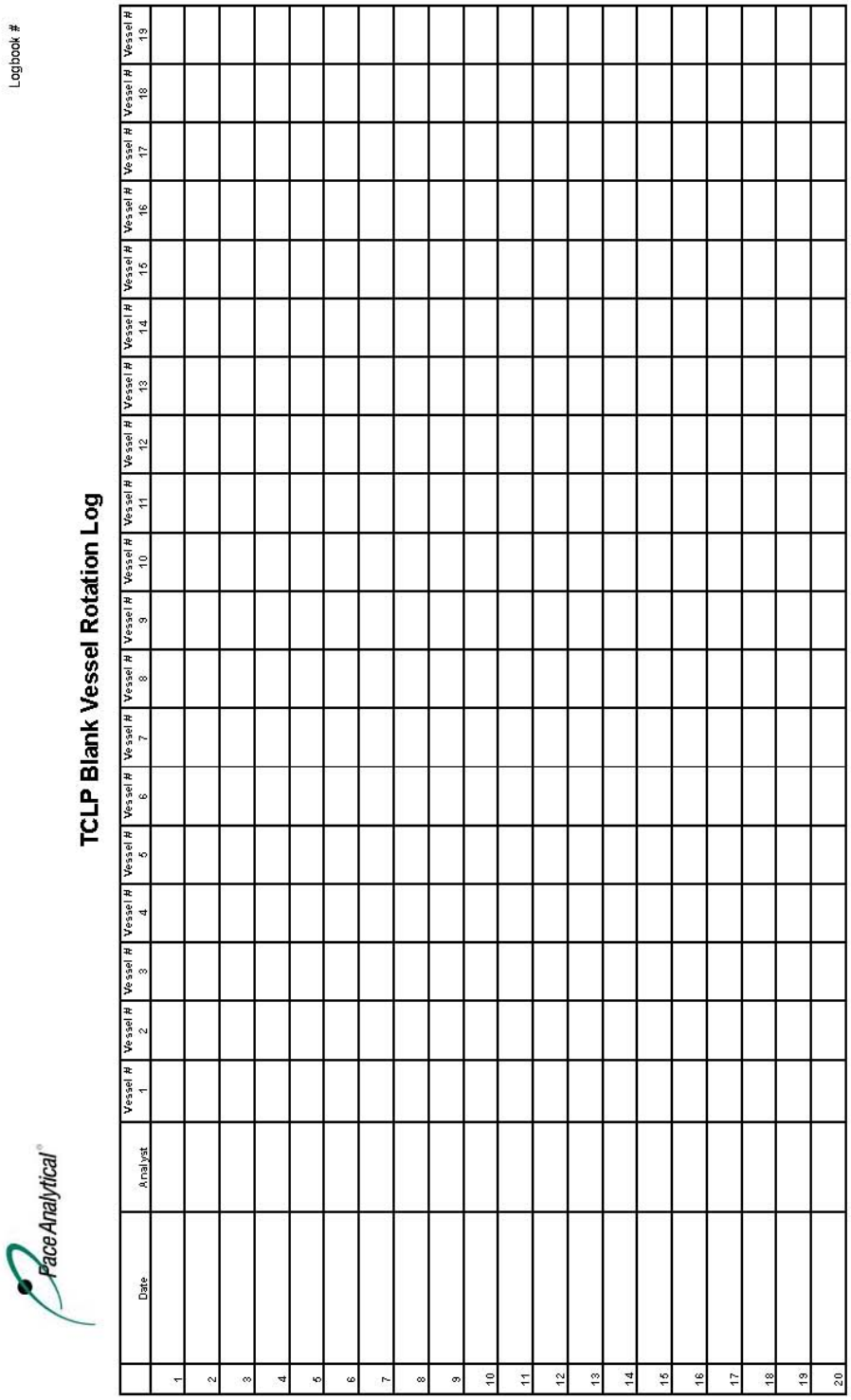
Attachment II: ZHE Blank Location logbook page



Logbook #

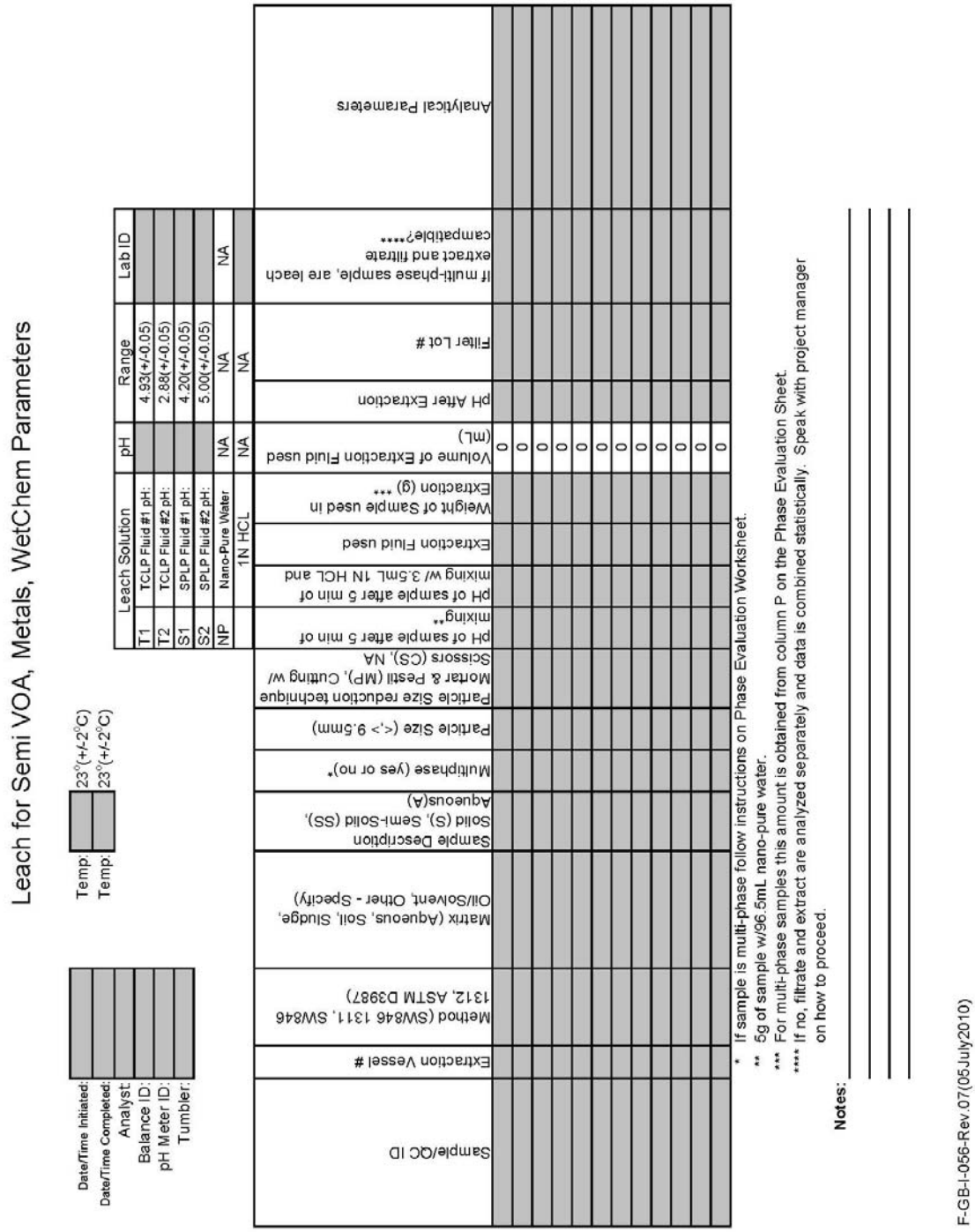
TCLP Blank Vessel Rotation Log

	Date	Analyst	Vessel # 1	Vessel # 2	Vessel # 3	Vessel # 4	Vessel # 5	Vessel # 6	Vessel # 7	Vessel # 8	Vessel # 9	Vessel # 10	Vessel # 11	Vessel # 12	Vessel # 13	Vessel # 14	Vessel # 15	Vessel # 16	Vessel # 17	Vessel # 18	Vessel # 19
1																					
2																					
3																					
4																					
5																					
6																					
7																					
8																					
9																					
10																					
11																					
12																					
13																					
14																					
15																					
16																					
17																					
18																					
19																					
20																					

B = Used for Blank
1 = Used for Sample

F-GB-I-092-Rev.00 (05July2010)
Green Bay Laboratory

Attachment III TCLP-SPLP Extraction Bench Sheet



Leach for Semi VOA, Metals, WetChem Parameters

Date/Time Initiated:
Date/Time Completed:
Analyst:
Balance ID:
pH Meter ID:
Tumbler:

Temp: 23°(+/-2°C)
Temp: 23°(+/-2°C)

	Leach Solution	pH	Range	Lab ID
T1	TCLP Fluid #1 pH:		4.93(+/-0.05)	
T2	TCLP Fluid #2 pH:		2.88(+/-0.05)	
S1	SPLP Fluid #1 pH:		4.20(+/-0.05)	
S2	SPLP Fluid #2 pH:		5.00(+/-0.05)	
NP	Nano-Pure Water	NA	NA	NA
	1N HCL	NA	NA	

Sample/QC ID	Extraction Vessel #	Method (SW846 1311, SW846 1312, ASTM D3987)	Matrix (Aqueous, Soil, Sludge, Oil/Solvent, Other - Specify)	Sample Description Solid (S), Semi-Solid (SS), Aqueous(A)	Multiphase (yes or no)*	Particle Size (<,> 9.5mm)	Particle Size reduction technique Mortar & Pestil (MP), Cutting w/ Scissors (CS), NA	pH of sample after 5 min of mixing**	pH of sample after 5 min of mixing w/ 3.5mL 1N HCL and	Extraction Fluid used	Weight of Sample used in Extraction (g) ***	Volume of Extraction Fluid used (mL)	pH After Extraction	Filter Lot #	If multi-phase sample, are leach extract and filtrate campatible?****	Analytical Parameters
												0				
												0				
												0				
												0				
												0				
												0				
												0				
												0				
												0				
												0				

* If sample is multi-phase follow instructions on Phase Evaluation Worksheet.
** 5g of sample w/96.5mL nano-pure water.
*** For multi-phase samples this amount is obtained from column P on the Phase Evaluation Sheet.
**** If no, filtrate and extract are analyzed separately and data is combined statistically. Speak with project manager on how to proceed.

Notes: _____

_____

_____

_____

F-GB-I-056-Rev.07(05July2010)

Phase Evaluation Worksheet for TCLP, SPLP, ASTM

Instructions
- Follow instructions from left to right, documenting data through column H.
- If result for column J is <0.5%, the the filtrate is the extract and no Buffer Solution or Tumble is needed.
- If result for column J is >0.5%, then filter and tumble with a fresh aliquote of sample. Use amount shown in column K. Document columns L, M, and N.
- Store filtrate from column N.
- Tumble the filter and the solids on it, documenting column P on the Leach Bench Sheet.

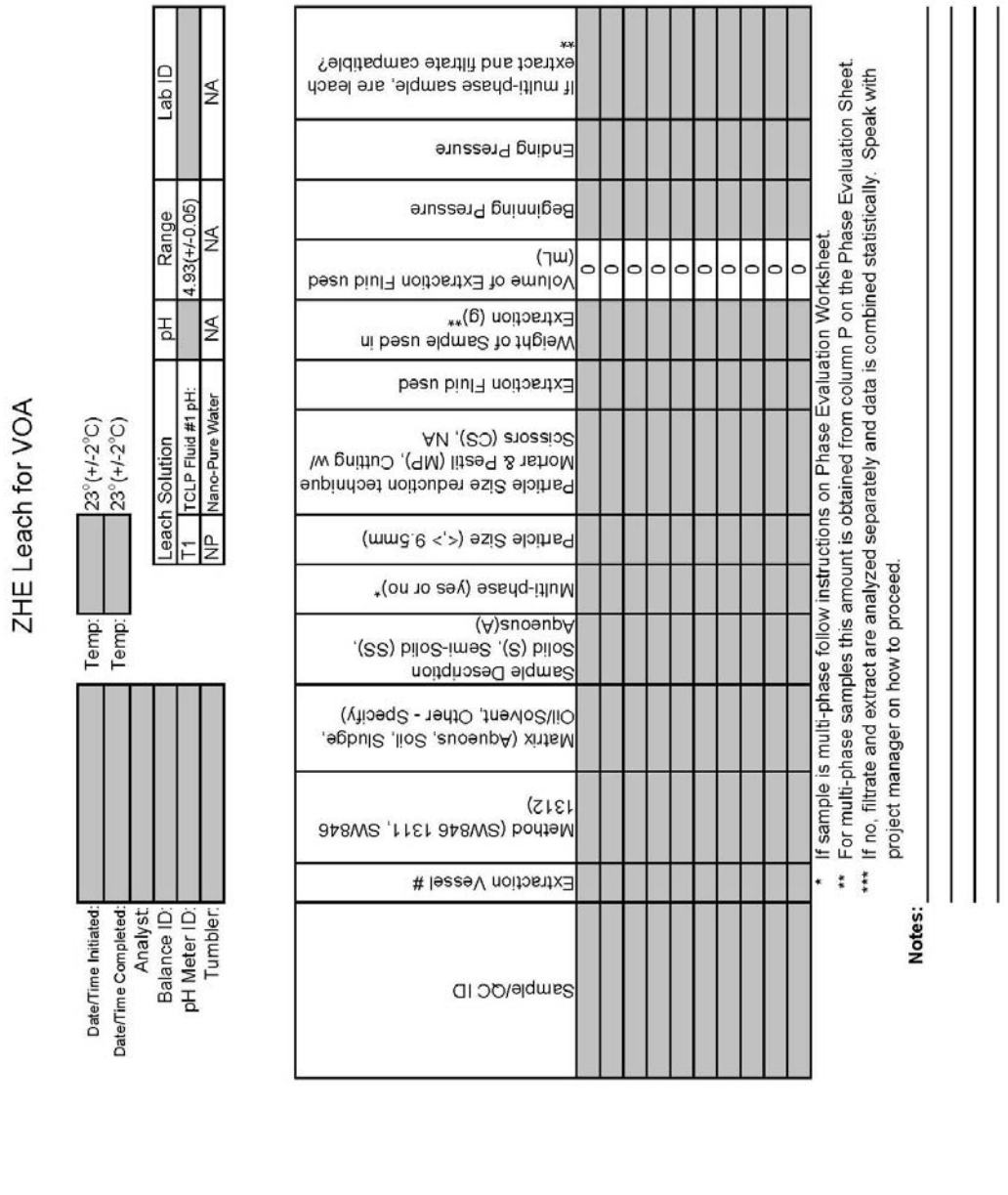
Filter Lot #:
Oven ID:
Date/Time Drying 1 Started:
Date/Time Drying 1 finished:
Date/Time Drying 2 Started:
Date/Time Drying 2 finished:

	Phase Determinative Procedure												Leach Procedure Documentation				
		A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
Sample ID	Record the weight of the glass fiber filter.	Glass Fiber Filter Weight (g)	Record the weight of the filtrate contaitner to be filtered into.	Filtrate Container Weight (g)	Weight out a 100g minimum and record weight. Quantitively transfer to filter device. Subtract residue weight if >1% of sample.	Sample Weight (g)	Finish assembly, apply pressure gradually in 10psi increments to 50psi. Stop after 2 min @ 50psi. Material on filter is solid phase and filtrate is liquid phase. Weigh the filled filtrate contaitner.	Filled Filtrate Contaitner Weight (g)	Liquid Phase Weight = D - B	Liquid Phase Weight (g)	Solid Phase Weight = C - E	Solid Phase Weight (g)	Dry the filter and solid phase @ 100° +/-20°C until two successive weightings are w/in 1%.	Dry Weight #1 of Solid Phase + Filter (g)	Dry Weight #2 of Solid Phase + Filter (g)	% Solids = (F/C)*100	% Solids	% Dry Solids = ((H-A)/C)*100	% Dry Solids(if <0.5%, the filtrate is the Extract and No Buffer Solution is Required)	Weight of sample to be filtered = (100/I)*100	Weight (g) of sample to be filtered and processed.	Actual sample Weight (g) used.	Record the weight of the filtrate contaitner to be filtered into.	Filtrate Container Weight (g)	Finish assembly, apply pressure gradually in 10psi increments to 50psi. Stop after 2 min @ 50psi. Material on filter is solid phase and filtrate is liquid phase. Weigh the filled filtrate contaitner. Tumble the filter and solid material on the filter.	Filled Filtrate Contaitner Weight (g)	Liquid Phase Weight (g) = N-M	Liquid Phase Weight (g)	Solid Phase Weight (g) = L-O	Solid Phase Weight (g) Record on Leach Bench Sheet

Notes:
- If some sample material adheres to the container and cannot be transferred into the filter device (>1 percent of sample weight), subtract this residue weight from the original sample weight (C).
- Instantaneous application of high pressuer during filtration can degrade the glass fiber filter and may cause plugging.
- Some samples, such as oily wastes or paint wastes, contain material that appears to be liquid, but may not filter. The material within the filtration device is defined as a solid. Do not replace the original filter with a fresh one.

F-GB-I-056-Rev.07(05July2010)

Attachment IV ZHE Extraction Bench Sheet



ZHE Leach for VOA

Date/Time Initiated: Temp: 23°(+/-2°C)
Date/Time Completed: Temp: 23°(+/-2°C)
Analyst:
Balance ID:
pH Meter ID:
Tumbler:

Leach Solution		pH	Range	Lab ID
T1	TCLP Fluid #1 pH:		4.93(+/-0.05)	
NP	Nano-Pure Water	NA	NA	NA

Sample/QC ID	Extraction Vessel #	Method (SW846 1311, SW846 1312)	Matrix (Aqueous, Soil, Sludge, Oil/Solvent, Other - Specify)	Sample Description Solid (S), Semi-Solid (SS), Aqueous(A)	Multi-phase (yes or no)*	Particle Size (<,> 9.5mm)	Particle Size reduction technique Mortar & Pestil (MP), Cutting w/ Scissors (CS), NA	Extraction Fluid used	Weight of Sample used in Extraction (g)**	Volume of Extraction Fluid used (mL)	Beginning Pressure	Ending Pressure	If multi-phase sample, are leach extract and filtrate campatible?**
										0			
										0			
										0			
										0			
										0			
										0			
										0			
										0			
										0			
										0			

* If sample is multi-phase follow instructions on Phase Evaluation Worksheet.
** For multi-phase samples this amount is obtained from column P on the Phase Evaluation Sheet.
*** If no, filtrate and extract are analyzed separately and data is combined statistically. Speak with project manager on how to proceed.

Notes: _____

F-GB-I-071-Rev.04(05July2010)

Phase Evaluation Worksheet for ZHE

Instructions
- Follow instructions from left to right, documenting data through column H.
- If result for column J is <0.5%, then the filtrate is the ZHE extract and no Buffer Solution or Tumble is needed.
- If result for column J is >0.5%, then filter and tumble with a fresh aliquote of sample. Use the amount shown in column K. Record data for columns L, M, and N.
- Store filtrate from column N in a Tedlar Bag.
- Document column P on the Leach Bench Sheet.
- Proceed with the ZHE leach procedure where the Buffer is added to the ZHE apparatus.

Filter Lot #:
Oven ID:
Date/Time Drying 1 Started:
Date/Time Drying **1** finished:
Date/Time Drying 2 Started:
Date/Time Drying 2 finished:

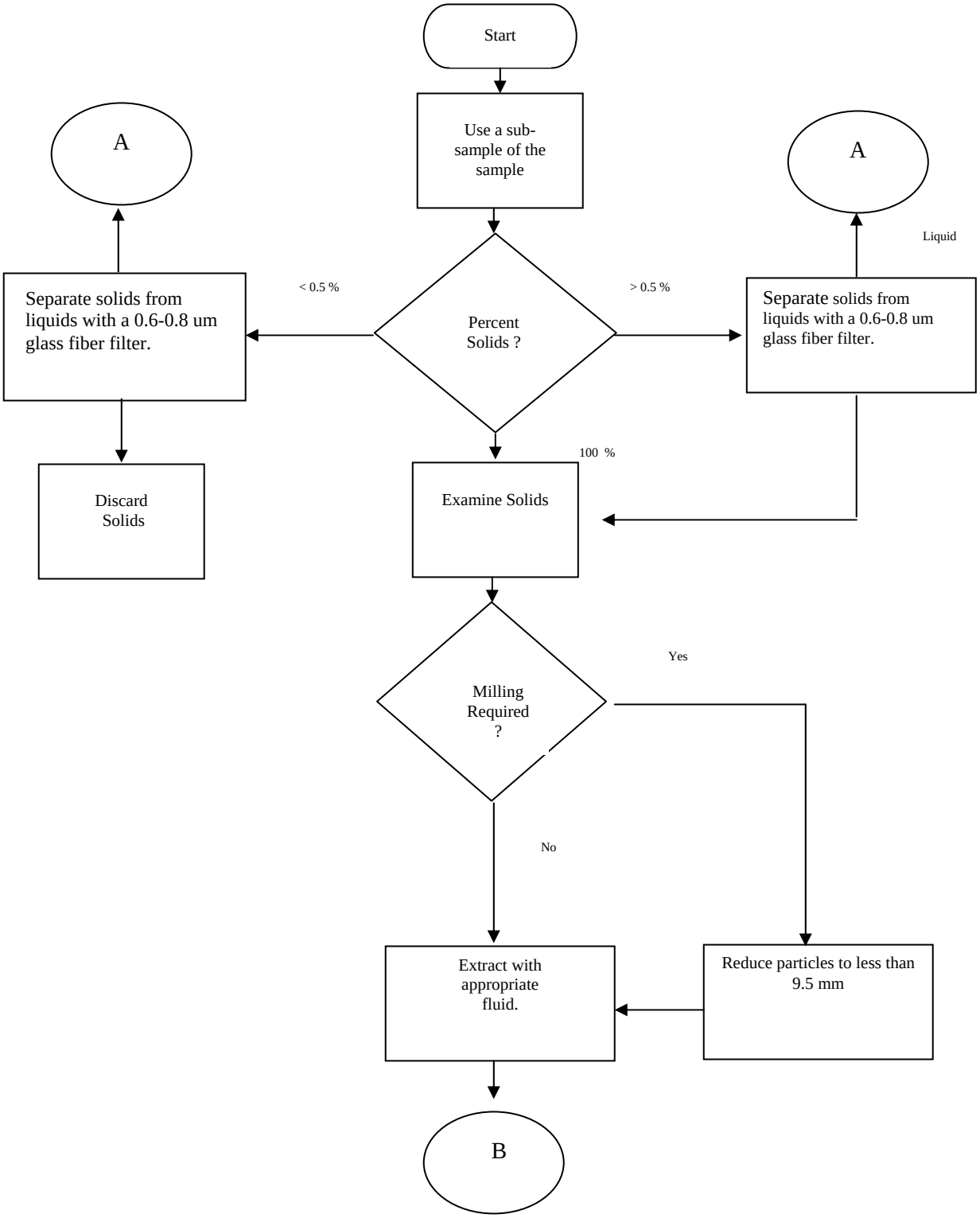
	Phase Determinative Procedure														Leach Procedure Documentation				
	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
Sample ID	Glass Fiber Filter Weight (g)	Filtrate Container Weight (g)	Sample Weight (g)	Filled Filtrate Container Weight (g)	Liquid Phase Weight (g)	Solid Phase Weight (g)	Dry Weight #1 of Solid Phase + Filter (g)	Dry Weight #2 of Solid Phase + Filter (g)	% Solids	% Dry Solids (If <0.5%, the filtrate is the ZHE Extract and No Buffer Solution is Required)	Weight (g) of Sample to be filtered and processed.	Actual sample Weight (g) used.	Filtrate Container Weight (g)	Filled Filtrate Container Weight (g)	Liquid Phase Weight (g) = (mL)
	Record the weight of the glass fiber filter.	Record the weight of the filtrate container to be filtered into.	Weight out 25g of sample and record weight. Quantitively transfer to filter device. Subtract residue weight if >1% of sample.	Finish assembly, apply pressure gradually in 10psi increments to 50psi. Stop after 2 min @ 50psi. Material on filter is solid phase and filtrate is liquid phase. Weigh the filled filtrate container.	Liquid Phase Weight = D - B	Solid Phase Weight = C - E	Dry the filter and solid phase @ 100° +/-20°C until two successive weightings are w/in 1%.		% Solids = (F/C)*100	% Dry Solids = ((H-A)/C)*100	Weight of sample to be filtered = (25/I)*100		Record the weight of the filtrate container to be filtered into.		Liquid Phase Weight (g) = N-M
	1.00	51.00	25.00	62.00	11	14	1.02	1.03	56	0.12	44.64	50.0	50.0	56.0	6.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0
					0	0			#DIV/0!	#DIV/0!	#DIV/0!				0.0

Notes:
- If some sample material adheres to the container and cannot be transferred into the filter device (>1 percent of sample weight), subtract this residue weight from the original sample weight (C).
- Instantaneous application of high pressuer during filtration can degrade the glass fiber filter and may cause plugging.
- fresh one.
  replace the original filter with a fresh one.

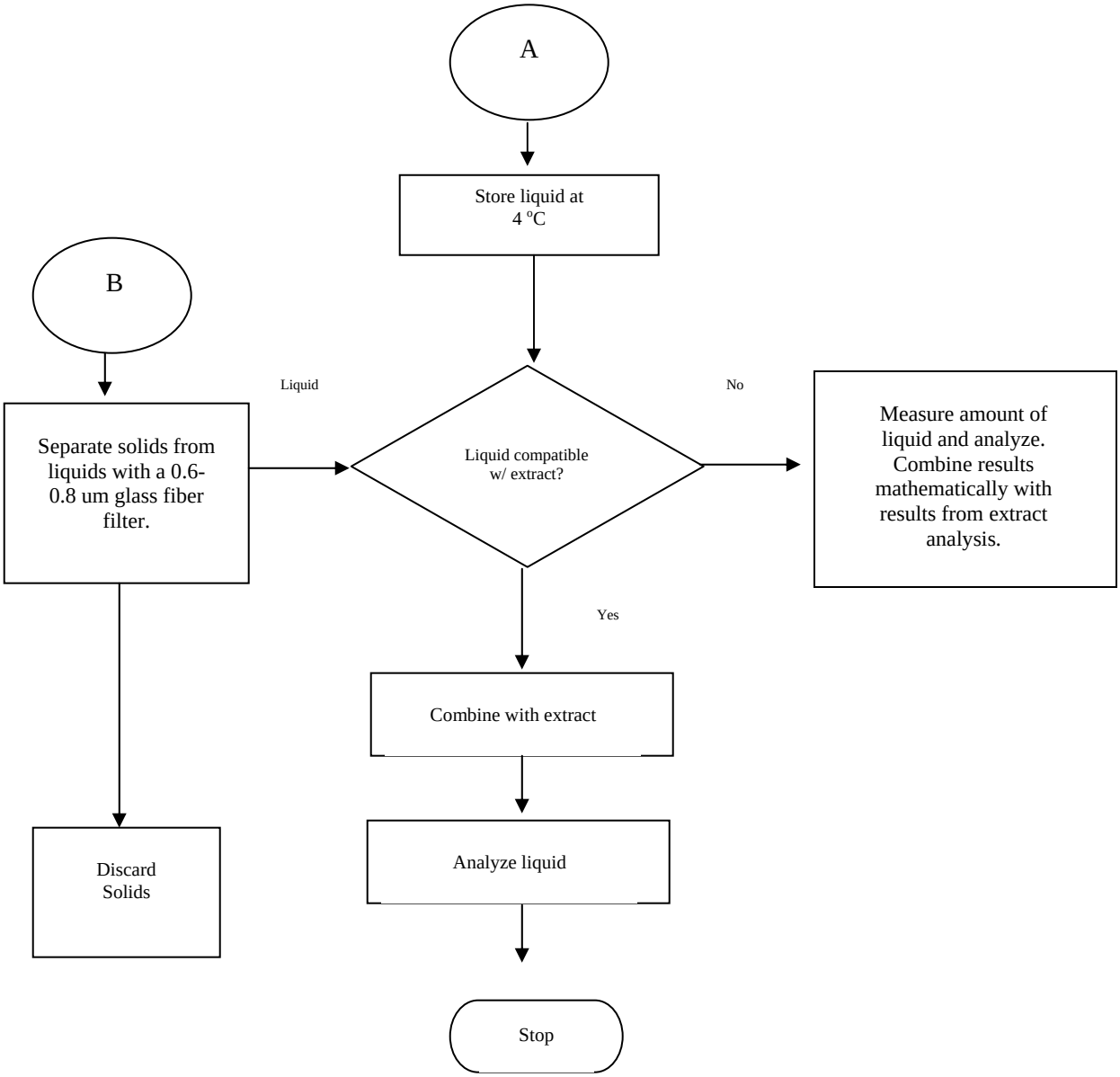
F-GB-I-071-Rev.04(05July2010)

Attachment V: Flow Charts

FLOWCHARTS



FLOWCHARTS


A
Store liquid at 4 °C
B
Liquid
No
Separate solids from liquids with a 0.6-0.8 um glass fiber filter.
Liquid compatible w/ extract?
Measure amount of liquid and analyze. Combine results mathematically with results from extract analysis.
Yes
Combine with extract
Discard Solids
Analyze liquid
Stop

Pace Analytical™
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Green Bay, WI 54302
Phone: 920.469.2436
Fax: 920.469.8827

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.00
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-ALL-GB-I-014-Rev.04

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 11/18/10

Kate Grams, Laboratory Quality Manager — Date: 11/8/10

Chad Rusch, Department Manager — Date: 11/15/2010

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) describes operations used to measure the parameters in the following table:

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	%
ASH	Ash	Solid	ASTM D2974-87(C)	%

2. Scope and Application

2.1 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.

2.2 This method is applicable to the determination of Total Solids (TS) and its Volatile (TVS) and Fixed Solids (FS) fractions in soil and semisolid samples. The terms total or fixed 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.

2.3 This method is applicable to the determination of Fractional Organic Carbon (FOC), ASH, 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.

2.4 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.

3. Summary of Method

3.1 Total Solids (TS) – A well-mixed sample is evaporated to dryness and the residue solids are measured gravimetrically.

3.2 Total Suspended Solids (TSS) – A well-mixed sample is filtered. The residue collected by the filter is dried and measured gravimetrically.

3.3 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.

3.4 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.

3.5 Fixed Solids (FS) - Residue obtained from the determination of TS is ignited at 550ºC in a muffle furnace. The weight remaining after ignition is reported as % Fixed Solids.

3.6 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.

3.7 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.

3.8 ASH - Residue obtained from the determination of TS is ignited at 440ºC in a muffle furnace. The weight remaining after ignition is used to calculate and report the % ASH.

4. Interferences

4.1 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.

4.2 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.

4.3 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.

4.4 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.

4.5 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.

4.6 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.

5. Safety

5.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

5.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

5.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

6. Definitions

6.1 Batch – A grouping of 20, or less, environmental samples of similar matrix, which are prepared and/or analyzed together with the same method.

6.2 Blank Sample – A sample analyzed to determine potential contamination attributed to sample handling and analysis.

6.3 Duplicate Sample – A second aliquot of the same environmental sample analyzed in the same manner as the original sample in order to evaluate precision.

6.4 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.

6.5 Laboratory Control Sample (LCS) – A blank matrix sample spiked with a known concentration of analytes of interest. Laboratory accuracy is evaluated using the LCS.

6.6 Laboratory Information Management System (LIMS) – This is a system for transferring, processing, storing, and reporting analytical results.

6.7 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.

6.8 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.

6.9 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.

6.10 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.

6.11 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.

6.12 Fixed Solids - Residue obtained from the determination of TS is ignited at 550ºC in a muffle furnace. The weight remaining after ignition is reported as % Fixed Solids.

6.13 Refer to Section 10 of the Pace Quality Manual for additional terms used in this SOP.

7. Sample Collection, Preservation and Handling

7.1 Samples are collected in plastic, glass, or any appropriate containers that prevents drying during storage.

7.2 Samples are stored at 4±2oC and analyzed as soon as possible to minimize microbiological decomposition of organic solids.

7.3 Samples should be analyzed as soon as possible and the holding time must not exceed **7 days** from time of collection. Samples analyzed after 7 days of collections must be qualified with the “H” qualifier.

8. Equipment and Supplies

8.1 Analytical balance – Electronic, capable of weighing to 0.1 mg.

8.2 Vacuum Filtration System – Includes filter holder, membrane filter funnel, vacuum flask and vacuum pump.

8.3 Glass Fiber Filters (for TDS) – GE Water & Process Technologies PreSep Prefiler, Glass, 1.0 Micron, 47mm, or equivalent.

8.4 Glass Fiber Filters (for TSS) – Pre-weighed from Environmental Express, ProWeigh 47mm Glass Fiber Filters, 1.5 μm, with matched aluminum evaporating dishes.

8.5 Graduated Cylinders – 25, 50, 100, 250, 500 and 1000mL.

8.6

8.7 Wide bore serological pipettes and pipette helper.

8.8 Drying Ovens – capable of maintaining 103-105ºC and 178-182ºC.

8.9 Desiccator

8.10 Indicating Desiccant – Drierite Anhydrous Calcium Sulfate, Stock # 2305, or equivalent.

8.11 Laboratory Scoopulas

8.12 Muffle furnace (for volatile and fixed solids) – Capable of maintaining temperature of 440ºC and 550ºC.

8.13 Evaporating Beaker (TS/TDS/TVS/OM/FOC) – 50 mL

9. Reagents and Standards

9.1 ERA DataPacK Total Volatile Solids Standard, Cat # 913 for TVS, OM, and FOC analysis.

9.2 ERA QC Plus Solids, Cat #4033 for TS, TSS*, and TDS analyses.

9.3 Alpha-Trol Standard, Cat #W-13 for TS, TSS, and TDS* analysis.

9.4 Nanopure Reagent Water – ASTM Type II, or equivalent.

* Not the preferred standard for this test.

10. Calibration

10.1 Balance Calibration – The only required calibration is the analytical balance.

10.2 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 ALL-Q-013 *Support Equipment* and associated addenda.

10.3 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

10.3.1 1g for TSS and TVSS with a tolerance of +/-0.0100g

10.3.2 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

11. Procedure

11.1 Create the analytical batch in Horizon.

11.2 Open LimsLink and create a new worksheet for the batch to be analyzed.

11.3 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.

11.4 Check that the oven temperature is recorded in the logbook each morning and is within required specifications before placing samples into oven as per the most current version of SOP ALL-Q-013 *Support Equipment* and associated addenda.

11.5 **Weighing Dry/Muffle Weights**

11.5.1 Oven temperatures are the same as those described for the test being conducted.

11.5.2 Place the samples back into the oven for 15 minutes and repeat desiccating, cooling, and weighing steps until weight change is $\leq$0.0005g.

11.5.3 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.

11.5.4 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.

11.5.5 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.

11.6 Total Solids Determination (TS)

11.6.1 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 ≤0.0005g before use.

11.6.2 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 LimsLink.

11.6.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of Alpha-Trol (or equivalent) and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in LimsLink.

11.6.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

11.6.5 Sample

12.5.4.1 For Solids Samples – Weight out 1 to 1.5g of sample in a tared and dried evaporating beaker. Record the weight in LimsLink.

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 LimsLink.

11.6.6 Place the samples to dry in the oven overnight at 103-105°C. (Overnight is a period of time ≥ 8 hours.)

11.6.7 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 12.5, and record in LimsLink.

11.6.8 If Total Volatile Solids (TVS) are also to be performed proceed to Section 12.9. Note: TVS utilizes a different LCS than the TS.

11.7 Total Suspended Solids Determination (TSS)

11.7.1 Record Filter IDs and weights in LimsLink for samples and batch QC.

11.7.2 Method blank (MB) – Using a graduated cylinder measure out 1000mL of Nanopure (or equivalent) water, record the volume in LimsLink, and start at 11.7.8.

11.7.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of Alpha-Trol (or equivalent), record the volume in LimsLink, and start at 11.7.8.

11.7.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

11.7.5 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.

11.7.6 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.

11.7.7 Shake the sample thoroughly and measure the chosen aliquot using a graduated cylinder.

11.7.8 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.

11.7.9 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.

11.7.10 Remove the filter with forceps and place it back into the evaporating dish.

11.7.11 Place the samples (filters and residue) to dry in the oven a minimum of 3 hours at 103-105°C.

11.7.12 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 12.5.

11.7.13 If Total Volatile Suspended Solids (TVSS) are also to be performed proceed to Section 11.10.

11.8 **Total Dissolved Solids (TDS)**

11.8.1 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.

11.8.2 Method blank (MB) – Using a graduated cylinder measure out 50mL of Nanopure (or equivalent) water, record the volume in LimsLink, and start at step 11.8.6.

11.8.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of APG Solids APGplus Standard (or equivalent), record the volume in LimsLink, and start at step 12.8.6.

11.8.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

11.8.5 Shake the sample thoroughly and measure a 50mL aliquot using a graduated cylinder.

11.8.6 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.

11.8.7 Filter the sample and transfer the filtrate to a pre-weighed dried evaporating beaker.

11.8.8 Place the samples to dry in the oven overnight at 178-182°C (Overnight is a period of time $\geq$ 8 hours.)

11.8.9 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 12.5.

11.9 **Total Volatile Solids (TVS)**

11.9.1 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 $\leq$0.0005g before use.

11.9.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pore into a tared evaporating beaker. Record the weight in LimsLink.

11.9.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pore into a tared evaporating beaker. Record the weight in LimsLink.

11.9.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

11.9.5 Sample

12.5.4.3 For Solids Samples – Weight out 1 to 1.5g of sample in a pre-dried, tarred evaporating beaker. Record the weight in LimsLink.

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 LimsLink.

11.9.6 Follow TS Sections 11.6.6 to 11.6.7 to obtain a dried residue.

11.9.7 Place the evaporating beaker or filter and evaporating dish into the muffle furnace at **550ºC** for 1 hour.

11.9.8 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 12.5.

11.10 **Total Volatile Suspended Solids (TVSS)**

11.10.1 Method blank (MB) – Using a graduated cylinder, measure out 1000mL of Nanopure (or equivalent) water, record the volume in LimsLink.

11.10.2 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

11.10.3 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 LimsLink.

11.10.4 Follow TSS Sections 11.7.8 to 11.7.12 to obtain a dried residue.

11.10.5 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.

11.10.6 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 12.5 in LimsLink.

11.11 **Fractional Organic Carbon (FOC), ASH, and Organic Matter (OM)**

11.11.1 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 $\leq$0.0005g before use.

11.11.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pore into a tared evaporating beaker, record the weight in LimsLink.

11.11.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pore into a tared evaporating beaker, record the weight in LimsLink.

11.11.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

11.11.5 Samples – Into a tared evaporating beaker weigh out about 1-1.5g of sample. Record the weight in LimsLink.

11.11.6 Follow Sections 11.6.6 to 11.6.7 to obtain a dried residue.

11.11.7 Place the evaporating beakers into the muffle furnace at **440ºC** for 1 hour.

11.11.8 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 12.5 in LimsLink.

11.12 **Entering Results into Horizon**

11.12.1 Post samples and batch QC using LimsLink.

11.12.2 Evaluate batch QC in Horizon. Non-reported LCSs need to be evaluated in LimsLink.

11.13 **Calculations**

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)

Volatile Solids (TVS and TVSS mg/L) $= \frac{(A - D) \times 1000 \times 1000}{V}$

Volatile Solids (TVS %) $= \frac{(A - D) \times 100}{(A - B)}$

% Fixed Solids $= \frac{(D - B) \times 100}{(A - B)}$

% FOC $= ((1-((D-B) / (A - B))) \times 100) \times (0.58)$

% OM $= ((1-((D-B) / (A-B))) \times 100)$

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)

11.13.1 Precision - The relative percent difference (RPD) is calculated as:

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

where: **S** = sample value
D = duplicate value

11.13.2 Constant Weight is calculated as:

$$\textbf{Constant Weight (\%)} = \frac{|FW - CW|}{(FW + CW)/2} * 100$$

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.

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 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.

12.2.1 TS, TDS, TVS, FOC, FS, and OM all use 50mL of deionized water for the method blank.

12.2.2 TSS and TVSS both use 1000mL of deionized water for the method blank.

12.2.3 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%.

12.2.4 For tests that are reported in mg/L the absolute value must be < PRL.

12.2.5 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

12.2.6 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.

12.3 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. The only test without an LCS is TVSS.

12.3.1 ERA DataPacK Total Volatile Solids Standard, Cat. # 913 is used for TVS, FOC, and OM analysis. Equivalent can be used.

12.3.2 APG Solids APGPlus standard, Cat #4033 is used for TDS analysis. Equivalent can be used.

12.3.3 Alpha-Trol Standard, CAT#NC9460010 is used for TS and TSS analysis. Equivalent can be used.

12.3.4 A Laboratory Control Spike Duplicate (LCSD) must be analyzed if the client requests one.

12.3.5 The LCS recovered concentration must be within calculated in-house limits or default limits of $\pm$ 20%.

12.3.6 For tests that are reported in %, the LCS is evaluated in mg/Kg in LimsLink and not reported in Epic Pro.

12.3.7 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.

12.3.8 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 out side 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.

12.3.9 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.

12.3.10 When an LCSD is performed, the precision between the LCS and LCSD must a be $\leq$20% RPD.

12.3.11 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.

12.3.12 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an "R1" data qualifier.

12.4 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 $\leq$10%.

12.4.1 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.

12.4.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an "R1" data qualifier

12.5 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 $\leq$0.0005g or the weight loss between the two weights is less than 4%.

12.6 Balance – The balance must be verified daily across the range of use, before use, as per the most current version of SOP ALL-Q-013 *Support Equipment* and associated addenda.

12.6.1 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

12.6.2 1g for TSS and TVSS with a tolerance of +/-0.0100g

12.6.3 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

12.7 Oven – The oven temperature is recorded in the logbook each morning and must be within required specifications before placing samples into oven as per the most current version of SOP ALL-Q-013 *Support Equipment* and associated addenda.

12.8 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 "H" data qualifier and fill out a LabTrack report.

12.9 See attachments Table A and Table B for a summary of QC

13. Method Performance

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 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.

14. Pollution Prevention and Waste Management

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

14.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of ALL-S-002, *Waste Handling*.

14.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

15. References

15.1 Method 2540-G, Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992.

15.2 ASTM D2974-87, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, May 29, 1987

15.3 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

16. Tables, Diagrams, Flowcharts, Appendices, Addenda, etc.

16.1 Table A: **QUALITY CONTROL**

16.2 Table B: **ANALYST/TECHNICIAN DATA ASSESSMENT**

16.3 Flow charts – Attachment I.

17. 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

Table A. QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SM 2540G – ASTM D2974-87**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. None performed on TVSS.
Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.

Table B. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**SM2540G – ASTM 2974-87 If these conditions are not achieved ⇒**
Method Blank	• 1
Precision Duplicate Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3

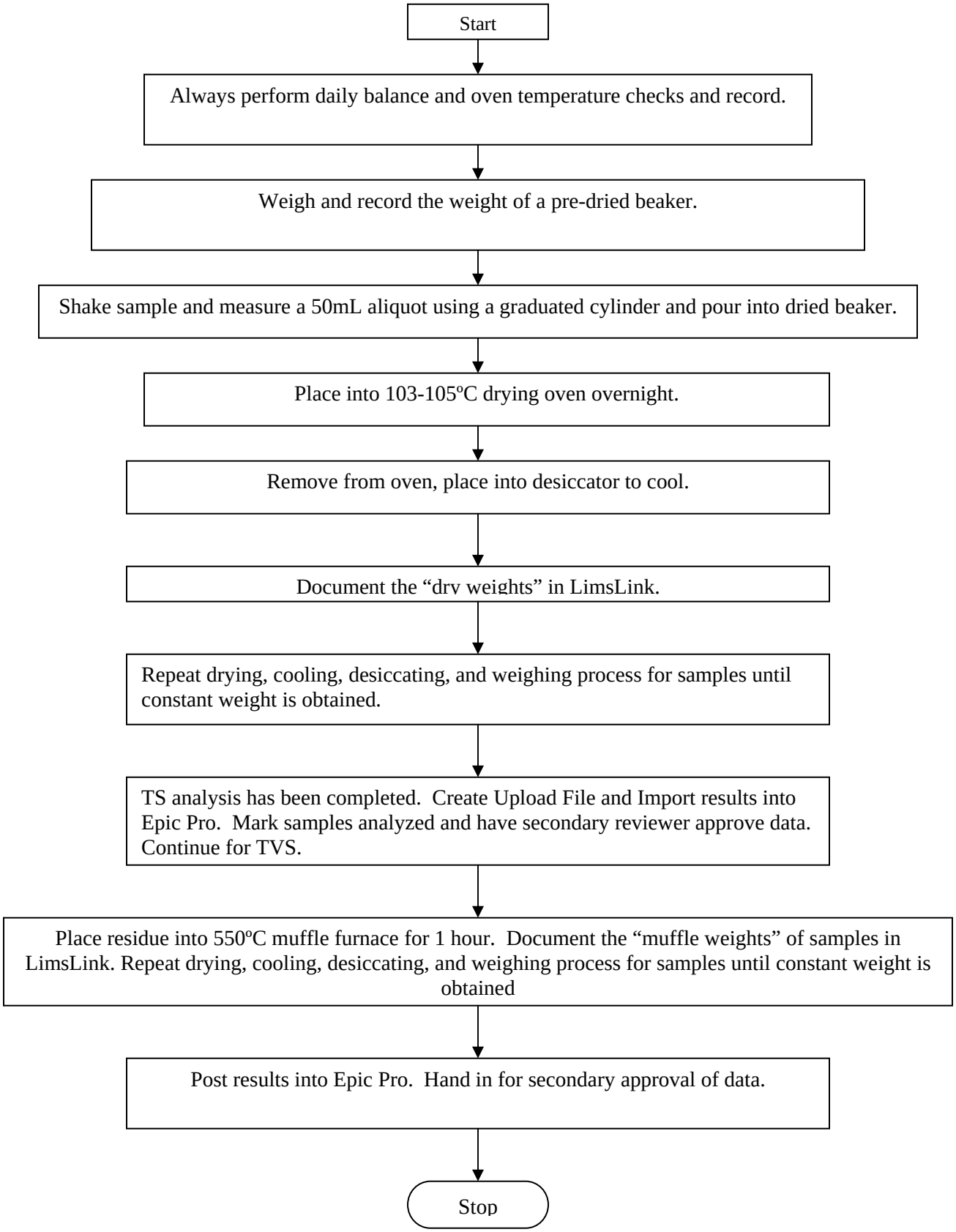
1. In the absence of project specific requirements, sample detects less than 20 times the method blank contamination level is reported with the "A" data qualifier. Sample detects greater than 20 times the method blank contamination are reported without qualification.

2. In the absence of project specific or method requirements, in-house generated limits will be used If the PS and PSD fails precision control limits flag the parent with the "R1" (precision) data qualifier.

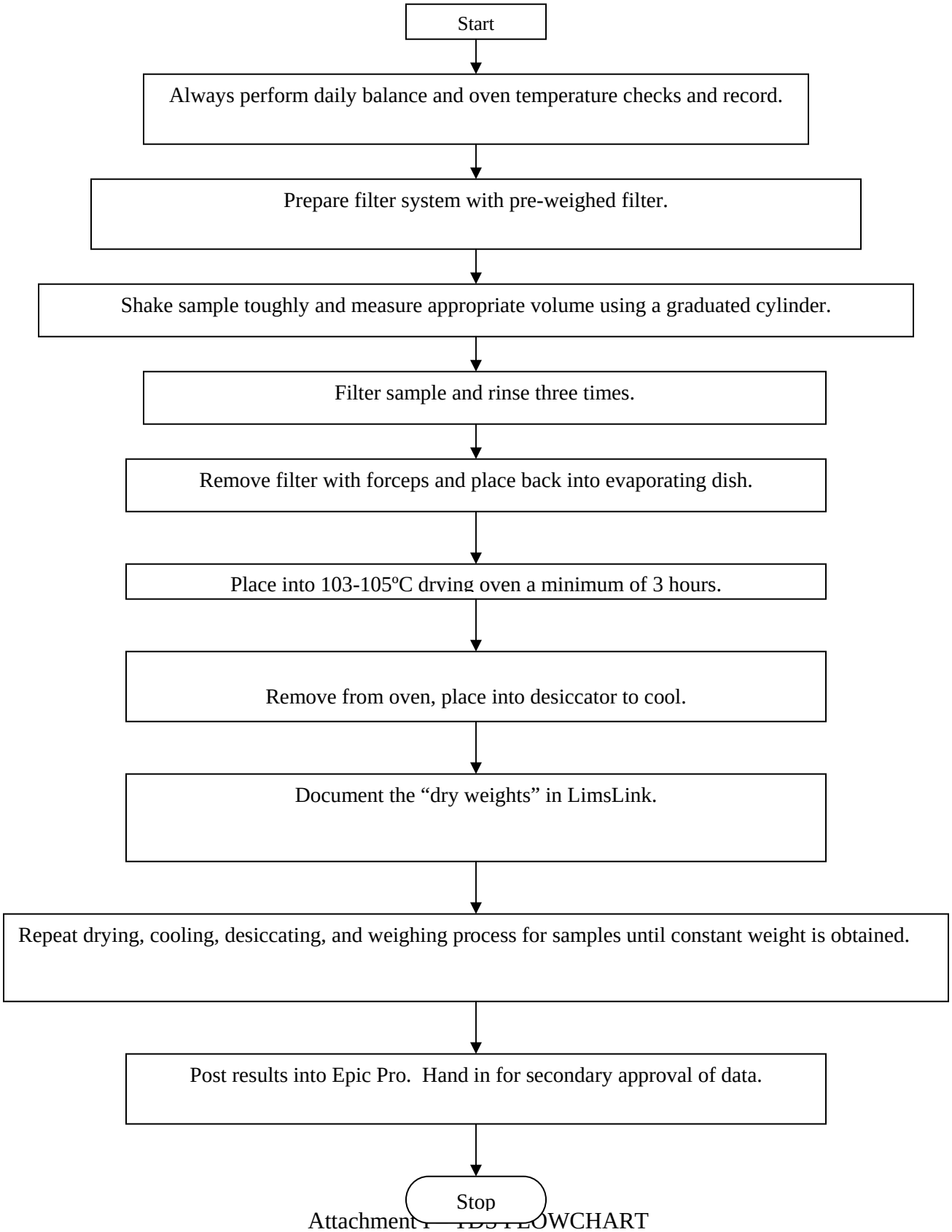
3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the "L0" (accuracy) and "R1" (precision) qualifier to reflect the deficiencies.

.

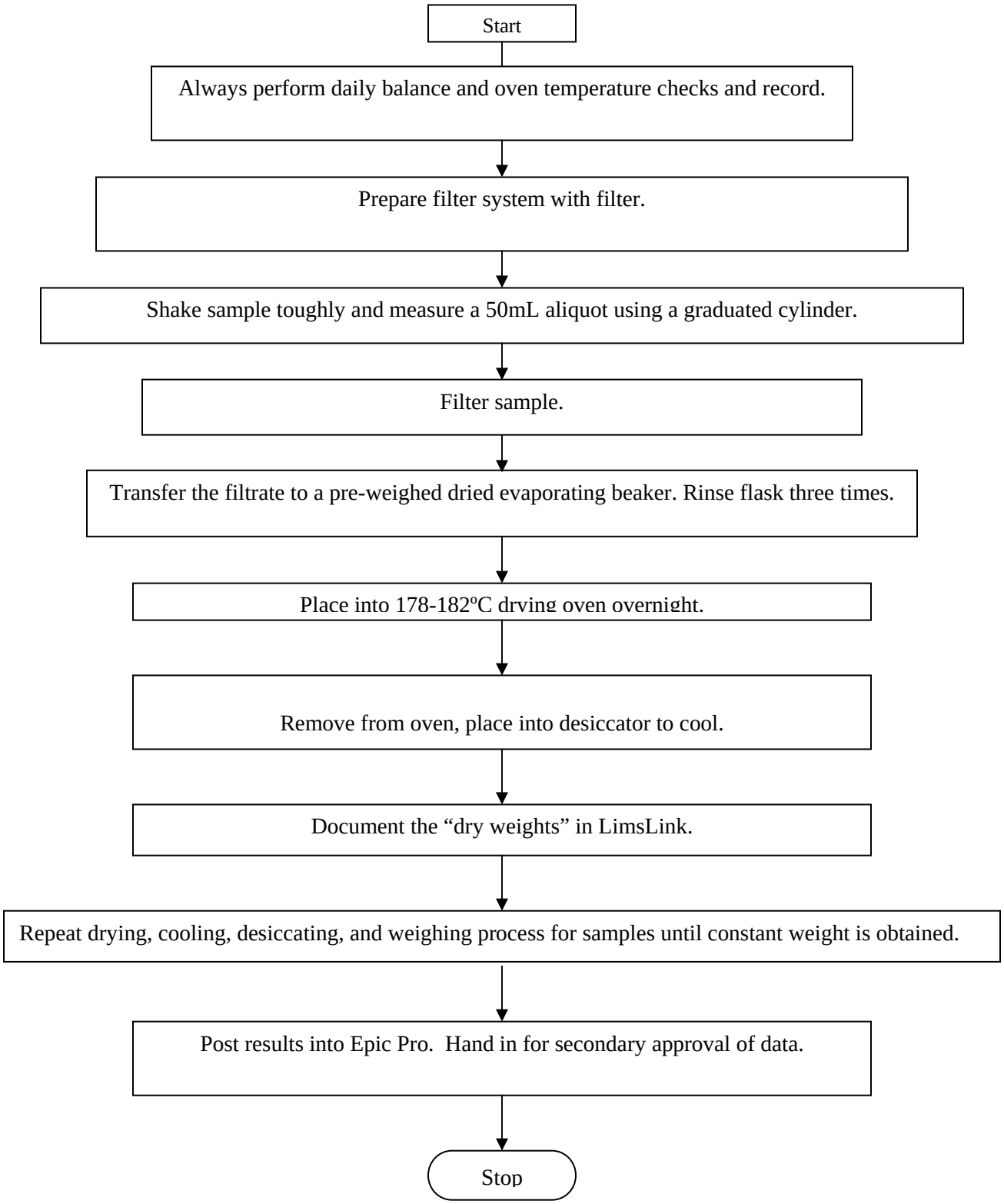
Attachment I – TS and TVS FLOWCHART



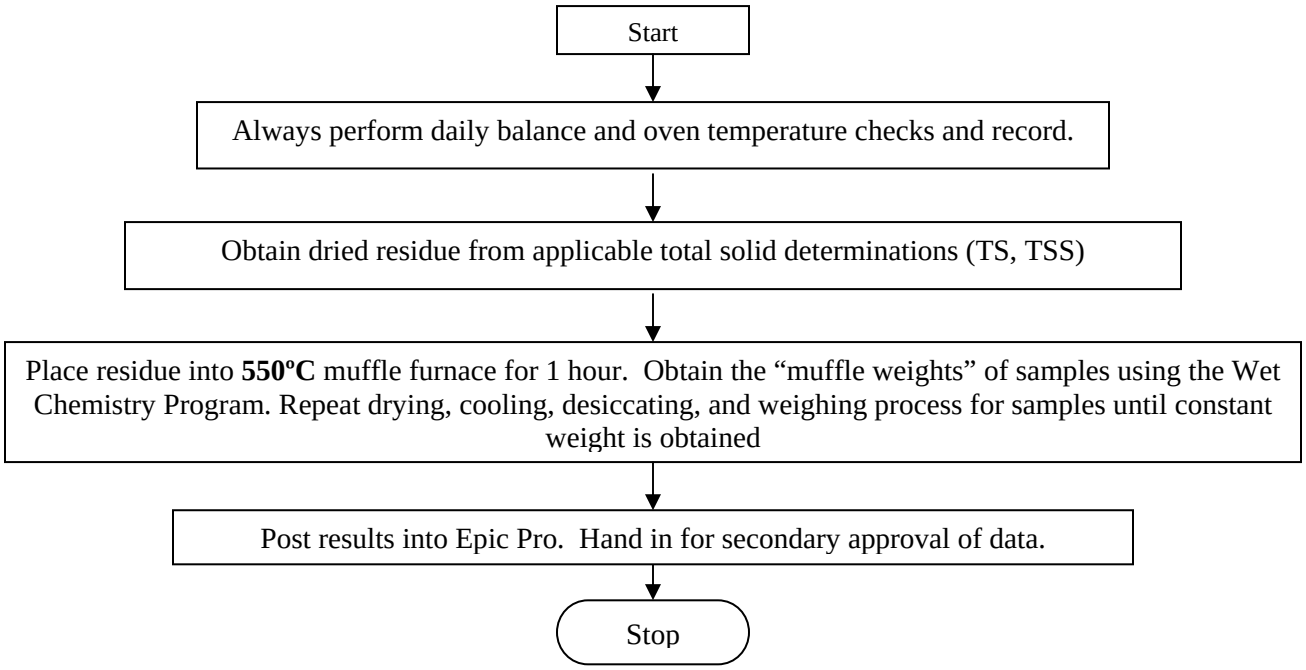
Attachment I – TSS FLOWCHART



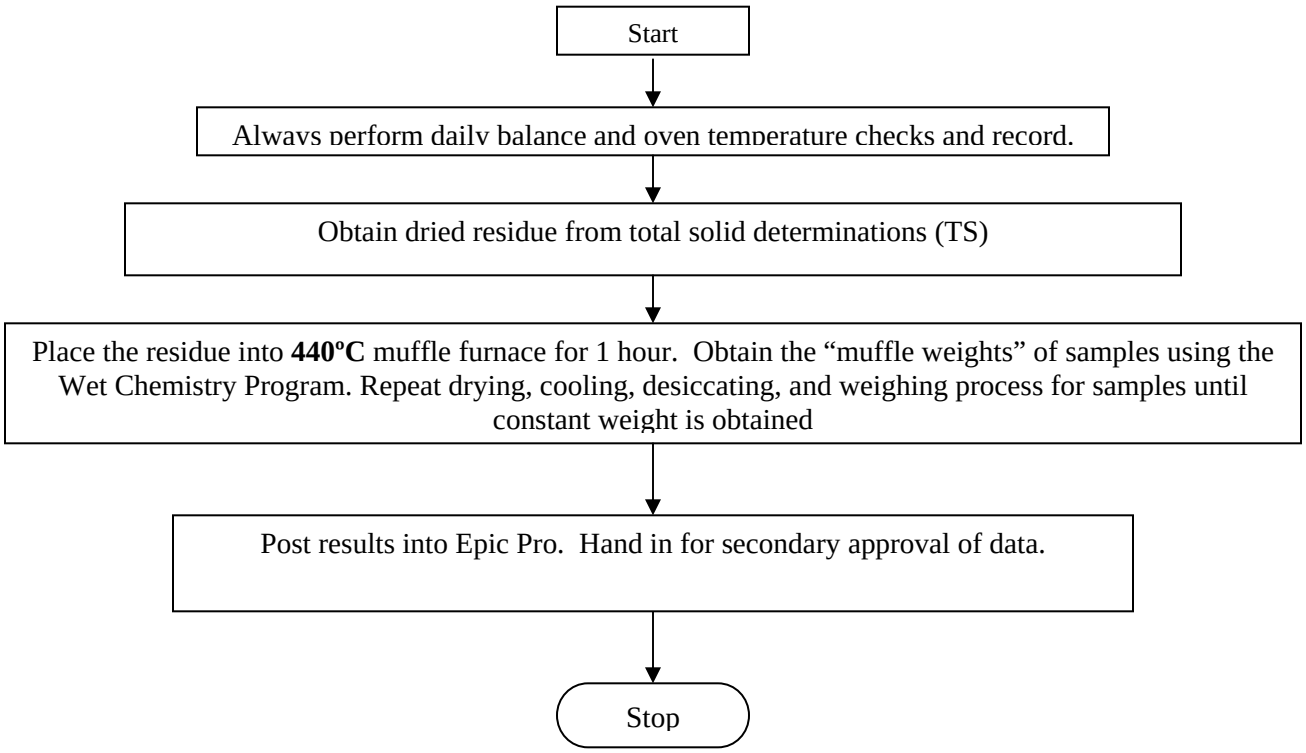
Attachment I – TDS FLOWCHART



Attachment I – TVS and TVSS FLOWCHART



Attachment I – OM and FOC FLOWCHART



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STANDARD OPERATING PROCEDURE

The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy

Reference Methods: EPA SW-846 Methods 7470A and 7471A/B

SOP NUMBER:	S-GB-M-004-REV.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-M-004-REV.02

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 9/16/10

Kate Grams, Laboratory Quality Manager — Date: 9/15/10

Chad Rusch, Department Manager — Date: 9/16/10

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the determination of mercury in water, soil/solid samples, and biological tissue.

2. SCOPE AND APPLICATION

2.1 This method is applicable to water samples (including drinking water, surface water, and domestic and industrial wastewaters), solid samples (including soils, sediments, sludges, and, solid wastes), wipes, TCLP leachates, and biological tissue samples.

2.2 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 atomic absorption spectrometry. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3. SUMMARY OF METHODS

3.1 Cold vapor atomic absorption utilizes the volatile property of elemental mercury at the 253.7 nm wavelength. To release mercury from organic complexes, the sample is digested with oxidizing reagents and acids in a hot block digester. After digestion, the oxidizing reagents are neutralized. Stannous chloride is added to reduce ionic mercury to the ground state. The Flow Injection Analysis System sweeps the volatile elemental mercury out of the sample and into the cell of an atomic absorption spectrophotometer. The absorbance signal is proportional to the amount of mercury in the sample.

4. INTERFERENCES

4.1 Biota samples can contain diverse matrix types, each of which may present analytical challenges. Spiked samples and Laboratory Control Samples are important for determining digestion efficiency.

4.2 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide, as NaS, do not interfere with recovery of added inorganic Mercury from D.I. water.

4.3 Copper has also been reported to interfere; however, copper concentrations as high as 10mg/L had no effect on recovery of Mercury from spiked samples.

4.4 Samples high in chlorides require up to 7.5 mL of additional potassium permanganate. During the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the Mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent. Samples may be diluted to decrease the chlorides. Alternatively, the sample may be allowed to stand for at least an hour under a hood (without active purging) to remove the chlorine.

4.5 Certain volatile organic materials that absorb at this wavelength may also interfere. A preliminary run without reagents would determine if this type of interference is present.

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.

5.2 Material Safety Data Sheets (MSDSs) – A reference file of MSDS are on file in the laboratory and available to all personnel. A formal safety plan has been prepared and distributed to all personnel with documented training.

5.3 Special care should 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.

5.4 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.

5.4.1 Extreme caution must be used when preparing rodents for digestion. The samples must undergo a special procedure to destroy any Hantavirus, which may be present. Refer to the most recent version of SOP S-GB-L-002 *Small Rodent Handling and Homogenization* for details.

5.5 Analysts should take necessary safety precautions when handling chemicals and samples. Proper personal protective equipment may include safety gloves, lab coats, and safety glasses or goggles. Analysts should be familiar with the MSDS sheets for all chemicals and reagents they use for this procedure and the location of the MSDS sheets within the laboratory. Any questions or concerns should be taken to the laboratory Chemical Hygiene/Safety Officer.

6. DEFINITIONS

6.1 Definitions of terms found in this SOP can also be found in the Pace Quality Manual. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP or the Pace Analytical Services' Quality Manual Glossary, Section 10.0.

6.2 Biota Control Blank (Matrix Blank) - A sample of a matrix that is used for the control spike. The biota control blank will either be catfish, tilapia, chicken, or other tissue which ever is available at the time of analysis. The biota control blank should be "farm-raised" to minimize background mercury levels. The concentration in the biota control blank will be subtracted from the concentration of the laboratory control spike. This is done because the biota control blank is known to have some contamination. This SOP will reference biota control blank for ease. Note: For plant material analysis, alfalfa can be used as the biota control blank matrix.

6.3 Reagent Grade – Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

6.4 Standardized Reference Material (SRM) – A certified reference material produce by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. A SRM is analyzed with each analytical batch of biota samples.

6.5 Pace Reporting Limit (PRL) – The level at which method, permit, regulatory and client 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.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 BIOTA SAMPLES

7.1.1 Biota samples can be collected in clean plastic or glass containers or plastic baggies.

7.1.2 Biota samples are kept frozen until time of preparation to preserve integrity. Hold time is 28 days from removal from freezer.

7.2 SOIL SAMPLES

7.2.1 Soil and waste samples should be collected in clean wide-mouth glass containers to facilitate obtaining representative aliquots for measurement.

7.2.2 Samples should be stored at 4±2°C and analyzed as soon as possible to minimize microbiological decomposition of organic solids. Samples must be analyzed within 28 days of collection.

7.3 WIPE SAMPLES

7.3.1 Wipe should be collected in clean wide-mouth glass containers.

7.3.2 Samples should be stored at 4±2°C and analyzed as soon as possible to minimize microbiological decomposition of organic solids. Samples must be analyzed within 28 days of collection.

7.4 WATER SAMPLES

7.4.1 Water samples may be collected in plastic or glass containers and must be preserved with HNO_3 to pH <2 and stored at room temp, acid not to exceed 5% of the container capacity.

7.5 NOTE: Aqueous samples that react violently to the addition of acid may be collected without chemical preservation with proper variances approved by the regulatory authority. The responsibility of requesting this variance lies with the sample collector.

7.6 NOTE: Samples may be preserved in the lab. The samples may not be processed until 24 hours after preservation with a pH test of <2.

7.6.1 Samples must be analyzed within 28 days of collection.

8. EQUIPMENT AND SUPPLIES

8.1 Heating Block – Operated at 95 ± 5°C. Heating block should be Environmental Express "Hot Block", or equivalent.

8.2 Digestion Vials – Pre-cleaned 68- mL screw cap vials, Environmental Express (SC475), or equivalent.

8.3 Analytical Balance – Capable of weighing to 0.01g.

8.4 Volumetric Flasks – Assorted, Class A

8.5 Graduated Cylinder – 50-mL

8.6 Mechanical Pipettes – Assorted adjustable air-displacement pipettes with disposable tips (Eppendorf or equivalent).

8.7 pH Strips

8.8 CVAA-FIMS with autosampler

8.9 Inert Sparging and Carrier Gas – Argon

8.10 Autosampler vials

8.11 Autosampler Pump Tubing

8.12 Specimen Cups

8.13 DOLT-3

8.14 Analytical Balance – Capable of weighing to 0.0001g.

9. REAGENTS AND STANDARDS

9.1 Trace Metals Water

9.2 Nitric Acid (HNO_3) – Trace metals grade

9.3 Hydrochloric Acid (HCl) – Trace metals grade

9.4 Sulfuric Acid (H_2SO_4) – Trace metals grade

9.5 Sodium Chloride (NaCl)

9.6 Potassium Permanganate ($KMnO_4$) solution (5%) – Dissolve 50.0 g of $KMnO_4$ into ~800 mL of trace metals water. Dilute to 1000 mL. Prepare fresh each month.

9.7 Potassium Persulfate ($K_2S_2O_8$) solution (5%) – Dissolve 50.0 g of $K_2S_2O_8$ into ~800 mL of trace metals water. Dilute to 1000 mL. Warm to dissolve. Prepare fresh each month.

9.8 Hydroxylamine Hydrochloride ($NH_2OH \bullet HCl$) Solution (12%) – Dissolve 120 g NaCl and 120g $NH_2OH \bullet HCL$ in ~800 mL of trace metals water. Dilute to 1000 mL. Prepare fresh each month.

9.9 Stannous Chloride Solution ($SnCl_2$) - Dissolve 11.0 g of SnCl in ~ 800 mL of trace metals water. Add 30 mL of HCL. Dilute to 1000 mL. Prepare fresh every other day.

9.10 Analytical Standards – Standard solutions are required calibration, calibration checks, and sample spiking solutions. The following describes the contents of each type of solution. The commercial sources for stock solutions, recipes for preparing dilutions and working standards, and concentrations in standard solutions are presented in Table 1, Table 2, and Table 3.

Table 1
Stock Standards

Standard	Concentration	Vendor	Catalog #
Hg Calibration Stock	1000µg/mL	High-Purity Standards	CGHG1-1
ICV Calibration Stock	1000µg/mL	Inorganic Ventures, Inc.	100033-1

Standards are used until they expire based on the manufacturer's expiration date.

Table 2
Intermediate and Calibration Standards
Waters

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO_3 used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate	Hg ICV Stock	1000 µg/ml	20 µL	6.0 mL	200 mL	100 ppb
Hg Cal. 1	-	-	-	-	20 mL	0.0 ppb
Hg Cal. 2 and PRLs	Hg Cal. Intermediate	100 ppb	40 µL	-	20 mL	0.2 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	200 µL	-	20 mL	1.0 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	500 µL	-	20 mL	2.5 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	1000 µL	-	20 mL	5.0 ppb
Hg Cal. 6	Hg Cal. Intermediate	100 ppb	2000 µL	-	20 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	800 µL	-	20 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	1000 µL	-	20 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	20 mL	0.0 ppb

All intermediate dilution solutions must be prepared as needed, or at least every 7 days. Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

Table 3
Intermediate and Calibration Standards
Soils/Biota

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO_3 used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate	Hg ICV Stock	1000 µg/ml	20 µL	6.0 mL	200 mL	100 ppb
Hg Cal. 1	-	-	-	-	30 mL	0.0 ppb
Hg Cal. 2 and PRLS	Hg Cal. Intermediate	100 ppb	60 µL	-	30 mL	0.2 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	300 µL	-	30 mL	1.0 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	750 µL	-	30 mL	2.5 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	1500 µL	-	30 mL	5.0 ppb
Hg Cal. 6	Hg Cal. Intermediate	100 ppb	3000 µL	-	30 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	1200 µL	-	30 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	1500 µL	-	30 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	30 mL	0.0 ppb

All intermediate dilution solutions must be prepared as needed, or at least every 7 days. Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

10. CALIBRATION

10.1 A digested calibration curve is made up every time there are samples prepared. The curve is associated to the sample batch it is prepped with. Calibration requires analysis of a calibration blank and at least five levels of calibration solutions. The lowest calibration concentration is the Pace Reporting Limit.

10.2 The resultant correlation coefficient must be greater than 0.995. If this does not occur, then the instrument must be recalibrated and or the calibration curve and the associated samples must be re-prepped.

10.3 The calibration curve must pass an Initial Calibration Verification (ICV) that is analyzed after the calibration standards and before any samples. The ICV concentration is near the midpoint of the calibration curve and is made from a source other than the one used to make the calibration standards.

10.4 Pace Reporting Limit Standard (PRL Std) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

10.5 Every ten samples or less must be bracketed by Continuing Calibration Verifications (CCV). The CCV concentration is near the midpoint of the calibration curve.

10.6 The ICV and CCV limits are +/- 10% of their expected values. If one fails, then the instrument must be re-calibrated and any samples after the failure or not bracketed by an acceptable CCV, must be reanalyzed.

10.7 An acceptable Initial Calibration Blank (ICB), must be analyzed after the ICV.

10.8 Continuing Calibration Blanks (CCB) are analyzed after the CCVs.

10.9 The control limit for the ICB and CCB is less than the Pace Reporting Limit. If one fails, then the instrument must be re-calibrated and any samples, after the failure or not bracketed by an acceptable CCB, must be reanalyzed.

11. PROCEDURE

11.1 Sample Preparation

11.1.1 Water samples

11.1.1.1 Verify preservation in the samples by checking their pH with a test strip (done in Sample Receiving).

11.1.1.2 Prior to analysis, the samples and the calibration curve must be digested.

11.1.1.3 Turn on the heating block and set the temperature to maintain a sample temperature of 95$\pm$5°C. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the temperature in the sample logbook. Verify that the hood is functioning.

11.1.1.4 Add 20 mL of trace metals water and the appropriate amounts (as shown in Table 2) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.

11.1.1.5 Add 20 mL of trace metals water to a digestion vial for the Method Blank.

11.1.1.6 Add 20 mL of trace metals water and 1.0 mL of Hg ICV Intermediate to two digestion vials for the Laboratory Control Spike and Laboratory Control Spike Duplicate. Only prep LCSD if requested by client or there is not enough sample to run an MS/MSD.

11.1.1.7 Add 20 mL of each well-mixed sample to digestion vials.

11.1.1.8 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random or assigned by the client. An additional 20 mL of this sample along with 1.0 mL of Hg ICV Intermediate is added to two digestion vials.

11.1.1.9 To each digestion vial add 0.5 mL of concentrated nitric acid. Swirl the digestion vials gently.

11.1.1.10 To each digestion vial add 1.0 mL of concentrated sulfuric acid. Swirl the digestion vials gently.

11.1.1.11 Add 3 mL of 5% $KMnO_4$, , swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, an additional 3 mL of $KMnO_4$ solution is added to all samples, QC, and standards. **Note: If the sample still does not maintain color, discard applicable samples and dilute the samples prior to digestion.**

11.1.1.12 Add 1.6 mL of 5% Persulfate solution.. Place lid or cap on tube so as to allow pressure to vent but minimizing evaporation. Swirl the digestion vials gently.

11.1.1.13 Digest samples for 2 hours at 95$\pm$5°C.

11.1.1.14 Remove samples and let cool to room temperature.

11.1.1.15 Add 1 mL of 12% hydroxylamine hydrochloride solution. Swirl the digestion vials gently.

11.1.1.16 Samples are now ready for analysis.

11.1.2 Soil/Solid/Biota/Wipe Samples

11.1.2.1 Prior to analysis the samples and calibration curve must be digested.

11.1.2.2 Turn on the heating block and set the temperature to maintain a sample temperature of 95$\pm$5°C. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the temperature in the sample logbook. Verify that the hood is functioning.

11.1.2.3 Add 30 mL of 3% HNO_3 and the appropriate amounts (as shown in Table 2) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.

11.1.2.4 Prepare one Method Blank by adding glass beads to it. For biota analysis, leave the Method Blank empty. A Biota Control Blank is also prepared by weighing 0.6 g of homogenized biota control blank tissue into a digestion vial.

11.1.2.5 Prepare the Laboratory Control Spike (LCS) for soils, wipes, and wastes by adding glass beads to it. Then, add 1.5 mL of Hg ICV Intermediate to the digestion vial. Prepare the LCS for biota samples by weighing 0.6 g of homogenized biota control blank into the digestion vial. Add 1.5 mL of Hg ICV Intermediate the vial. Only prep a Laboratory Control Spike Duplicate (LCSD) if requested by client or there is not enough sample to run an MS/MSD. Prepare the LCSD in the same fashion as the LCS.

11.1.2.6 For biota samples weigh approximately 0.05 g of Standard Reference Material on an analytical balance (must read to at least 4 places past the decimal) into a labeled digestion vial.

11.1.2.7 Weigh 0.6 g of homogenized sample into a labeled digestion vial. Record the weight to the nearest 0.01 g in the digestion logbook. For samples with high liquid content, a larger sample size may be used, as long as the digestion is complete. For wipes, put the WHOLE wipe in the digestion vial.

11.1.2.8 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random. Weigh 2 additional 0.6 g aliquots into digestion vials. Add 1.5 mL of Hg ICV Intermediate to each vial.

11.1.2.9 Add 30 mL of 3% HNO_3 solution to all QC and samples. Swirl digestion vials to gently mix.

11.1.2.10 To each digestion vial add 0.5 mL of concentrated nitric acid. Swirl digestion vials to gently mix.

11.1.2.11 To each digestion vial add 2.0 mL of concentrated hydrochloric acid. Swirl digestion vials to gently mix.

11.1.2.12 Add 3 mL of 5% $KMnO_4$, place lid or cap on tube so as to allow pressure to vent but minimizing evaporation, swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, an additional 3 mL of $KMnO_4$ solution is added to all samples, QC, and standards. **Note: If the sample still does not maintain color, discard set and dilute the sample prior to digestion.** For Biota samples, add 6ml of KMnO4 to start with.

11.1.2.13 Digest samples for 30 minutes at 95$\pm$5°C.

11.1.2.14 Remove samples and let cool to room temperature.

11.1.2.15 Add 3 mL of 12% hydroxylamine hydrochloride solution. Swirl digestion vials to gently mix.

11.1.2.16 Samples are now ready for analysis.

11.2 BASIC SYSTEM OPERATION – ANALYTICAL

11.2.1 This portion of the SOP is designed to allow the user to set up and run a method, print a sample report, then shut down using the more basic software functions. For a more detailed explanation of the many other options, the user should refer to the Reference Manual.

11.2.1.1 Verify the carrier argon gas supply is on.

11.2.1.2 Switch on the FIMS 100.

11.2.1.3 Verify the computer is on. If not switch on the computer and enter network password and click on Start.

11.2.1.4 Go to programs, FIMS, and click on the AA WinLab Analyst and choose a custom workspace.

11.2.1.5 Click on GBWRK.FMS and then OK.

11.2.1.6 Go to "Sample Information Editor", File, Open Sample info File.

11.2.1.7 Choose the most recent applicable file and click OK.

11.2.1.8 Make changes to the calibration curve IDs in locations 1-9 and enter sample information starting with Autosampler Location #10. Enter the LIMS ID numbers under sample ID columns.

11.2.1.9 Go to File, Save As and change the Sample Info File identification to match the data file name and click OK.

11.2.1.10 In the menu click on Print and sample information to get a copy of the sample information you just entered to check for errors.

11.2.1.11 Close the window by clicking on the X in the upper right-hand corner.

NOTE: Only sample information should be entered in this file. Information on blanks, standards and QC samples is entered in the QC page of Method Editor.

11.2.1.12 Under the QC page you set up the QC sample ID, i.e. ICVs and CCVs and the autosampler location. Set the QC concentration and the control limits.

11.2.1.13 On the Schedule Timing page set up when the QC will be analyzed, i.e. after calibration, after 10 samples, and at the end. IF re-calibration occurs the QC must be analyzed at the same frequency.

11.2.1.14 Under the Actions Calib. And Periodic page, specify what to do if calibration QC fails, for example recalibrate, recalibrate and rerun, or stop.

11.2.1.15 On the Actions: End and Retry page, specify what QC to run at the end of a run and corrective action if QC fails.

11.2.1.16 Under the Options page, set how you want data printed (usually default). It is also possible to specify saving the peaks and add comments about the run.

11.2.1.17 In the Automated Analysis Window there are 2 pages: Set UP, and Analyze.

11.2.1.18 Under "Use Autosampler Locations listed Below", type in the numbers or range of numbers to be analyzed.

11.2.1.19 Select the name of the Results Data Set to save the results. Naming example: 0412101, denotes the, month, day and year. A number is used for describing each run within a day's output.

11.2.1.20 The Save Data box and Print Log box should already be selected.

11.2.1.21 Off After Analysis. Pumps should be checked.

11.2.2 Starting the Analysis

11.2.2.1 Prepare the required reagents: Carrier solution and reducing agent.

11.2.2.2 Clamp down the tubes by swinging the pressure levers until they lock. Place inlets of each solution in proper container: Yellow/blue in the carrier and red/red in the reductant. Click on the FIAS icon to turn on the pump to draw solutions through the tubing. Check on the flow.

11.2.2.3 Click on the Analyze All button. The system will run the blank and standards specified in the Element Parameter file, then the entire sample specified in the Sample Info. File. Check the calibration curve by clicking on Calibration icon on the tool bar.

11.2.2.4 To run the blank and standards only: In order to check the calibration curve before running any samples:

11.2.2.5 Click on Calibration, the system will run the blank and standards specified in the Element File and complete the calibration curve. To check the calibration curve, click on the Calibration icon on the toolbar.

11.2.2.6 To continue the run, click on the Auto control window then click on Analyze sample. The system will run the samples specified in the Sample Information file.

11.2.2.7 To change or stop the element file that has already started: Click on Analyze. A dialogue box will appear with these options:

Stop immediately

Stop after current replicate

Stop after completing all replicates for current sample

11.2.2.8 To restart the analysis from the beginning: In the Automated Analysis Control window, click on RESET. Start the analysis as usual.

11.2.2.9 To continue the analysis where it was interrupted or continue with a specific sample: In the Automated analysis Control window, click on Analyze All or Analyze Samples. A dialog box appear, elect one of the options. Click on OK.

11.2.2.10 Performing a recalibration: You define the type and frequency of automatic recalibration on the Checks page of the Method.

Stop the analysis.

In the Automated Analysis Control window, click on Reset.

In the Automated Analysis Control window, click on Calibrate.

11.2.2.11 Analyzing One or More Selected Samples: IN the Automated Analysis Control window, click on Select Location – a dialog box appears. Select the Analyze Samples In Locations option. Enter the autosampler locations of the solutions to analyze in the test box: Enter individual locations or a range of locations. Use comas to separate the locations and ranges, e.g. 10-15,18,20,25-30. Click OK.

11.2.2.12 Resetting the Autosampler: In the Automated Analysis Control window, click on Reset. Analyze the sample; the system will begin with the first sample in the sample list on the Set Up page of the Automated Analysis Control window.

11.2.2.13 Raising or Lower Sample Probe: In the Automated Analysis Control window, click on Probe Up/Down. The sample probe moves up or down but remains at the sample autosampler locations.

11.2.2.14 Rinsing the Sampling Probe and Sampling Tube: In the Automated Analysis Control window, select Location – a dialog box appears. Select "Go to Wash." Click on OK. The sampling probe moves to the wash location. In the FIAS Control window: Make sure the FIAS-valve is in the correct position to rinse the sample channel. Click on the Pump.

11.2.2.15 The sample pump feeds the rinse fluid from the wash container through the sample channel. Click on Pump again to stop it. Click on Move Probe Up/Down to raise the probe out of the wash container in the automated analysis Control window.

11.2.3 System Shutdown

11.2.3.1 Place inlets of the carrier and reagent lines in a container of DI water. Rinse the sample, carrier, and reagent tubing with DI water. In the FIAS Control window, click on Valve Fill/Inject a number of times while the pump is running. This ensures that the sample channel and the inside of the FIAS-valve are rinsed effectively.

11.2.3.2 After adequate time, remove, all the tubes from the rinse containers. Click on Move Probe Up/Down to raise the sampling probe.

11.2.3.3 Allow the pump to run until all the tubes and the gas/liquid separator are empty. Then click on the Pump button to stop the pump.

11.2.3.4 Make sure that the FIAS-valve is in the Fill position.

11.2.3.5 Swing all the pressure levers away from the pump tube magazines.

11.2.3.6 Exit the AA WinLab application.

11.2.3.7 Switch off the FIMS spectrometer.

11.3 CALCULATIONS

11.3.1 **Water Samples** – Since initial sample aliquot and final digestate volumes are the same, the mercury analyzer data system will calculate the concentration directly. No further calculations are necessary unless the sample was diluted.

11.3.2 **Soil/Solid/Biota Samples** -

Final Result (mg/kg dry weight corrected) =

$$\frac{\text{Raw data result (ug/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{Sample weight (g)} * \text{Dry weight for soil/solid (decimal form)}}$$

Biota results are reported on an as is/wet weight basis. The dry weight correction in the formula is not applicable.

11.3.3 Wipes

Final Result (ug/wipe) =

$$\frac{\text{Raw data result (ug/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{1 wipe}}$$

11.3.4 **Accuracy**-

$$\text{Spike Percent Recovery} = \frac{\text{Spike Sample Result} - \text{Sample Result}}{\text{Spike Added}} \times 100$$

11.3.5 **Precision**-

Relative Percent Difference =

$$\frac{\text{Spike Sample Result} - \text{Spike Sample Duplicate Result}}{(\text{Spike Sample Result} + \text{Spike Sample Duplicate Result})/2} \times 100$$

12. QUALITY CONTROL

12.1 Calibration Blanks – Calibration blanks (ICB and CCB) may not contain concentrations in excess of the Pace Reporting Limit. If blank results are not acceptable, analysis of the batch should be halted. Corrective actions could include preparation of fresh calibration standards and blanks.

12.2 Calibration Checks- Calibration checks (ICV and CCV) must agree within 10% of the true value. If the results are unacceptable, re-calibrate the system and then re-analyze the check solution and samples.

12.3 Pace Reporting Limit Standard (PRL Std) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

12.4 Laboratory Control Spike – A Laboratory Control Spike (LCS) must be prepared and analyzed with every sample batch or every 20 samples, whichever is more frequent. A Laboratory Control Spike Duplicate (LCSD) is performed if there is insufficient sample available for a MS/MSD or if requested by the client. The LCS and LCSD must meet criteria 85-115% recovery or all the samples prepared in the batch must be re-prepped and re-analyzed. The acceptance criteria for precision is 20% RPD. If insufficient sample remains to re-prep and re-analyze, the data qualifiers is given to all associated samples.

12.5 Method Blank – A Method Blank (MB) must be prepared and analyzed with every sample batch or every 20 samples, whichever is more frequent. The MB must not contain mercury at a concentration at or above the Pace Reporting Limit. Any samples digested with an unacceptable method blank must be re-prepped and analyzed unless the sample concentrations are less than the reporting limit or more than 10 times the value found in the method blank. In those cases that MDL reporting is required, the MB must be evaluated to the MDL. For MDL reporting, an appropriate data qualifier is given to samples associated with +/-MB hits between the +/-MDL and +/-PRL where the sample results are less than 10 times the value found in the method blank. Please refer to the attachment section for the flagging charts.

12.6 Matrix Spike/ Matrix Spike Duplicate – A Matrix Spike/Matrix Spike Duplicate (MS/MSD) must be prepared and analyzed for each group of samples that are similar in matrix at a frequency of 5%. The sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD. If one or both spike recoveries are outside 85-115% recovery acceptance criteria, the parent sample is given an appropriate data qualifier. If the RPD is outside 20% RPD criteria the parent sample is given an appropriate data qualifier. Parent sample is chosen at random or assigned by the client.

12.7 All reported results must be within the range of the calibration curve. Dilute when results are greater than the high standard in the curve.

13. METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies,* for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program,* to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

15. REFERENCES

15.1 SW-846 7470A, Revision 1, September 1994

15.2 SW-846 7471A, Revision 1, September 1994

15.3 SW-846 7471B, Revision 2, February 2007

15.4 PASI Quality Manual, current revision

15.5 National Environmental Laboratory Accreditation Conference (NELAC), July 2003 Standards

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ETC.

16.1 Mercury Process Flow Chart

16.2 Flagging chart A: LOD/LOQ Blank Flagging and Reporting

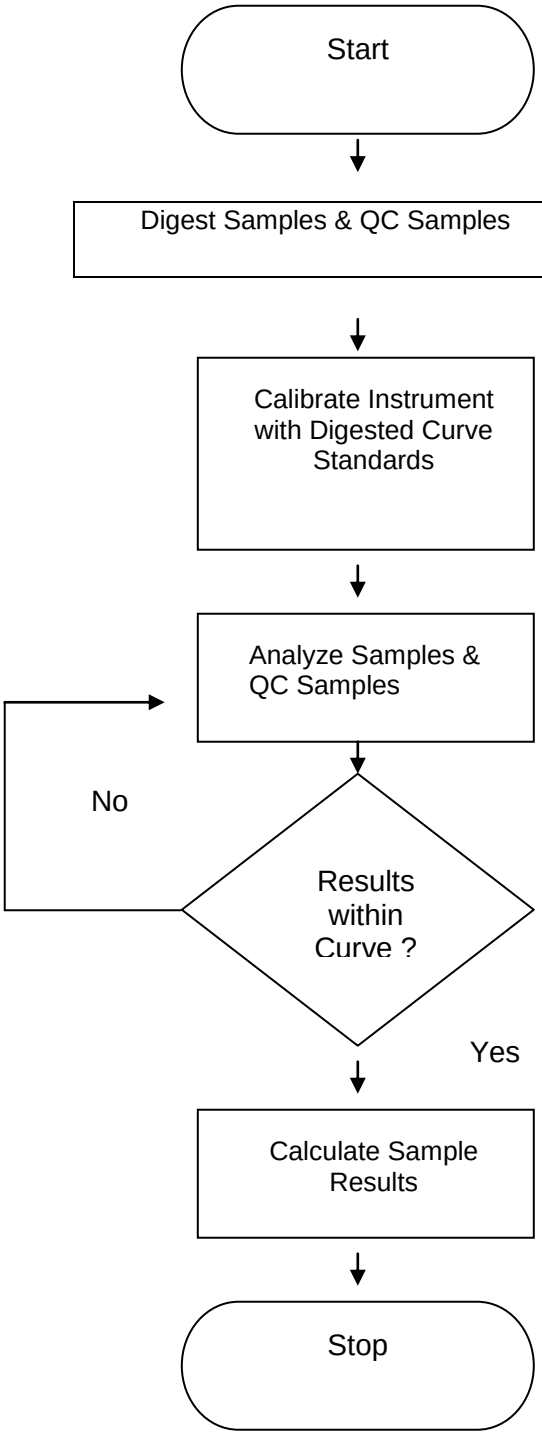
16.3 Flagging chart B: PRL or PSRL Blank Flagging and Reporting

16.4 Flagging chart C: MDL Blank Flagging and Reporting

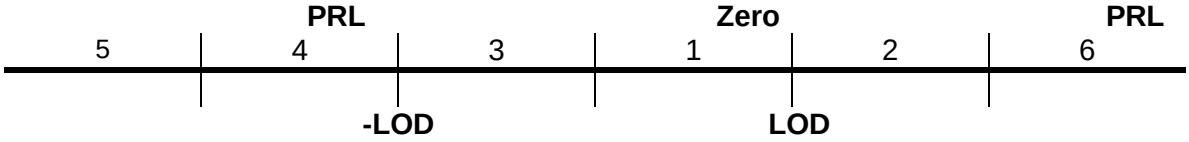
17. REVISIONS

Document Number	Reason for Change	Date
GB-I-036-Rev.0	Combined G2-MET-17 Rev.2 and G2-MET-Rev1 into 1 SOP in new format.	August 28, 2006
GB-I-036-Rev.1	Added Reference to Wipes	December 15, 2006
GB-I-036-Rev.2	Updated Signature Page Updated Section 4.4 - Samples may be diluted to decrease the chlorides Added Section 6.2 Updated Tables 2 and 3. Added Sections 12.1.1.11 and 12.1.1.12 Added Sections 12.1.2.4 and 12.1.2.5 Added Sections 12.2.1, 12.2.3 thru 12.2.10 Updated Section 14	January 2, 2008
S-GB-M-004-REV.00	Updated Method Reference from SW846 7471A to SW846 7471B throughout document. Updated SOP reference to S-GB-M-004-REV.00. Section 4.4 – Added last sentence as alternative Section 5.4.1 – Update SOP reference and name Section 6.2 – Changed biota control blank source from "must" to "should" be farm raised. Section 7 – Deleted Renumbered document Section 10.4 – Added PRLS Table 3 – Added PRLS to HgCal 2 standard Section 12.3 – Added PRLS Section 12.4, 12.5, and 12.6 – Updated flagging language to reflect EpicPro. Section 12.5 – Changed MB contamination from 20 times to 10 times to reflect 7471B requirements. Section 15 – Updated SW-846 7471 reference Flagging Charts A, B, and C – Where ever "A flag" appeared, changed to "appropriately flag". Flagging Charts A, B, and C – Where ever "20 times" appeared, changed to "10 times".	September 12, 2008
S-GB-M-004-REV.01	Updated Signature Page	May 26, 2009
S-GB-M-004-Rev.02	Updated signature page Section 11.1.1.15 – Updated Solution Table 1 – Updated Vendors Tables 2 and 3 – Changed Hg CCV Standard to Hg Cal Intermediate	12Apr2010
S-GB-M-004-Rev.02	Updated Signature Page Added Method 7471A to title Section 15: Updated References (7471A)	14Sept2010

Mercury Process Flow Chart



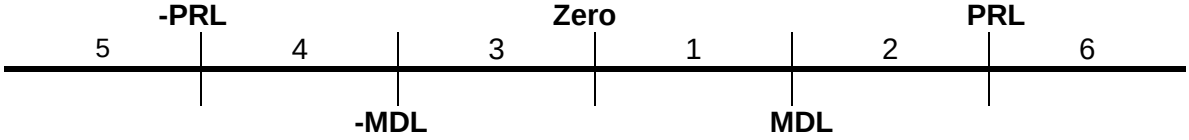
17.1 Flagging Chart A: LOD/LOQ Blank Flagging and Reporting



Blank Position→ Sample Position↓	5	4	3	1	2	6
1	R	Appropriately Flag Report < LOD Comment	Report < LOD	Report < LOD	Report < LOD	Report < LOD
2	R	Appropriately Flag Report result w/10X Rule Comment	Report result	Report result	Appropriately Flag Report result w/10X Rule Comment	R
3	R	Appropriately Flag Report <LOD Comment	Report <LOD	Report <LOD	Report <LOD	Report <LOD
4	R	Appropriately Flag Report <LOD Comment	Report <LOD	Report <LOD	Appropriately Flag Report result w/10X Rule Comment	Report <LOD
5	R	R	R	R	R	R
6	R	Appropriately Flag Report result w/10X Rule Comment	Report result	Report result	Appropriately Flag Report result w/10X Rule Comment	Report result w/10X Rule -or- R if result is not 10X MB

R= Analyst discretion to Re-run, Re-calculate, or Re-digest
X= Value in Method Blank (MB).

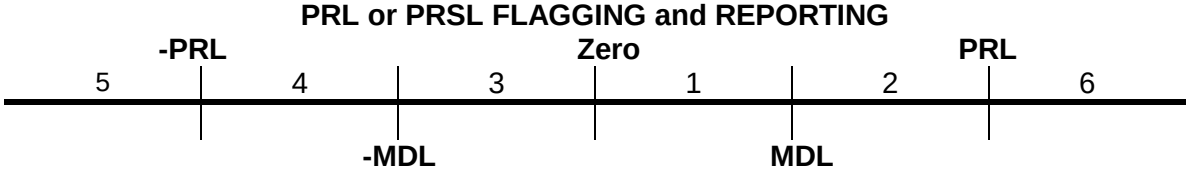
17.2 Flagging Chart B: PRL or PSRL Blank Flagging and Reporting



Blank Position→ Sample Position↓	5	4	3	1	2	6
1	R	Report < PRL	Report < PRL	Report < MDL	Report < PRL	Report < PRL
2	R	Report < PRL	Report < PRL	Report result	Report < PRL	Report < PRL
3	R	Report < PRL	Report < PRL	Report <MDL	Report < PRL	Report PRL
4	R	Report < PRL	Report < PRL	Report < PRL	Report < PRL	Report < PRL
5	R	R	R	R	R	R
6	R	Report result	Report result	Report result	Report result	Report result w/10X Rule -or- R if result is not 10X MB

R= Analyst discretion to Re-run, Re-calculate, or Re-digest
X= Value in Method Blank (MB).
PRL= Pace Reporting Limit

17.3 Flagging Chart C: MDL Blank Flagging and Reporting



Blank Position→ Sample Position↓	5	4	3	1	2	6
1	R	Report < PRL	Report < PRL	Report < PRL	Report < PRL	Report < PRL
2	R	Report Result Appropriately Flag W/ 10X Rule Comment	Report Result	Report Result	Report Result Appropriately Flag W/ 10X Rule Comment	R
3	R	Report < PRL	Report < PRL	Report < PRL	Report < PRL	Report < PRL
4	R	Report < PRL	Report < PRL	Report < PRL	Report < PRL	Report < PRL
5	R	R	R	R	R	R
6	R	Report result Appropriately Flag W/ 10X Rule Comment	Report result	Report result	Report Result Appropriately Flag W/ 10X Rule Comment	Report result Appropriately Flag w/10X Rule -or- R if result is not 10X MB

R= Analyst discretion to Re-run, Re-calculate, or Re-digest
X= Value in Method Blank (MB).
PRL= Reporting Limit

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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.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-M-005-REV.02

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 07/06/11

Kate Grams, Laboratory Quality Manager — Date: 7/1/11

Chad Rusch, Department Manager — Date: 07/05/2011

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 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 (mg/L)
Ag	Silver	328.0	10.0	1.0	0.050
Al	Aluminum	396.1	500.0	50.0	0.500
As	Arsenic	189.0	20.0	2.0	0.050
B	Boron	208.9	100.0	10.0	0.100
Ba	Barium	455.4	5.0	0.50	0.500
Be	Beryllium	313.0	4.0	0.40	0.050
Ca	Calcium	317.9	200.0	20	1.000
Cd	Cadmium	228.8	5.0	0.50	0.005
Co	Cobalt	228.6	5.0	0.50	0.050
Cr	Chromium	267.7	5.0	0.50	0.050
Cu	Copper	324.7	10.0	1.0	0.050
Fe	Iron	259.9	100.0	10.0	0.500
K	Potassium	766.4	200.0	20.0	1.000
Mg	Magnesium	279.0	200.0	20.0	1.000
Mn	Manganese	257.6	5.0	0.50	0.050
Mo	Molybdenum	202.0	20.0	2.0	0.050
Na	Sodium	589.5	1000.0	100.0	1.000
Ni	Nickel	231.6	10.0	1.0	0.050
Pb	Lead	220.3	7.5	1.0	0.0075
Sb	Antimony	206.8	20.0	2.0	0.050
Se	Selenium	196.0	20.0	2.0	0.050
Sn	Tin	189.9	50.0	5.0	0.050
Sr	Stronium	407.7	5.0	0.50	0.050
Ti	Titanium	334.9	5.0	0.50	0.050
Tl	Thallium	190.8	40.0	4.0	0.050
V	Vandaium	292.4	5.0	0.50	0.050
Zn	Zinc	206.2	40.0	4.0	0.050

* 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.

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.

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 4+/- 2°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.	Refrigerate at 4±2 °C Nitric Acid (HNO_3) to pH<2, preserved at time of digestion.	Refrigerate at 4±2 °C Room Temperature	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 that the sample does not have a buffering affect that raises the pH above <2. The data must be qualified as not being properly preserved in the field. (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-2030P (Orange/Blue 0.25mm), PT-2160P (White-White 1.02mm), PT-2120P (Orange/White 0.64mm)	Sample, Internal Standard, and Waste Pump tubing.
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
Plunger Filters	Environmental Express	TRACE LEVEL	SC0408	2.0 µm
Wash Bottle				
pH Strips				
Y Connectors	Hewlett-Packard		G1820-65106	
Duo Torch / Duo Ceramic Cone	Thermo Fisher Scientific		8423-120-51241 / 84223-120-51261	
SeaSpray Nebulizer	Thermo Fisher Scientific		AR30-07-FSS2E	
Spray Chamber/Spray Chamber adapter	Thermo Fisher Scientific		8423-120-51411 / 8423-120-51251	
2mm Center Tube	Thermo Fisher Scientific		8423-120-51371	

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 #
De-ionized Reagent Water (DI)	ASTM Type II, or equivalent (18mOhm or higher resistivity)	Nanopure 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 reagent 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 until they expire per manufacturer's expiration date.
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 reagent 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 volumetric flasks with reagent 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 (**m**L)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	2 ml	25 mL HNO3/ 25 mL HCl	200 ml	1.0 ppm
	Ba	100 ppm	2 ml	"	"	1.0 ppm
	Be	100 ppm	2 ml	"	"	1.0 ppm
	Cd	100 ppm	2 ml	"	"	1.0 ppm
	Co	100 ppm	2 ml	"	"	1.0 ppm
	Cr	100 ppm	2 ml	"	"	1.0 ppm
	Cu	100 ppm	2 ml	"	"	1.0 ppm
	Mn	100 ppm	2 ml	"	"	1.0 ppm
	Ni	100 ppm	2 ml	"	"	1.0 ppm
	Pb	100 ppm	2 ml	"	"	1.0 ppm
	Se	100 ppm	2 ml	"	"	1.0 ppm
	Sr	100 ppm	2 ml	"	"	1.0 ppm
	Tl	100 ppm	2 ml	"	"	1.0 ppm
	V	100 ppm	2 ml	"	"	1.0 ppm
	Zn	100 ppm	2 ml	"	"	1.0 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	4 ml	"	"	2.0 ppm
	Mo	100 ppm	4 ml	"	"	2.0 ppm
	Sb	100 ppm	4 ml	"	"	2.0 ppm
	Sn	100 ppm	4 ml	"	"	2.0 ppm
	Ti	100 ppm	4 ml	"	"	2.0 ppm
	Ag	50 ppm	4 ml	"	"	1.0 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	10 ml	"	"	50 ppm
	Ca	1000 ppm	10 ml	"	"	50 ppm
	Fe	1000 ppm	10 ml	"	"	50 ppm
	K	1000 ppm	10 ml	"	"	50 ppm
	Mg	1000 ppm	10 ml	"	"	50 ppm
	Na	1000 ppm	10 ml	"	"	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	2.5 ml	10 mL HNO3/ 10 mL HCl	500 ml	0.5 ppm
	Ba	100 ppm	2.5 ml	"	"	0.5 ppm
	Be	100 ppm	2.5 ml	"	"	0.5 ppm
	Cd	100 ppm	2.5 ml	"	"	0.5 ppm
	Co	100 ppm	2.5 ml	"	"	0.5 ppm
	Cr	100 ppm	2.5 ml	"	"	0.5 ppm
	Cu	100 ppm	2.5 ml	"	"	0.5 ppm
	Mn	100 ppm	2.5 ml	"	"	0.5 ppm
	Ni	100 ppm	2.5 ml	"	"	0.5 ppm
	Pb	100 ppm	2.5 ml	"	"	0.5 ppm
	Se	100 ppm	2.5 ml	"	"	0.5 ppm
	Sr	100 ppm	2.5 ml	"	"	0.5 ppm
	Tl	100 ppm	2.5 ml	"	"	0.5 ppm
	V	100 ppm	2.5 ml	"	"	0.5 ppm
	Zn	100 ppm	2.5 ml	"	"	0.5 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	5.0 ml	"	"	1.0 ppm
	Mo	100 ppm	5.0 ml	"	"	1.0 ppm
	Sb	100 ppm	5.0 ml	"	"	1.0 ppm
	Sn	100 ppm	5.0 ml	"	"	1.0 ppm
	Ti	100 ppm	5.0 ml	"	"	1.0 ppm
	Ag	50 ppm	5.0 ml	"	"	0.5 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	12.5 ml	"	"	25 ppm
	Ca	1000 ppm	12.5 ml	"	"	25 ppm
	Fe	1000 ppm	12.5 ml	"	"	25 ppm
	K	1000 ppm	12.5 ml	"	"	25 ppm
	Mg	1000 ppm	12.5 ml	"	"	25 ppm
	Na	1000 ppm	12.5 ml	"	"	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 ml	"	"	0.5 ppm
	Be	200 ppm	0.5 ml	"	"	0.5 ppm
	Cd	200 ppm	0.5 ml	"	"	0.5 ppm
	Co	200 ppm	0.5 ml	"	"	0.5 ppm
	Cr	200 ppm	0.5 ml	"	"	0.5 ppm
	Cu	200 ppm	0.5 ml	"	"	0.5 ppm
	Mn	200 ppm	0.5 ml	"	"	0.5 ppm
	Ni	200 ppm	0.5 ml	"	"	0.5 ppm
	Pb	200 ppm	0.5 ml	"	"	0.5 ppm
	Se	200 ppm	0.5 ml	"	"	0.5 ppm
	Sr	200 ppm	0.5 ml	"	"	0.5 ppm
	Tl	200 ppm	0.5 ml	"	"	0.5 ppm
	V	200 ppm	0.5 ml	"	"	0.5 ppm
	Zn	200 ppm	0.5 ml	"	"	0.5 ppm
IV #2 (PA-STD-2B)	B	200 ppm	1.0 ml	"	"	1.0 ppm
	Mo	200 ppm	1.0 ml	"	"	1.0 ppm
	Sb	200 ppm	1.0 ml	"	"	1.0 ppm
	Sn	200 ppm	1.0 ml	"	"	1.0 ppm
	Ti	200 ppm	1.0 ml	"	"	1.0 ppm
	Ag	100 ppm	1.0 ml	"	"	0.5 ppm
IV #3 (PA-STD-3B)	Al	2000 ppm	2.5 ml	"	"	25 ppm
	Ca	2000 ppm	2.5 ml	"	"	25 ppm
	Fe	2000 ppm	2.5 ml	"	"	25 ppm
	K	2000 ppm	2.5 ml	"	"	25 ppm
	Mg	2000 ppm	2.5 ml	"	"	25 ppm
	Na	2000 ppm	2.5 ml	"	"	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	No acid Added	200 ml	50 ppm	500 ppb
	Ba	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Be	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Cd	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Co	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Cr	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Cu	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Mn	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Ni	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Pb	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Se	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Sr	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Tl	200 ppm	25 ml	"	"	50 ppm	500 ppb
	V	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Zn	200 ppm	25 ml	"	"	50 ppm	500 ppb
IV #2 (PA-STD-2B)	B	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Mo	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Sb	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Sn	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Ti	200 ppm	25 ml	"	"	50 ppm	500 ppb
	Ag	100 ppm	25 ml	"	"	25 ppm	250 ppb
IV #3 (PA-STD-3B)	Al	2000 ppm	25 ml	"	"	500 ppm	5000 ppb
	Ca	2000 ppm	25 ml	"	"	500 ppm	5000 ppb
	Fe	2000 ppm	25 ml	"	"	500 ppm	5000 ppb
	K	2000 ppm	25 ml	"	"	500 ppm	5000 ppb
	Mg	2000 ppm	25 ml	"	"	500 ppm	5000 ppb
	Na	2000 ppm	25 ml	"	"	500 ppm	5000 ppb

*** Use 1.0 ml of spiking std to 50 ml**

Table 9.3.5 – Low Level Check Standard (CRDL, RLVS) 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 (ppb)
CPI4400	Ag	10 ppm	0.01 ml	5 mL HNO3/ 5 mL HCl	100 ml	10 ppb
	Al	500 ppm	0.01 ml	"	"	500 ppb
	As	20 ppm	0.01 ml	"	"	20 ppb
	B	100 ppm	0.01 ml	"	"	100 ppb
	Ba	5 ppm	0.01 ml	"	"	5 ppb
	Be	4 ppm	0.01 ml	"	"	4 ppb
	Ca	200 ppm	0.01 ml	"	"	200 ppb
	Cd	5 ppm	0.01 ml	"	"	5 ppb
	Co	5 ppm	0.01 ml	"	"	5 ppb
	Cr	5 ppm	0.01 ml	"	"	5 ppb
	Cu	10 ppm	0.01 ml	"	"	10 ppb
	Fe	100 ppm	0.01 ml	"	"	100 ppb
	K	200 ppm	0.01 ml	"	"	200 ppb
	Mg	200 ppm	0.01 ml	"	"	200 ppb
	Mn	5 ppm	0.01 ml	"	"	5 ppb
	Mo	20 ppm	0.01 ml	"	"	20 ppb
	Na	1000 ppm	0.01 ml	"	"	1000 ppb
	Ni	10 ppm	0.01 ml	"	"	10 ppb
	Pb	7.5 ppm	0.01 ml	"	"	7.5 ppb
	Sb	20 ppm	0.01 ml	"	"	20 ppb
	Se	20 ppm	0.01 ml	"	"	20 ppb
	Sn	50 ppm	0.01 ml	"	"	50 ppb
	Ti	5 ppm	0.01 ml	"	"	5 ppb
	Tl	40 ppm	0.01 ml	"	"	40 ppb
	V	5 ppm	0.01 ml	"	"	5 ppb
	Zn	40 ppm	0.01 ml	"	"	40 ppb
Ultra Scientific ICP-38	Sr	1000 ppm	0.000025 ml	"	"	5 ppb

***A total of X1000 dilution needs to be made off the Stock standard expect for Sr uses a X200,000 dilution. A series of Serial Dilutions may be made in order to accomplish this.**

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	Aluminum	500	10000
Single Element Ca	Ultra Scientific	ICP-120	10	200	Calcium	500	10000
Single Element Mg	Ultra Scientific	ICP-112	10	200	Magnesium	500	10000
Single Element Fe	Ultra Scientific	ICP-126	4	200	Iron	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	Aluminum	475	10000
Single Element Ca	Ultra Scientific	ICP-120	9.5	200	Calcium	475	10000
Single Element Mg	Ultra Scientific	ICP-112	9.5	200	Magnesium	475	10000
Single Element Fe	Ultra Scientific	ICP-126	3.54	200	Iron	175	10000
IV #1 (PA-STD-1B)	Inorganic Ventures	PA-STD-1B	0.5	200	As	0.5	200 ppm
				200	Ba	0.5	200 ppm
				200	Be	0.5	200 ppm
				200	Cd	0.5	200 ppm
				200	Co	0.5	200 ppm
				200	Cr	0.5	200 ppm
				200	Cu	0.5	200 ppm
				200	Mn	0.5	200 ppm
				200	Ni	0.5	200 ppm
				200	Pb	0.5	200 ppm
				200	Se	0.5	200 ppm
				200	Sr	0.5	200 ppm
				200	Tl	0.5	200 ppm
				200	V	0.5	200 ppm
				200	Zn	0.5	200 ppm
IV #2 (PA-STD-2B)	Inorganic Ventures	PA-STD-2B	1.0	200	B	1.0	200 ppm
				200	Mo	1.0	200 ppm
				200	Sb	1.0	200 ppm
				200	Sn	1.0	200 ppm
				200	Ti	1.0	200 ppm
				200	Ag	1.0	100 ppm
IV #3 (PA-STD-3B)	Inorganic Ventures	PA-STD-3B	2.5	200	Al	25	2000 ppm
				200	Ca	25	2000 ppm
				200	Fe	25	2000 ppm
				200	K	25	2000 ppm
				200	Mg	25	2000 ppm
				200	Na	25	2000 ppm
Single Element Ni	Ultra Scientific	ICP-028	0.1	200	Ni	1	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 until they expire per manufacturer's expiration 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-ALL-Q-025 *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 (RLVS)

With every Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) must also be analyzed and meet established acceptance criteria. The RLVS is analyzed prior to any samples being analyzed and at the end of the analytical sequence. Additional RLVSs may be analyzed throughout the analytical sequence at the analyst's discretion. The limits for the RLVS 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.
RLVS 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 RLVS Standard Problems

10.4.4.1 Reanalyze the original RLVS standard to determine instrument consistency.
10.4.4.2 Prepare and analyze a new RLVS standard to determine preparation consistency / standard integrity.
10.4.4.3 Perform instrument maintenance, document in instrument log book
10.4.4.4 Recalibrate instrument according to procedures in Section 10.
10.4.4.5 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 RLVS that is outside criteria may be footnoted or the RLVS 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

10.4.6.1Reanalyze 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 reagent 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 de-ionized reagent water. Place on screw cap and shake thoroughly.
4		Centrifuge or filter (with plunger 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 – 1.2 g for analysis (0.5g dry sample). Weigh the aliquot to the nearest 0.01g and transfer to a digestion vial. Refer to Pace SOP S-ALL-Q-021, *Sub-Sampling*, 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 w*ipe 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 reagent water into digestion vials. 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.

Digestion Program for Soil/ Solid samples by method 3050B

Steps	Reagents[1]	Actions
1	Reagent 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 until the volume is reduced to 5 mLs or 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 de-ionized reagent water. Place screw cap on vials, shake, allow sample to settle, centrifuge at 2000-25000 rpm for 10 minutes, OR and filter with 2.0µm plunger 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.

11.2.7 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.

11.3 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	0.60 L/min
RF Output	1150 W
Pump Speed	40 rpm
Aux Gas Flow	0.5 L/min
Coolant Gas Flow	14 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	RLVS	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	Sample 30
25	Sample 13	51	CCV
26	Sample 14	52	CCB

11.4.2 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 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)	Reagent 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)	Reagent 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 Intregration 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 yearly.

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.

Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in S-GB-S-006, *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

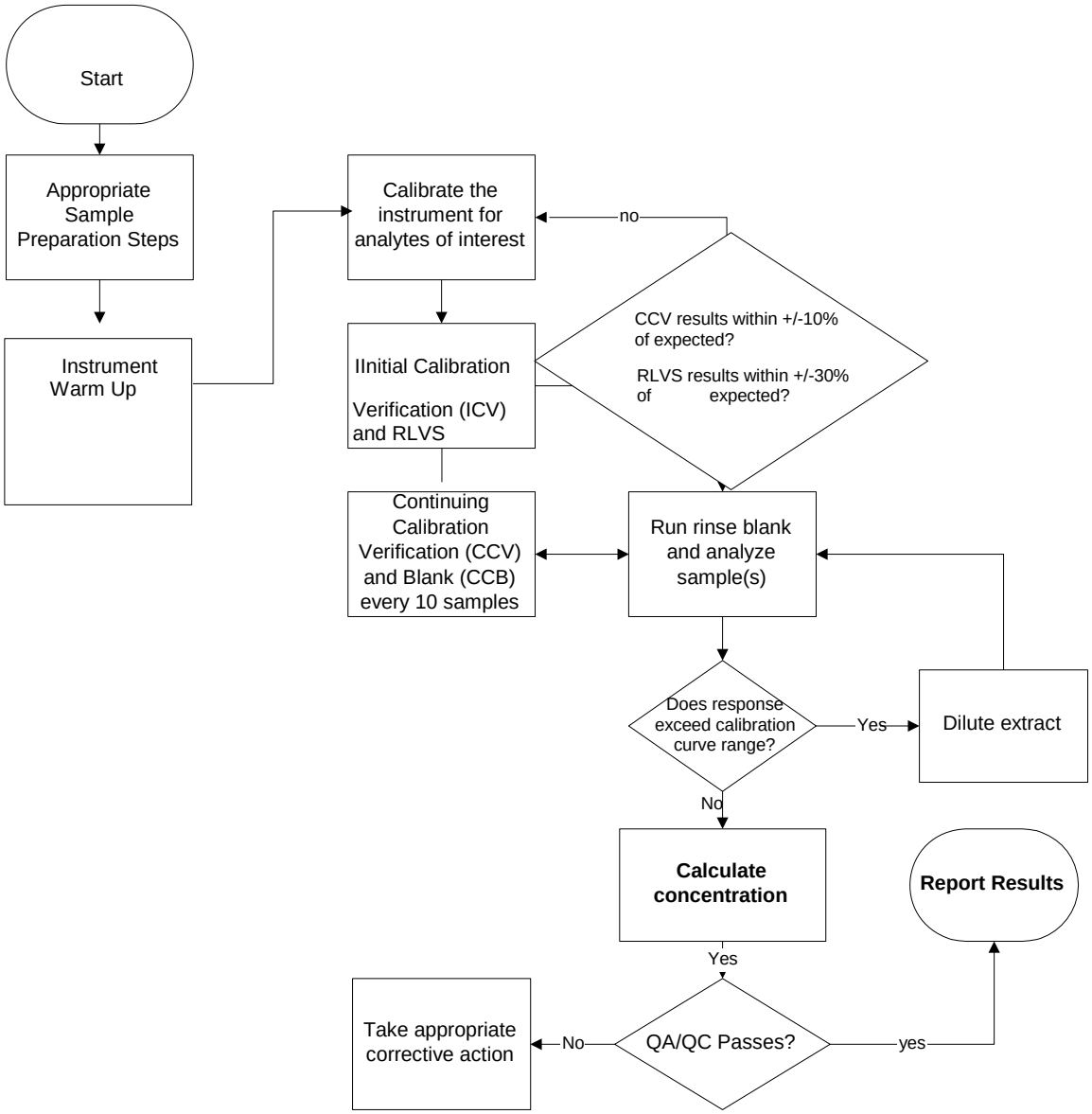
Attachment I

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

Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO

Reference Methods:

WI Modified Method for the Determination of Gasoline Range Organics
SW-846 Method 5030B
SW-846 Method 5035

SOP NUMBER:	S-GB-O-005-REV.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-005-REV.02

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 06/08/2010

Kate Grams, Laboratory Quality Manager — Date: 05/20/2010

Jeff Bushner, Department Manager — Date: 06/08/2010

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 This SOP details the steps and conditions involved in preparing soil/semisolid samples for the determination of gasoline range organics (GRO) and petroleum volatile organic compounds (PVOCs) following The Method for Determining Gasoline Range Organics (Wisconsin DNR 1995).

1.2 Samples prepared by this SOP are analyzed by the latest revisions of SOPs S-GB-O-006 *Modified Method for Determination of Gasoline Range Organics*, S-GB-O-008 *Total Petroleum Hydrocarbons – Gasoline by Gas Chromatography Using Flame-ionization Detectors*, and S-GB-O-009 *Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors*.

2. SCOPE AND APPLICATION

2.1 The policies and procedures contained in this SOP are applicable to all personnel involved in the preparation of soil/semisolid samples for the determination of GRO and PVOCs using a purge and trap, Gas Chromatography procedure.

3. SUMMARY OF METHOD

3.1 Samples are collected in methanol or using En Core™ samplers. Prior to sample introduction to the GC system for analysis, the samples are weighed to determine mass, homogenized physically, and batched for quality control measurements to assess the precision and accuracy of the prep and analytical procedures. It is specified in the method that a maximum weight of 35 grams in a 2 oz jar or 20 grams in a 40 mL VOC vial can be collected and considered valid.

4. INTERFERENCES

4.1 The primary introductions of laboratory interferences come from dirty glassware or impure reagents. Clean laboratory apparatus, quality reagents and conscientious laboratory personnel are essential in the production of high quality data.[1]

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located in Sample Receiving. A formal safety plan has been prepared and is distributed to all personnel with documented training.

[1] Pace Analytical Services, Inc – Green Bay Laboratory purchases high quality solvents to eliminate this potential of contaminations and require these vendors to stand behind claims made on the solvents purchased.

6. DEFINITIONS

6.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Wisconsin has promulgated a 21 day hold time for volatile soil samples preserved in the field with methanol. If an En Core™ is used for sample, the sample must be preserved within 48 hours of collection with methanol.

7.2 After reception in the laboratory, samples are to be stored at $4^{o} \pm 2°$ Celsius at all times when not being prepared.

7.3 A maximum weight of 35.4 grams can be collected and considered valid using an En Core™ sampler, a 2 oz. certified clean jar, or 20.4 grams if collected in a 40 mL VOC vial.

8. EQUIPMENT AND SUPPLIES

8.1 Microsyringes: 10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL:

8.2 Shaker Table

8.3 Syringe, 50 mL, for holding sample aliquot and diluent solution.

8.4 Balance: Analytical, capable of accurately weighing 0.01 g.

8.5 Sonication bath

8.6 40 mL VOC vials for GC autosampler.

8.7 En Core™ samplers

8.8 Computer

8.9 Repipettor, to dispense methanol

8.10 10 mL Graduated Cylinder, to measure dispensed methanol.

9. REAGENTS AND STANDARDS

9.1 Reagent water: Reagent water is defined as water in which an interferent is not observed at the method detection limit of the compounds of interest.

9.2 Methanol (MeOH): Purge and Trap grade. Store away from other solvents.

9.3 10,000 ppm – 10 component Gasoline Range Organics Standard.

9.4 100 µg/mL ααα-Trifluorotoluene surrogate solution added by the autosampler.

10. CALIBRATION

10.1 Not Applicable.

11. PROCEDURE

11.1 The balance is to be leveled before calibration. Calibration verification of the analytical balance is done with S-class weights of 2.0g, 5.0g, 20.0g, and 200.0g. These values are to be noted in the Balance calibration logbook. The frequency of balance calibration verification is once per day before the balance is used or when the balance is moved.

11.2 Zero the balance.

11.3 Place the vial or jar on the balance and write the weight on the vial or jar and in the Soil Prep Logbook.

11.4 In most instances, 40 mL sample vials and 2 oz jars are prepared at Pace Analytical Services Inc. – Green Bay Laboratory and sent to the client prior to sampling. See SOP S-ALL-GB-C-004 *Bottle Preparation* for the specifics on the preparation of sample bottles. (40 mL vials contain 10 mL of Methanol (MeOH) and have an average weight of 25.62 g without MeOH. 2 oz. jars contain 20 mL of MeOH and have an average weight of 87.32 g including MeOH.) If you are unsure of the amount of MeOH or the weight of the client sampling containers, you must contact the Project Manager before proceeding with the samples.

11.4.1 Preserved 40 mL vials generally have a weight range of 32.XX g to 34.XX g. (with 10 mL of MeOH). The specific weight prior to sampling should be recorded on the vial and is a measurement of the vial, cap and MeOH.

11.4.1.1 A 1:1 ratio of soil weight (g) to MeOH volume (mL) is required. The amount of soil in the sample may be calculated as follows for 40 mL vials:

where:

$$W_S = W_T - W_J$$

W_s = Weight of soil in the sample (g)
W_t = Total weight of the sample including vial, cap, soil, and MeOH (g)
W_J = Weight of the jar including the vial, cap, and MeOH (g)

11.4.1.2 The volume of MeOH required to achieve the 1:1 ratio of soil weight (g) to MeOH (mL) may be calculated as follows:

where:

$$V_M = W_S - 10mL$$

V_M = Volume of MeOH required to achieve 1:1 ratio of soil (g):MeOH (mL)
W_S = Weight of soil in the sample (g)
10 = Volume of MeOH initially added (mL)

11.4.2 2 oz jars generally have a weight range of 83.XX g to 87.XX g. (with 20 mL of MeOH). The specific weight of the jar prior to sampling should be recorded on the jar and is a measurement of the jar and cap.

11.4.2.1 A 1:1 ratio of soil weight (g) to MeOH (mL) is required. The amount of soil in the sample may be calculated as follows for 2oz jars:

where:

$W_S = W_T - W_J - 15.8$

W_s = Weight of soil in the sample (g)
W_t = Total weight of the sample including vial, cap, soil, and MeOH (g)
W_J = Weight of the jar including the vial, and cap (g)
15.8 = Weight of 20 mL of MeOH added to the jar (g)

11.4.2.2 The volume of MeOH required to achieve the 1:1 ratio of soil weight to MeOH may be calculated as follows:

where:

$V_M = W_S - 20$ mL
V_M = Volume of MeOH required to achieve 1:1 ratio of soil (g):MeOH (mL)
W_S = Weight of soil in the sample (g)
20 = Volume of MeOH initially added (mL)

11.5 If 1:1 ratio cannot be achieved, write the sample weight and volume of Methanol in the prep logbook, you must contact the Project Manager before proceeding with the samples. If the sample is analyzed, the appropriate data qualifier and comment is entered into LIMS.

11.6 Add the appropriate amount of Methanol using a graduated cylinder or syringe.

11.7 Place prep sheet in the SOILS PREP BOOK.

11.8 Sign over the face of the printout and page to insure removal of page will be noticeable. Date the top of the page of the notebook and sign and date the bottom of the notebook.

11.9 The QC set blank is made for the set, which includes 10 grams of Ottawa sand and 10 mL of methanol placed in a 40 mL VOA Vial.

11.10 The QC set laboratory control spike and laboratory control spike duplicate (LCS/LCSD) are made for the set which includes 10 grams of Ottawa sand, 10 mL of methanol, and 10 µL of the 10,000 ppm revised WISC PVOC/GRO mix standard placed in 40 mL VOA Vial.

11.11 Samples and QC set are then shaken on shaker table for two minutes to pre-homogenize the samples.

11.12 Samples are then placed in the sonicator, which is set at the sixth (6) setting for power and the coldest temperature setting. The samples are sonicated for 20 minutes to finish the homogenization process.

11.13 Samples are pre-screened using a rapid GC headspace technique. See SOP S-GB-O-001, *Sample Screening Volatile Organics Prior to Preparation* for the specifics on the pre-screening of samples.

11.14 A standard analytical dilution is 1:50. Add 1.0 mL of the sample extract measured with a microsyringe of appropriate volume to 49 mL of reverse osmosis water in a 50 mL syringe. Non-standard dilutions are made from the results of the screening analysis.[2]

11.15 To make dilutions other than a standard 1:50 dilution, fill a 50 mL syringe to a volume of 49 mL with reverse osmosis water. Using a 1.0 mL syringe, inject methanol into the 50 mL syringe to bring the total volume of sample and methanol to equal 1.0 mL. Using an appropriate volume syringe, inject the sufficient amount of sample to reach desired dilution. See Table 1 for dilutions.

11.16 The 50 mL syringe contents are place into a 40 mL VOC vial by slowly deploying the plunger and injecting on the side of the vial to eliminate cavitation and loss of analytes to volatilization or sparging. Enough of the contents are injected to created a meniscus at the top of the vial that when capped will produce a no headspace sample.

11.17 The vial is capped and checked for headspace. If vial is free of headspace, it is ready for analysis.

12. QUALITY CONTROL

12.1 A maximum of 20 samples define a QC set. One method blank (MB), laboratory control spike (LCS), laboratory control spike duplicate (LCSD), matrix spike (MS) and matrix spike duplicate (MSD), are necessary per 20 samples OR per extraction batch whichever is more frequent.

12.2 A maximum weight of 35.4 grams can be collected and considered valid using an En Core™ sampler or a 2 oz. certified clean jar, or 20.4 grams if collected in a 40 mL VOC vial.

13. METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDOC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDOC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

[2] On instrument values are to be above 500 ppm GRO. This is the point considered the midpoint of the analysis curve. Samples containing heavy petroleum products can be lower than 500 ppm GRO on column to prevent contamination carryover effects. The cut off is 300 ppm GRO in these instances. The top calibration point is 3000/5000 ppm (instrument dependent) for GRO and 300/500 ppb (instrument dependent) for individual PVOC components. Linear results are possible out to 3000/5000 ppm (instrument dependent) and 300/500 ppb (instrument dependent) respectively.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies,* for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program,* to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management.*

15. REFERENCES

SW-846 5030B Purge and Trap for Aqueous Samples: Revision 2, December 1996

SW-846 5035 Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples. Revision 0, December 1996

Modified GRO, Method for Determining Gasoline Range Organics : Wisconsin DNR, WI-PUB-SW-141, September 1995

Pace Analytical Services, Inc – Green Bay, S-GB-O-006 *Modified Method for Determination of Gasoline Range Organics*

Pace Analytical Services, Inc – Green Bay, S-GB-O-008 *Total Petroleum Hydrocarbons – Gasoline by Gas Chromatography Using Flame-ionization Detectors*

Pace Analytical Services, Inc – Green Bay, S-GB-O-009 *Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors*

Pace Analytical Services, Inc – Green Bay, S-GB-O-001 *Sample Screening Volatile Organics Prior to Preparation*

Pace Analytical Services, Inc – Green Bay, S-ALL-GB-C-004 *Bottle Preparation.*

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

Table 1. Methanol Dilutions (All dilutions are performed in 49 mLs of water in a 50 mL syringe)

Dilutions	Volume of Sample (µL)	Volume of Methanol (µL)
50	1000	0
100	500	500
125	400	600
200	250	750
250	200	800
400	125	880
500	100	900
625	80	920
1000	50	950
1250	40	1000
2000	25	1000
2500	20	1000
4000	12.5	1000
5000	10	1000
6250	8	1000
10000	5	1000
25000	2	1000

17. REVISIONS

Document Number	Reason for Change	Date
GB-O-005-Rev.0	Reformatted SOP body to Pace analytical format	08Apr2005
S-GB-O-005-Rev.1	Up-dated cover page to new format. Up-dated instances of “EnChem” to Pace Analytical Services, Inc. – Green Bay Laboratory, throughout document. Up-dated Section 12 Procedures. Section 12/13 – Up-dated Blank Spike/Blank Spike Duplicate (BS/BSD) to Laboratory Control Spike/Laboratory Control Spike Duplicate (LCS/LCSD) Section 15.2: Up-dated to current SOP nomenclature. Section 16: Updated references	04Dec2007
S-GB-O-005-REV.2	Updated signature page Deleted Section 7. Renumbered document. Sections 11.13 and 13.1.2 – Updated SOP references.	14Oct2008
S-GB-O-005-Rev.03	Updated signature page Section 9: Removed Balance to computer interface and GRO Autoprogram	20May2010

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STANDARD OPERATING PROCEDURE

Total Petroleum Hydrocarbons - Gasoline By Gas Chromatography Using Flame-ionization Detectors

Reference Methods:
SW-846 8015C

SOP NUMBER:	S-GB-O-008-REV.04
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-008-REV.03

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 9/15/10

Kate Grams, Laboratory Quality Manager — Date: 9/15/10

Jeff Bushner, Department Manager — Date: 9/15/10

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 This SOP details the steps and conditions involved in analyzing aqueous, solid, and semi-solid samples for the determination of volatile petroleum hydrocarbons – gasoline following SW-846 8015C. The samples analyzed by this SOP are prepared by the current versions of S-GB-O-005, *Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO*, and S-GB-O-010, *Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics.*

2. SCOPE AND APPLICATION

2.1 This method is used to determine the concentration of various nonhalogenated volatile organic compounds. This method may also by applicable to the analysis of petroleum hydrocarbons, including gasoline range organics (GROs). GROs correspond to the range of alkanes from C_6 to $C_{10,}$ (can vary from C_5 to C_{12} depending on State program) and covering a boiling point range of approximately 60°C - 170°C. A list of applicable compounds and Method Detection Limits (MDL) for aqueous and solid samples is shown in Table A. The identification of specific fuel types may be complicated by environmental processes such as evaporation, biodegradation, or when more than one fuel type is present.

2.2 This method is restricted for use by, or under the supervision of, analysts experienced in the use of gas chromatographs and skilled in the interpretation of gas chromatograms. In addition, if this method is used for the analysis of petroleum hydrocarbons, it is limited to analysts experienced in the interpretation of hydrocarbon data. Each analyst must demonstrate the ability to generate acceptable results with this method.

3. SUMMARY OF METHOD

3.1 This method provides gas chromatographic conditions for the detection of certain volatile petroleum fractions such as gasoline. Samples are analyzed utilizing purge and trap concentrator technology. The use of a temperature program with the GC unit enhances the separation of the organic constituents for which the concentrations are being determined. Detection of the compounds is achieved through the use of PID/FID set in series. The use of PID's identification of various petroleum products is performed by comparison of the chromatograms of samples and commercial products. Quantitation is based on FID response to an external standard response for gasoline. A surrogate standard is quantitated from a PID in series with an FID.

3.2 Soil samples are dispersed in methanol to dissolve the organic constituents. Then an aliquot of this methanol solution is combined with water and analyzed. Aqueous samples are either analyzed in the vial they are received in or a dilution is performed.

3.3 Soil core samples are collected in wide mouth VOC vials, 40 mL VOC vials, or in 25 g Encore samplers. Aqueous samples are collected in 40 mL VOC vials. See section entitled “Sample Collection, Preservation, Shipment and Storage”. Minimum handling is required to reduce TPH loss.

4. INTERFERENCES

4.1 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. Trip blanks are carried through sampling and subsequent storage and handling to serve as a check on such contamination.

4.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and/or purging device must be rinsed between samples with reagent water or solvent. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling compounds or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water, and then dry in a 105°C oven. The trap and other parts of the system are also subject to contamination, therefore, frequent bake-out and purging of the entire system may be required. A screening step is done to protect analytical instrumentation.

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located in Sample Receiving. A formal safety plan has been prepared and is distributed to all personnel with documented training

6. DEFINITIONS

6.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Aqueous samples should be collected in triplicate without agitation and without headspace in contaminant-free 40 mL glass VOC vials with Teflon-lined septa in the caps. The Teflon liner must contact the sample. Samples must be preserved with 500 µL of Hydrochloric Acid (HCl) at the time of collection, (acid should be added to the vial prior to adding the sample). Pace Analytical Services, Inc. uses pre-cleaned, level 3 certified, 40 mL VOC vials purchased from QEC. These vials contain 0.5 mL of 18 % HCL. The samples will need to be cooled to $\leq$6°C after collection. Aqueous samples will be stored in refrigerators at Pace Analytical Services, Inc. and must be held at 4°C (+/- 2°C). Preserved samples need to be analyzed within 14 days from the date of collection. Unpreserved samples need to be analyzed within 7 days from the date of collection.

7.2 Solid samples can be collected with the ENCORE sampler where accrediting authority permits it, or in a 40 mL VOC vial, or 2 oz. jar where local regulations allow. A sufficient number of samplers should be collected to provide for backup analyses in the event of breakage or instrument malfunctions and to allow for screening. One vial should be collected for dry weight determination. One trip blank is required per sampling event (for each site and each day that samples are collected). Solid samples will be stored in refrigerators at Pace Analytical Services, Inc. and must be held at 4°C (+/- 2°C). Samples collected using the ENCORE sampler will be preserved upon receipt to the lab by Pace Analytical Services, Inc. personnel.

7.3 Samples need to be shipped by available carriers as soon as the samples are collected. Sample temperature must be determined upon receipt to the lab. Sample temperature upon receipt is recorded. This is accomplished reading the temperature of a "temperature blank" or melt water in the coolers.

7.4 The pH of aqueous samples must be determined. The pH will be recorded in the analytical run logbook.

7.5 See Appendix A Table G for a summary.

8. EQUIPMENT AND SUPPLIES

8.1 See the following Tables in Appendix A for a summary:

Equipment	Table C
Supplies	Table D

9. REAGENTS AND STANDARDS

9.1 See the following Tables in Appendix A for a summary:

Reagents	Table E
Standards	Table F

10. CALIBRATION

10.1 A marker compound is analyzed prior to the initial calibration to determine the starting and end points of the various hydrocarbon ranges. Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required periodically throughout sample analysis. After initial calibration is successful, a calibration check is required at the beginning and end of each 12-our period during which analyses are preformed, and at requisite intervals (every 10 analytical samples or 12 hours, whichever is more frequent).

10.2 Initial calibration accuracy must be evaluated before any samples are analyzed through the analysis of a Quality Control Sample called an Initial Calibration Verification standard (ICV) which includes all compounds of interest. The ICV should be at or near the midpoint of the calibration range, derived from a source independent of the calibration standard, (i.e., second source), and must quantitate within +/- 20% of the expected value. The ICV concentration is at 1000 ug/l on column for the TPH gasoline fraction.

10.3 Calibration Standards can be found in Table F. Instructions on how to make the calibration standards can be found in Table P. Acceptance Criteria and Frequency of Calibration can be found in Table I.

10.4 Internal standard is added automatically by the auto sampler to each sample and calibration standard for aqueous, air and solid matrices. Surrogate standards are added to soil calibration standards and soil sample extracts at 50 µg/l on column. Surrogate standards are added automatically by the auto sampler to each calibration standard and sample for aqueous matrices at 20 µg/L.

10.5 The vial auto sampler will inject 5 mL of each calibration standard into the sparge tube. The response used for calibration must represent the entire area of the chromatogram within the retention time range for the gasoline range organics. Pace Analytical Services, Inc. will quantitate the area between 0.1 minute before the onset of the 2-Methylpentane peak and ending 0.1 minutes after the conclusion of the 1,2,4-Trimethylbenzene peak for analyses requesting C6 – C10 hydrocarbon range. Pace Analytical Services, Inc. will quantitate the area between 0.1 minute before the onset of the 2-Methylpentane peak and ending 0.1 minutes after the conclusion of the n-Dodecane peak for analyses requesting C6 – C12 hydrocarbon range.

10.6 External calibration technique along with linear regression will be used for quantitation. The response factors are determined by a ratio of amount/area. Amount being the concentration in µg/L and area being the integrated area of the retention time range window. The response factors are then determined by taking the amount/area ratio for each calibration level. Since the Internal Standard and Surrogate standard are added at the same concentrations to each calibration level (and the samples), the areas of the IS/SS are taken into account in the calibration.

10.7 Subtraction of the IS/SS areas is not permissible. Soil extracts diluted greater than the normal x50 dilution will have a portion of the Surrogate standard area diluted out. This area is considered to be insignificant when compared to the total area of the retention time window. Samples diluted greater than the x50 dilution will need to have a response near the upper half of the curve.

10.8 Reporting Limit Verification Standard (RLVS) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, raise the EQL and verify.

10.9 See the following Tables in Appendix A for a summary:

Standards	Table F
Calibration and Standardization	Table I
Directions for Standards	Table P

11. PROCEDURE

11.1 Volatile compounds are introduced into the gas chromatograph by purge-and-trap. Purge-and-trap will be used directly on aqueous samples. Medium level soils, solids and air samples should be analyzed by methanol extraction followed by purge-and-trap. Soil concentrations are reported on a dry weight basis unless local regulations require different.

11.2 The temperature conditions for the DB-624 0.32 mm id columns are as follows:

Parameter	**Setting**	**Duration**
Helium Column Flow	appx. 1.5-2.0 mL/min	
Initial Column Temperature	50° C	Held for 3 minute
Initial Ramp Temperature	10° C/minute	To a temp. of 150° C (No Hold)
Second Ramp	25° C/minute	To a final temperature of 220° C
Hold until five minutes past the elution of 1,2,4-Trimethylbenzene n-Dodecane to reduce carry over effects. Conditions may be altered to improve resolution of individual components		

11.3 Introduce samples into the gas chromatograph using the purge-and-trap method. Adjust the purge gas flow rate (helium) to 40-45 mL/min on the purge-and-trap device. Load the auto sampler rack with samples that correspond with the order written up in the instrument logbook. Samples need to be written in logbooks and keyed into the sample log tables in HPCHEM as follows for automated data processing:

10100001001SA1

Where, 10100001001 = lab number
S = matrix code (S = Soil)
A = test code (A = TPHGAS)
1 = dilution factor, up to a 4 digit dilution factor

11.4 Dilutions requiring more than four digits can be done by using the letter H as a x100 multiplier and the letter K as a x1000 multiplier. Examples are given on the bottom of Appendix A, Table Q. Spaces are not allowed in any of the 16 available character positions. See Table Q for additional codes. The logbook, as well as the auto sampler, will be checked by the analyst performing the analysis. A second analyst will also check the logbook, as well as the auto sampler. Both individuals will initial the sample log table, which is a computer generated hard copy of the analysis written in the logbook. The second analyst will initial the logbook indicating that the auto sampler has been double checked. The initial check of the auto-sampler was done when the samples were loaded.

11.5 Samples not requiring dilutions will be loaded directly into the auto sampler rack. In the event of insufficient vials, or one needs to use the last vial, the analysis should be performed during the shift that an analyst is present. The data will need to be reviewed and reset as needed. 20 mL vials can be filled (no headspace) with sample and stored in case additional analysis are needed. Diluted samples are prepared in 50 mL syringes and then transferred into 40 mL VOC vials for the auto sampler rack. The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delay until the diluted sample is in a gas-tight 40 mL VOC vial.

11.6 Dilutions are made in 50 mL syringes. Choose a syringe that will deliver the proper aliquot for the desired dilution. Adjust the volume in the 50 mL syringe to accommodate the sample aliquot by introducing a bubble into the 50 mL syringe. Inject the aliquot into the 50 mL syringe keeping the syringe needle tip below the surface of the water. Invert the 50 mL syringe three times and transfer the diluted sample to a labeled 40 mL VOC vial. Methanol (MEOH) extracts will contain 2% MEOH or 1mL total MEOH to 49mL of water (x50) which is for clean extracts. Samples requiring larger dilutions will have aliquots of sample and purge and trap grade MEOH to equal 1mL (2 % by volume) total. Aliquots of less than 1 µL are not recommended.

11.7 The vial auto sampler will automatically add the IS/SS mix (or IS mix only) when it transfers the 5 mL of sample to the sparge tube.

11.8 Start the vial auto sampler (see operating and instruction manuals for the different types of auto samplers). The purge time is 11 minutes and will be at ambient temperature for aqueous matrices and soil extracts. The sample syringe is then flushed 1-3 times during purge.

11.9 At the conclusion of the purge time, dry purge the sample for 5 minutes if using a Tekmar 3000 concentrator with a 0.53 mm id DB624 column. Dry purge the sample for 3 minutes for the DynaWaters/Dynatrap systems. There is not a dry purge step for the Tekmar 3000 systems that have 0.32 mm id DB624 column and EPC (Electronic Pressure Control). The purge and trap will step to desorb ready. When the GC is ready (30°C for non EPC systems, 50° C for the EPC systems) the purge and trap will step to desorb. The desorb mode (2 minutes) will begin the gas chromatographic temperature program and GC data acquisition. Concurrently, the trapped materials are introduced to the gas chromatographic column by rapidly heating the trap to 180°C to 250°C, depending on the system, and back flushing the trap with Helium between 20 and 60 mL/min for 2 minutes. The sparge tube is also drained at this time. See Table R for instrument specific purge parameters.

11.10 After desorbing the sample, the purge and trap steps into the bake mode. The vial auto sampler will automatically flush the sparge tube, plumbing lines and sample syringe 3-5 times between samples. The bake cycle reconditions the trap, cleans the plumbing lines, sample syringe and sparge tube. During bake, the trap temperature will be brought to 270°C, while being flushed with helium. The bake cycle is for 7-10 minutes depending on the system. See manufactures suggested bake temperatures and bake times for the different traps and systems used. After bake, the trap cools and the next sample is introduced. Vial auto samplers will continue to cycle through the programmed auto-sampler position. See Table R for instrument specific settings.

11.11 If the initial analysis of a sample or a dilution of the sample has a concentration that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Monitor carry-over by carefully looking at the sample (or samples) immediately following a high range sample. If carryover is suspected, blanks are run and/or the sample(s) affected are reanalyzed if needed.

11.12 All samples that are diluted higher than the normal dilution (x1 for waters and x50 for soil extracts) will require a response near the upper half of the calibration curve. The sample results will be reported if the majority of compounds are greater than 10 times the PQL. Since samples are screened, 500 µg/l is used as the TPH midpoint for gasoline like samples, and 500 µg/l for diesel like or late eluting fuels as guidance. The TPH midpoint for samples received and analyzed under client specific technical requirements is 2500 ug/L.

11.13 Calculations:

11.13.1 Calculations will be performed using linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention.

11.13.2 The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where:

y = Instrument response (peak area or height)
a = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = The intercept

11.13.3 The analyst should not force the line through the origin, but have the intercept calculated from the five data points. In addition, do not include the origin (0,0) as a sixth calibration point. The use of linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.995.

11.13.4 In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$X = (y - b)/a$$

11.13.5 If a linear calibration that does not pass through the origin has been employed, then the regression equation is rearranged as shown above, and the concentration (x) is calculated from the area response (y), the slope (a), and the intercept (b). When using this form of linear calibration, it is the laboratory's responsibility to ensure that the calculations take into account the volume or weight of the original sample, the dilution factor (if any), and dry weight (as applicable). One approach to this calculation is to perform the original linear regression using the concentration in the final extract volume or the volume purged. The concentration in the sample may then be calculated as follows:

where:

$Cs = (C_{ex})(V_t)/V_s$

C_s = Concentration in the sample
C_{ex} = Concentration in the final extract
V_t = Total volume of the concentrated extract
V_s = Volume of the sample extracted or purged

For solid samples, substitute the weight of the sample, W_s, for V_s

12. QUALITY CONTROL

12.1 All quality control data should be maintained and be available for easy reference or inspection.

12.2 The retention time window is determined by analyzing one of the four marker compounds prior to initial calibration. The retention time window is defined as beginning approximately 0.1 minute before the onset of Pentane (C5) or the 2-Methylpentane (C6) peak and ending 0.1 minute after the conclusion of the 1,2,4-Trimethylbenzene (C10) or n-Dodecane peak (C12). The instrument is recalibrated if the fuel standard alkanes exceed the retention times. If the alkanes fall within the retention time windows, the quantitation window is updated to these times for the sample analysis set.

12.3 Before processing any samples, the analyst shall demonstrate through the analysis of a method blank that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank shall be analyzed as a safeguard against chronic laboratory contamination. The blanks shall be carried through all stages of sample preparation and measurement.

12.4 The calibration shall be verified at a frequency of every 12 hours or every 10 analytical samples, whichever is more frequent, with the analysis of a Calibration Verification Standard (CVS). A sequence will begin with the analysis of a CVS followed by a blank. The sequence will end with a CVS.

12.5 A Laboratory Control Spike and Duplicate (LCS/LCSD) are analyzed using the same sample preparations, analytical methods and QA/QC procedures employed for the analytical samples. One LCS/LCSD pair is prepared and analyzed for each sample batch at a frequency of one LCS/LCSD pair for each 20 samples or less for SW846 citations. Limits are attained from the cited method references.

12.6 Documenting the effect of the matrix shall include the analysis of at least one matrix spike/matrix spike duplicate pair. The sample use for the MS/D pair is either determined by the client or selected at random from client samples as sample volume allows. One MS/D pair is prepared and analyzed for each sample batch at a frequency of one MS/D pair for each 20 samples or less for SW846 citations. In-house laboratory limits are developed from samples analyzed during the previous year. A minimum of 40 spike samples' recoveries, 10 from each of the previous years four quarters will represent the data pool. The laboratory must evaluate surrogate recovery data from individual samples. All samples prepared and analyzed by this method are spiked with surrogate standard. Limits are established the same as matrix spikes described above. All quality control data should be maintained and be available for easy reference or inspection.

12.7 See Appendix A Table H for a summary

13. METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDOC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDOC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:

15.1 At a minimum there are three levels of data review:

15.2 Analyst/Technician verification at the bench top are looking at instrument performance as it relates to initial calibration, calibration verification, cleanliness.

15.3 Supervisor/Analyst verification after analysis are looking for sample concentration versus linear range of the instrument, typical patterns resulting from the compounds or elements in question, and quality control measure criteria. Data is either accepted without qualification, accepted with qualification, or rejected with samples reprocessed.

15.4 Report Reviewer/Project Management personnel look at the presentation of the data on final reports. The reports are verified for holding time compliance, receipt conditions, method citation and reasonableness, which may include field duplicate analysis, analytical comments and qualifications presented by the previous reviews, and comparing results of similar analytical techniques as it relates to the project site information when available.

15.5 A fourth level of review, data validation, is determined from a project or program scope of laboratory services and encompasses the generation of Quality Control summaries in the media of "Form Generation".

15.6 See the following Tables in Appendix A for a summary:

Analyst/Technician	Table K
Supervisor/Analyst	Table L
Report Reviewer/Project Management	Table M

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 Assessment of Quality Control measures is done to provide a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis.

16.2 See Table N for corrective actions taken in cases where statistical goals were not achieved.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, with received sample volumes, data generated along with measures that do not meet statistical goals are re-analyzed again to see if the statistical goal can be achieved. When environmental samples do not meet statistical goals, unacceptable data is generated. These events are different from those pertaining to instrument operating conditions. These events occur when the instruments are operating under ideal conditions.

17.2 The tables list routine assessment and not those specific to a project/program where data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation",

17.3 Contingencies for handling sample out-of-control quality control assessments are found in Table O.

18. REFERENCES

Method SW846 8000C Revision 3 March 2003

Method SW846 8015C Revision 3 February 2007

Pace Analytical Services, Inc – Green Bay, S-GB-O-005, *Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO*

Pace Analytical Services, Inc – Green Bay, S-GB-O-010, *Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics*.

19. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

Appendix A
Tables

Table A **Reporting Limits**
Table B **Required Safety Equipment**
Table C **Equipment**
Table D **Supplies**
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Table J **Matrix Spike Control Limits**
Table J1 **Blank Spike Control Limits**
Table J2 **Surrogate Standard Control Limits**
Table K **Analyst/Technician Data Assessment**
Table L **Analyst/Supervision Data Assessment**
Table M **Report Reviewer/Project Management Data Assessment**
Table N **Corrective Actions**
Table O **Contingencies for Handling Out of Control Data**
Table P **Directions for Standards**
Table Q **Additional Codes for Automated Data Transfer**
Table R **Purge Parameters**

Table A
REPORTING LIMITS

COMPOUND NAME	AQUEOUS		SOLIDS (MEOH EXTRACT)	
	MDL* (ug/L)	EQL (ug/L)	MDL* (mg/Kg)	EQL (mg/Kg)
TPH-Gasoline (C6-C10)	49.0	100	4.0	10
TPH C5-C12	49.0	100	4.0	10
TPH C6-C12	49.0	100	4.0	10
*Method Detection Limit (MDL) is subject to change.				

Table B
REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses	X	
2	Lab Coat		X
3	Gloves		X
4	Face Shield		X

Table C
EQUIPMENT

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
40GCV1	Hewlett Packard	5890A Series II	3310A48054	3/92
40GCV2	Hewlett Packard	5890A Series II	3140A39241	6/93
40GCV3	Hewlett Packard	5890A Series II	3203A40701	6/93
40GCV4	Hewlett Packard	5890A Series II	3336A57281	4/95
40GCV5	Hewlett Packard	5890A Series II	3310A47921	7/95
P&T # 1	Tekmar	Tekmar 3000	94005013	6/02
P&T # 2	Tekmar	Tekmar 3000	94067018	1/04
P&T # 3	Tekmar	Tekmar 3000	95296017	4/95
P&T # 4	Tekmar	Tekmar 3000	99042009	6/00
P&T # 5	Tekmar	Tekmar 3000	95198009	9/00
Auto Sampler # 1	DynaTech	Archon 5100A	11911-496A	6/02
Auto Sampler # 2	DynaTech	Archon 5100A	11950-696A	5/95
Auto Sampler # 3	Varian	Archon	12561	12/98
Auto Sampler # 4	DynaTech	Archon 5100A	11930-596A	4/03
Auto Sampler # 5	Varian	Archon	13300	9/00

Table D
SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #
10µL Gastight 1701	Hamilton	Fisher Scientific	14-815-1
25µL Gastight 1702	Hamilton	Fisher Scientific	14-815-29
50µL Gastight 1705	Hamilton	Fisher Scientific	14-824-30
100µL Gastight 1710	Hamilton	Fisher Scientific	13-684-100
250µL Gastight 1725	Hamilton	Fisher Scientific	13-684-102
500µL Gastight 1750	Hamilton	Fisher Scientific	13-684-106
1mL Gastight 1001	Hamilton	Fisher Scientific	14-824-25
5mL Gastight 1005	Hamilton	Fisher Scientific	13-684-96
50mL Gastight 1050	Hamilton	Fisher Scientific	14-815-195
DB-624 Capillary column, 30m X 0.32mm id X 1.8 um film	J&W Scientific	VWR Scientific	123-1334
DB-624 Capillary column, 30 m X 0.53 mm id X 3 um film	J&W Scientific	VWR Scientific	125-1334
Purge Trap K for Tekmar 3000 Concentrator	Supelco	Supelco	24920-U
Fritless 5-mL Sparge Tube	Supelco	Supelco	22780
IceBlue Septa, 11mm	Restek	Restek	22392
0.8mmVespel/Graphite ferrules	Restek	Restek	20213
1/16" x 0.5 mm ID Vespel / Graphite ferrules	Restek	Restek	20231
Stir Bar	Fisher Brand	Fisher Brand	14-511-60A
2 oz. jars	QEC	QEC	2114-0002
40 mL VOA vials	QEC	QEC	3112-40HCL
40 mL VOA vials	QEC	QEC	3112-40mL
10 mL volumetric	Kimax Brand	Fisher Scientific	10-212AA
25 mL volumetric	Kimax Brand	Fisher Scientific	10-212BB
50 mL volumetric	Kimax Brand	Fisher Scientific	10-212A
100 mL volumetric	Kimax Brand	Fisher Scientific	10-212B
200 mL volumetric	Kimax Brand	Fisher Scientific	10-212C
500 mL volumetric	Kimax Brand	Fisher Scientific	10-218D
Pasteur Pipettes	Fisher Scientific	Fisher Scientific	13-678-20A
Spatula with spoon	Fisher Scientific	Fisher Scientific	14-375-10
Pipette bulb	Fisher Scientific	Fisher Scientific	14-065B
0.1-2.5 mL Repipetter	Brinkmann	Fisher Scientific	13-688-130
1-5 mL Repipetter	Brinkmann	Fisher Scientific	13-688-131
2-10 mL Repipetter	Brinkmann	Fisher Scientific	13-688-133
5-25 mL Repipetter	Brinkmann	Fisher Scientific	13-688-134
1.8 mL amber vials & caps	Restek	Restek	24637
10 mL graduate cylinder	Kimex Brand	Fisher Scientific	08-554B "to deliver"
10 mL pipette "to deliver" (TD)	Kimex Brand	Kimex Brand	13-6644G
pH Indicator Paper	Whitman	M&G Scientific	2613991

Table E
REAGENTS

Reagent	Purity	Concentration	Manufacturer	Vendor	Catalog #
Unleaded Gasoline Composite Standard (Curve Standard)	99%	50,000 ppm	Restek	Restek	30205
Gasoline Composite Standard (Verification Standard)	GC/FID Tech Mix	5000 ppm	AccuStandard, Inc.	AccuStandard, Inc.	AK101.0 – GCS
N-Dodecane	GC/MS	5.0 mg/mL	AccuStandard, Inc.	AccuStandard, Inc.	AS-E0238
Fluorobenzene (IS)	99.9 %	20 mg/mL	AccuStandard Inc.	AccuStandard Inc.	M-524-IS-2-10X
α,α,α-Trifluorotoluene (SS), (TFT)	99.4 %	20 mg/mL	AccuStandard Inc.	AccuStandard Inc.	M-602-Ss-100X
Ottawa Sand Standard	ASTM C-190 and Method T-132	20-30 Mesh	Fisher Scientific	Fisher Scientific	S23-3
Methanol	Purge & Trap Grade		Burdick & Jackson	Burdick & Jackson	232-1
Reverse Osmosis Water (ROW)	Organic Free	---	Flowmatic	Water Care	

Table F
STANDARDS

Standard	Acronym	Concentration	Direction found in Section:	Alias
Internal Standard and Surrogate Standard Mix	IS/SS	100 ppm	Table P	
Internal Standard Only	IS Only	100 ppm	Table P	
Surrogate Spike Solution	100 ppm SS	100 ppm	Table P	
Surrogate Spike Soln.	2500 ppm SS	2500 ppm	Table P	
Surrogate Spike Soln.	1000 ppm SS	1000 ppm	Table P	
Calibration Verification Standard (Water)	CVSGAS	1000 ug/l gas on column	Table P	Water Gas Check, CCV
Calibration Verification Standard (MeOH)	CVSGAS	1000 ug/l gas on column	Table P	MeOH Gas Check, CCV
Laboratory Control Spike (Water)	LCS/LCSD	1000 ug/l on column	Table P	
Laboratory Control Spike (MeOH)	LCS/LCSD	50 mg/kg gas (1000 ug/l on column)	Table P	
Matrix Spike (Water)	MS/MSD	1000 ug/l on column	Table P	
Matrix Spike (MeOH)	MS/MSD	50 mg/kg gas (1000 ug/l on column)	Table P	
Level 1 Calibration Standard (water) and RLVS.	CAL STD	100 ppb gas	Table P	Level 1, C100
Level 2 Calibration Standard (water)	CAL STD	200 ppb gas	Table P	Level 2, C200
Level 3 Calibration Standard (water)	CAL STD	500 ppb gas	Table P	Level 3, C500
Level 4 Calibration Standard (water)	CAL STD	1000 ppb gas	Table P	Level 4, C1000
Level 5 Calibration Standard (water)	CAL STD	2000 ppb gas	Table P	Level 5, C2000
Level 6 Calibration Standard (water)	CAL STD	5000 ppb gas	Table P	Level 6, C5000
Level 1 Calibration Standard (MeOH) and RLVS.	CAL STD	200 ppb gas	Table P	Level 1, C200
Level 2 Calibration Standard (MeOH)	CAL STD	500 ppb gas	Table P	Level 2, C500
Level 3 Calibration Standard (MeOH)	CAL STD	1000 ppb gas	Table P	Level 3, C1000
Level 4 Calibration Standard (MeOH)	CAL STD	2000 ppb gas	Table P	Level 4, C2000
Level 5 Calibration Standard (MeOH)	CAL STD	5000 ppb gas*	Table P	Level 5, C5000

1 Some instruments are linear only to 3000 ppb, so will substitute this concentration for the 5000 ppb standard.

Table G
SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Aqueous	SW846 5030B	40 mL VOC Vials (3)	0.5 mL of 18 % HCL	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
Solid	SW846 5030B	4 oz ZH packed tight 40 mL VOC Vials (3) 25 g Encore	Refrigerate 4+/- 2° Celsius*	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius

- Methanol preservation acceptable with Accrediting Authority approval.

Table H
QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	SW846 8015C
Initial Calibration	Minimum of five levels; lowest level near but above MDL
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Calibration Verification Standard (CVS)	1 CVS at the beginning of an analytical sequence and 1 CVS per 12 hour time clock or 10 samples, whichever is more frequent. The sequence will end with a CVS.
Initial Calibration Verification (ICV)	The ICV is from a second source independent from the calibration standard used to verify the initial calibration curve.
Reporting Limit Verification Standard (RLVS)	A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Internal Standards	Added to every sample.
Surrogate Standards	Added to every sample during extraction step.

Table I
CALIBRATION AND STANDARDIZATION

Analytical Method Acceptance Criteria⇨ Calibration Measure ⇩	**Frequency**	**Acceptance Criteria**
Initial Calibration	• Installation of new column • Installation of new PID • Installation of new FID or jet • Installation of new Purge Trap • Creation of new analytical method • CVS fails criteria	• Linear Regression and Correlation Coefficient is > 0.990.
Independent Calibration Verification Standard	• Analyzed after initial calibration to verify the calibration with a second source standard.	• ICVS must be ≤ 20% difference from initial calibration.
Reporting Limit Verification Standard (RLVS)	• It is analyzed after the calibration and monthly thereafter	• RLVS recovery must be 60-140% of true value.
Continuing Calibration Verification	• See Table H	• CVS must be ≤ 20% difference from initial calibration.
Laboratory Control Spike and Duplicate	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	• See Table J1 under Lab Control Spike Recovery Limits
Matrix Spike and Duplicate	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	• See Table J under Matrix Spike Recovery Limits
Internal Standards	• Added to every sample.	• Internal Standard area limits established from the area of the first CVS starting a sequence or method. Limit is 50%-100%
Surrogate Standards	• Added to every sample.	• In house limits determined. See Table J2 under Surrogate recovery Limits

Table J
Matrix Spike Recovery Limits

Preparation Method ⇨ **Effective Date:11/20/2007**	**SW846 5030B Aqueous**			**SW846 5030B Solid**		
Matrix Spike Recovery Parameters ⇩	**LCL***	**UCL***	**RPD***	**LCL***	**UCL***	**RPD***
TPH-Gasoline (C6- C10)	66	124	20	64	131	20
C6 – C12	80	120	20	64	123	20
C5 – C12	80	120	20	70	120	20

* Control limits are subject to change at any time.

Table J1
Laboratory Control Spike Recovery Limits

Preparation Method ⇨ **Effective Date:11/20/2007**	**SW846 5030B Aqueous**			**SW846 5030B Solid**		
Lab Control Spike Recovery Parameters ⇩	**LCL***	**UCL***	**RPD***	**LCL***	**UCL***	**RPD***
TPH-Gasoline (C5 – C10)	80	120	20	80	120	20
C6 – C12	80	120	20	78	120	20
C5 – C12	80	120	20	80	120	20

* Control limits are subject to change at any time.

Table J2
Surrogate Standard Recovery Limits

Preparation Method ⇨ **Effective Date: 11/20/2007**	**SW846 5030B Aqueous**		**SW846 5030B Solid**	
Surrogate Spike Recovery Parameters ⇩	**LCL***	**UCL***	**LCL***	**UCL***
α,α,α,Trifluorotoluene	80	120	76	124

* Control limits are subject to change at any time.

Table K
ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method **Acceptance Criteria⇨** **Data Assessment Measure ⇩**	**Method Citation SW** **If these conditions are not achieved ⇒**
Initial Calibration	• 1
Initial Calibration Verification	• 1
Calibration Blank	• 1
Method Blank	• 2
Internal Standard Area	• 3
Continuing Calibration Verifications	• 4
Surrogate Recovery	• 5
Accuracy & Precision Laboratory Control Spikes.	• 6
Accuracy & Precision Matrix Spikes	• 7

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. Samples that are "non-detect", or have detects greater than 20 times the method blank contamination level are reported without qualification. Sample detects less than 20 times the method blank contamination are re-analyzed or qualified. Samples with a detect less than the EQL are reported. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non-Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows, report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not allow for re-analysis, the parent sample shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table L
ANALYST/SUPERVISOR DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**
Initial Calibration	• 1
Initial Calibration Verification	• 1
Calibration Blank	• 1
Method Blank	• 2
Internal Standard Area	• 3
Continuing Calibration Verifications	• 4
Surrogate Recovery	• 5
Accuracy & Precision Laboratory Control Spikes	• 6
Accuracy & Precision Matrix Spikes	• 7

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo. Samples that are "non-detect", or have detects greater than 20 times the method blank contamination level are reported without qualification. Sample detects less than 20 times the method blank contamination are re-analyzed or qualified. Samples with a detect less than the EQL are reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows, report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user.

5. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table M
REPORT REVIEWER/PROJECT MANAGEMENT DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**
Holding Time Compliance	• 1
Sample Receipt Conditions and Preservation	• 2
Method Citation	• 3
Reasonableness: Field Duplicate	• 4
Reasonableness: Analytical Comments Qualifications	• 5

1. EpicPro flags results with appropriate H qualifier.
2. In prose describe the receipt conditions as they relate to the acceptance criteria list in Table G
3. Compare to regulatory program in QAPP or Chain of Custody.
4. Are duplicate with 40% agreement. If not, internally flag for review.
5. Do related suites of tests agree or are reasonable.

Table N
CORRECTIVE ACTIONS

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
Initial Calibration		• 1
Reporting Limit Verification Standard		• 2
Method Blank		• 3
Continuing Calibration Verification		• 4

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration.

A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

3. Samples that are "non-detect", or have detects greater than 20 times the method blank contamination level are reported without qualification. Sample detects less than 20 times the method blank contamination are re-analyzed or qualified. Samples with a detect less than the EQL are reported.

4. Immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV.

Table O
CONTINGENCIES FOR HANDLING
OUT-OF-CONTROL OR UNACCEPTABLE DATA

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**Method Citation SW846 If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
Laboratory Control Spike and Duplicate	•	• 1
Matrix Spike and Duplicate	•	• 2
Internal Standards	•	• 3
Surrogate Standards	•	• 4

1. If Laboratory Control Spike/Duplicate fail on water analysis, the entire batch is re-analyzed with new QC, sample volume permitting. Inadequate sample volume results in flagging with appropriate qualifier all associated samples. If Laboratory Control Spike/Duplicate fail on MeOH analysis, the LCS/LCSD is reanalyzed to confirm failure. Confirmation of failure results in flagging with appropriate qualifier all associated samples. If non-confirmation of failure, reanalyze all samples with the valid QC.

2. If Laboratory Control Spikes pass acceptance criteria, flag parent sample with appropriate qualifier to indicate matrix effects.

3. When sample volume is available reanalyze the samples at a higher dilution. If the second analysis indicates the same internal standard recoveries add analytical comments and effects on data.

4. When sample volume is available reanalyze the samples. If the second analysis indicates the same surrogate standard recoveries add analytical comments and flag with appropriate qualifier to demonstrate the matrix effects on data.

TABLE P DIRECTIONS FOR STANDARDS

Standard	Acronym	Concentration	Reagents Used	Final Volume
Internal Standard and Surrogate Standard Mix	IS/SS	100 ppm	1000 µL of 20 mg/mL Fluorobenzene + 1000 µL of 20 mg/mL α,α,α-Trifluorotoluene to 198 mL MeOH	200 mL (MeOH)
Internal Standard Only	IS Only	100 ppm	1000 µL of 20 mg/mL α,α,α,Trifluorotoluene to 199 mL MeOH	50 mL (MeOH)
Surrogate Spike Solution	100 ppm SS	100 ppm	50 µL of 20 mg/mL α,α,α,Trifluorotoluene to 9.95 mL MeOH	10 mL (MeOH)
Surrogate Spike Soln.	2500 ppm SS	2500 ppm	1250 µL of 20mg/mL α,α,α,Trifluorotoluene to 8.750 mL MeOH	10 mL (MeOH)
Surrogate Spike Soln.	1000 ppm SS	1000 ppm	50 µL of 20 mg/mL α,α,α,Trifluorotoluene to 950 µL MeOH	10 mL (MeOH)
2000 ppm Intermediate Gasoline Standard	2000 ppm Gas	2000 ppm	400 µL of 50,000 ppm Unleaded Gasoline Composite Stock Standard	10 mL (MeOH)
500 ppm Intermediate Gasoline Curve Standard	500 ppm Gas Curve Standard	500 ppm	100 µL of 50,000 ug/mL to 9.9 mL MeOH	10 mL (MeOH)
500 ppm Intermediate Gasoline Verification Standard (ICV)	500 ppm Gas ICV Standard	500 ppm	1000 µL of 5.0 ug/mL to 9.0 mL MeOH	10 mL (MeOH)
Calibration Verification Standard (Water)	CVSGAS	1000ug/l gas on column	100 µL of 2000 ppm Intermediate gas to 200 mL RO WATER	200 mL RO WATER
Calibration Verification Standard (MeOH)	CVSGAS	1000 ug/l gas on column	100 µL of 2000 ppm Intermediate gas + 100 µL of 100 ppm α,α,α,Trifluorotoluene to 196 mL of RO WATER containing 3.8 mL MeOH	200 mL RO WATER
Laboratory Control Spike (Water)	LCS/LCSD	1000 ug/l on column	100 µL of 2000 ppm Intermediate gas to 200 mL RO WATER	200 mL RO WATER
Laboratory Control Spike (MeOH)	LCS/LCSD	50 mg/kg gas (1000 ug/l on column)	250 µL of 2000 ppm gas + 10 µL of 2500 ppm α,α,α,Trifluorotoluene to 10 g Ottawa Sand and 10 mL MeOH	10 mL MeOH 10 g Sand
Matrix Spike (Water)	MS/MSD	1000 ug/l on column	21.5 µL of 2000 ppm Intermediate gas to 43 mL sample in 40 mL VOC vial	43 mL
Matrix Spike (MeOH)	MS/MSD	50 mg/kg gas (1000 ug/l on column)	250 µL of 2000 ppm gas + 10 µL of 2500 ppm α,α,α,Trifluorotoluene to 10 g Sample and 10 mL MeOH	10 mL MeOH 10 g Sample
Level 1 Calibration Standard (water) and RLVS	CAL STD	100 ppb gas	10 µL of 500 ppm Intermediate Gasoline Curve Standard to 50 mL RO WATER	50 mL RO WATER
Level 2 Calibration Standard (water)	CAL STD	200 ppb gas	20 µL of 500 ppm Intermediate Gasoline Curve Standard to 50 mL RO WATER	50 mL RO WATER
Level 3 Calibration Standard (water)	CAL STD	500 ppb gas	50 µL of 500 ppm Intermediate Gasoline Curve Standard to 50 mL RO WATER	50 mL RO WATER
Level 4 Calibration Standard (water)	CAL STD	1000 ppb gas	100 µL of 500 ppm Intermediate Gasoline Curve Standard to 50 mL RO WATER	50 mL RO WATER
Level 5 Calibration Standard (water)	CAL STD	2000 ppb gas	200 µL of 500 ppm Intermediate Gasoline Curve Standard to 50 mL RO WATER	50 mL RO WATER
Level 6 Calibration Standard (water)	CAL STD	5000 ppb gas	500 µL of 500 ppm Intermediate Gasoline Curve Standard to 50 mL RO WATER	50 mL RO WATER
Or Level 6 Calibration Standard (water)	CAL STD	3000 ppb gas	300 µL of 500 ppm Intermediate Gasoline Curve Standard to 50 mL RO WATER	50 mL RO WATER
C5 Marker Compound (One year expiration date)	C5 Mark (Pentane)	50 ppb	50 µL of 100 ug/mL Pentane to 100 mL RO WATER	100 mL RO WATER
C6 Marker Compound (One year expiration date)	C6 Mark (2-Methylpentane)	75 ppb	5 µL of 10,000 ppm Tenn GRO to 100 mL RO WATER	100 mL RO WATER
C10 Marker Compound (One year expiration date)	C10 Mark (1,2,4-TMB)	50 ppb	5 µL of 10,000 ppm Tenn GRO to 100 mL RO WATER	100 mL RO WATER
C12 Marker Compound	C12 Mark (n-Dodecane)	50 ppb	2 µL of 5.0 ug/mL n-Dodecane to 100 mL RO WATER	100 mL RO WATER

TABLE P (CONT.)
DIRECTIONS FOR STANDARDS

Standard	Acronym	Concentration	Reagents Used	Final Volume
Level 1 Calibration Standard (MeOH) and RLVS	CAL STD	200 ppb gas	20 µL of 500 ppm Intermediate Gasoline - curve standard + 25 µL of 100 ppm α,α,α,Trifluorotoluene + 950 µL of MeOH to 49 mL RO WATER	50 mL RO WATER
Level 2 Calibration Standard (MeOH)	CAL STD	500 ppb gas	50 µL of 500 ppm Intermediate Gasoline - curve standard + 25 µL of 100 ppm α,α,α,Trifluorotoluene + 920 µL of MeOH to 49 mL RO WATER	50 mL RO WATER
Level 3 Calibration Standard (MeOH)	CAL STD	1000 ppb gas	100 µL of 500 ppm Intermediate Gasoline - curve standard + 25 µL of 100 ppm α,α,α,Trifluorotoluene + 880 µL of MeOH to 49 mL RO WATER	50 mL RO WATER
Level 4 Calibration Standard (MeOH)	CAL STD	2000 ppb gas	200 µL of 500 ppm Intermediate Gasoline - curve standard + 25 µL of 100 ppm α,α,α,Trifluorotoluene + 780 µL of MeOH to 49 mL RO WATER	50 mL RO WATER
Level 5 Calibration Standard (MeOH)	CAL STD	5000 ppb gas or 3000 ppb gas	500 µL of 500 ppm Intermediate Gasoline - curve standard + 25 µL of 100 ppm α,α,α,Trifluorotoluene + 480 µL of MeOH to 49 mL RO WATER, or 300 µL of 500 ppm Gasoline - Regular, Unleaded (curve standard) + 25 µL of 100 ppm α,α,α,Trifluorotoluene + 680 µL of MeOH to 49 mL RO WATER	50 mL RO WATER

Table Q
DATA TRANSFER CODES

4010001001WA1

Letter occupying position of "W"

S SOIL ON SOLID SAMPLE

W AQUEOUS

I IMPINGER

C CHARCOAL TUBE

M METHANOL BLANK

L LOW LEVEL SOIL

MEDIUM LEVEL EXTRACTABLE SOLID IF LOGGED IN –S

Letter occupying position of "A"

A GRO, TPHGAS, OA-1 ONLY

B BENZENE ONLY

C BTEX W/ MTBE

D BTEX

E BTEX &MTBE W/ NAPH

F MTBE ONLY

G NAPH ONLY

H PVOC

I PVOC W/ NAPH

J BTEX W/ MTBE 602

K BTEX 602

L PVOC 602

N BTEX W/ NAPH

P BTEX W/ TOTAL XYLENES

R BTEX &MTBE W/ TOTAL XYLENES

if lower case no rear FID analysis

NUMBER occupying position of "1"

DILUTION FACTOR (up to 4 digits)

10K = 10,000 (10 x 1000)
1HK = 100,000 (1 x 100 x 1000)
10HK = 1,000,000 (10 x 100 x 1000)

TABLE R
PURGE AND TRAP OPERATING PROCEDURES

Analysis Method (Current Edition of 8015B)

Parameter	Tekmar 3000
Trap	Purge Trap K For Tekmar 3000
Trap contents	Carbopack B/Carboxen 1000 & 1001
Column (DB-24, 30m)	0.32mm ID x 1.8um film
Electric Pressure Control (EPC)	Yes
Purge Gas	He
Purge Gas Flow Rate (mL/Min)	40-45
Purge Time (min)	11
Purge Temp	Ambient
Desorb Temp (C)	250
Desorb Time (min)	2
Dry Purge Time (min)	None
Bake Temp (C)	270
Back flush Gas Flow (mL/min)	20-60

20. REVISIONS

Document Number	Reason for Change	Date
GB-O-008-Rev.0	Reformatted SOP body to Pace analytical format	11Apr2005
S-GB-O-008-Rev.1	Updated cover page to new format Section 2.1 - Added carbon ranges C6 to C10, C5 to C12, and C6 to C12. Section 3.3 – Added 40mL VOC Vials Section 6.1 - Updated Definitions source Section 11.2 – Defined ICV as second source, replacing CVS Section 11.5 – Updated Carbon ranges Section 12.4 – Updated auto-processing code Section 12.5 – Updated dilution criteria Section 13.2 – Defined retention time window Section 13.4 – Replaced BS/BSD with LCS/LCSD throughout document as necessary. Section 14 – Updated with current SOPs in use Section 15 – Updated with current SOPs in use Section 19 – Updated references Tables – Updated all as necessary	27Nov2007
S-GB-O-008-Rev.02	Updated Method Reference to SW –846 8015C throughout document. Deleted Section 7. Renumbered document. Section 7.4 – Updated ph recording Section 10.2 – Changed ICV recovery requirement from +/- 15% to +/-20%. Section 10.8 – Added Reporting Limit Verification Standard and requirements. Section 11.4 – updated to Epic Pro procedure Section 11.13 – Added midpoint requirement for BP samples Section 13.1.2 – updated SOP reference and name. Section 18 – updated 8015C reference Table I – Changed ICVS and CVS recovery limits from $\leq$ 15% to $\leq$ 20%. Table M – Updated item 1 to EpicPro. Tables F, H, I, N, and P – added Reporting Limit Verification Standard. Tables J, J1, and J2 – Control limits subject to change caveat added. Table O – Updated items 2 and 4. Table Q - Updated	9Sept2008
S-GB-O-008-Rev.03	Updated Signature Page Section 11.1, Table D/R: Removed information pertaining to Dynatrap and Tekmar 3000 “A” Section 11.2: Updated column information to 0.32 mm ID information. Table C: Removed Gas#6 Instrument information. Updated to Epic Pro instrument naming convention i.e., GAS#1 to 40GCV1 Through-out document: Removed references to client specific technical documents.	20May2010
S-GB-O-008-Rev.04	Section 10.1: Added marker compound criteria. Section 11.13.1: Updated R^2 to 0.995 from 0.99. Table P: Added C5, C6, and C10 marker compounds, added one-year expiration date.	15Sept2010

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STANDARD OPERATING PROCEDURE

Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics

Reference Methods:
WI Modified Method for the Determination of Gasoline Range Organics
SW-846 Method 5030B

SOP NUMBER:	S-GB-O-010-REV.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-010-REV.02

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 06/08/2010

Kate Grams, Laboratory Quality Manager — Date: 05/24/2010

Jeff Bushner, Department Manager — Date: 06/08/2010

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 This SOP details the steps and conditions involved in preparing aqueous samples for the determination of gasoline range organics (GRO) and petroleum volatile organic compounds (PVOCs) following The Method for Determining Gasoline Range Organics (Wisconsin DNR 1995) and SW-846 5030B.

1.2 Samples prepared by this SOP are analyzed by the latest revisions of SOPs S-GB-O-006 *Modified Method for Determination of Gasoline Range Organics*, S-GB-O-008 *Total Petroleum Hydrocarbons – Gasoline by Gas Chromatography Using Flame-ionization Detectors,* and S-GB-O-009 *Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors.*

2. SCOPE AND APPLICATION

2.1 The policies and procedures contained in this SOP are applicable to all personnel involved in the preparation of aqueous samples for the determination of GRO and PVOCs using a purge and trap, Gas Chromatography procedure

3. SUMMARY OF METHOD

3.1 Water samples are collected and stored at $4^{o}C \pm 2°C$ in an area free of solvent fumes. All samples should be analyzed within 14 days of collection for preserved samples; 7 days for unpreserved. Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by dilution of the sample prior to the purge-and-trap process. Screening of the samples prior to purge-and-trap analysis may provide guidance on whether sample dilution is necessary. Pace routinely screens samples to eliminate exceedances of the calibration curve and to protect sensitive instruments from damage.

4. INTERFERENCES

4.1 The primary introduction of laboratory interference comes from dirty glassware or impure reagents. Clean laboratory apparatus, quality reagents and conscientious laboratory personnel are essential in the production of high quality data.[1]

[1]Pace Analytical Services, Inc. – Green Bay Laboratory purchases high quality solvents to eliminate this potential of contaminations and requires these vendors to stand behind claims made on the solvents purchased.
Pace Analytical Services, Inc. – Green Bay Laboratory has adopted and implemented SOP's addressing proper glassware cleaning techniques, tailored to the department using the apparatus.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water is carried through sampling and handling protocols to serve as a check on such contamination

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located in Sample Receiving. A formal safety plan has been prepared and is distributed to all personnel with documented training

6. DEFINITIONS

6.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Aqueous samples should be collected in triplicate without agitation and without headspace in contaminant-free 40 ml glass VOC vials with Teflon-lined septa in the caps. The Teflon liner must contact the sample. Samples must be preserved with 500 µL of Hydrochloric Acid (HCl) at the time of collection, (acid should be added to the vial prior to adding the sample).

Note: Pace Analytical Services, Inc. – Green Bay Laboratory uses pre-cleaned, level 3 certified, 40 ml VOC vials purchased from QEC. These vials contain 0.5 ml of 18 % HCL.

Note: If targeted compounds breakdown with the addition of HCl, samples must be collected unpreserved. Unpreserved samples have a 7-day holding time. The pH of the samples are taken using pH indicator paper and recorded in a logbook.

7.2 The samples will need to be cooled to 4° C after collection. Aqueous samples will be stored in refrigerators at Pace Analytical Services, Inc. – Green Bay Laboratory and must be held at 4° C (+/- 2C). Preserved samples have a 14-day holding time or as specified by the Accrediting Authorities.

7.3 One trip blank is required per sampling event and site.

7.4 Samples need to be shipped to the lab as soon as the samples are collected.

7.5 Sample temperature must be determined upon receipt to the lab.

Note: Sample temperature is recorded by use of a temperature blank. The temperature must be between 4° C (+/- 2C) unless superseded by the accrediting authority. For certain authorities recording sample temperature as "received on ice (ROI)" is acceptable.

8. EQUIPMENT AND SUPPLIES

8.1 Microsyringes: 10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL:

8.2 Syringe: 50 mL, for holding sample aliquot and diluent solution.

8.3 40 mL VOC vials for GC autosampler.

9. REAGENTS AND STANDARDS

9.1 Reverse Osmosis Water

9.2 10,000 ppm – 10 component Gasoline Range Organics Standard from Restek or Supelco.

9.3 100 ug/mL ααα-Trifluorotoluene surrogate solution added by the autosampler.

10. CALIBRATION

10.1 Not Applicable.

11. PROCEDURE

11.1 Batch samples in groups of 20 or less using the WIGROW A-code in EpicPro. Use the Batch Candidate Report in EpicPro to batch similar compound requests together. To obtain this report, key in “Systems” > “Submit Job” > “F9” > “Select Batch Candidates” > “Enter” or “OK”, type in “GCV” for the Queue and “WIGROW” for the Batch Rule > “F10” (save). Print the report using “Print” in EpicPro toolbar.

11.2 Batch the samples by selecting “Batching” > “New Batch” > “GCV” in the Queue field of the “Advanced Find” Window > “Enter” or “OK”. Double click the WIGROW A-code in the “Rule” column. Click the “Clear Selection” button, which will de-select the first 20 samples so you can select the samples you want from the Batch Candidate Report.

11.2.1 After selecting the samples, click the “Build Batch” button. This will put the sample in a unique Batch with its own Batch number and default QC samples with their own QC number IDs. If necessary, add QC by using the “Add QC” button. The cursor must be on a particular sample to add a MS/MSD.

11.2.2 Deleting QC or samples is done by putting the cursor on what you want to delete and clicking the red "Delete Record" button in the Toolbar Menu. Press "F10" (save) when all is done. Print the newly created Batch Work list report using the Print button in the EpicPro toolbar. Select "All" for all pages to print.

11.2.3 Print the Batch Compound report for the analyst by going "System" > "Submit Job" > "F9" > "Batch Compound List" > "Enter" or "OK" > Select "GCV" as Queue and enter the Batch number > "F10". Print using the Print command in the EpicPro toolbar, select "All" > "OK". This will give the analyst a report of analytes to report per sample.

11.2.4 Pull samples from storage refrigerator.

11.3 Higher concentration samples are pre-screened using a rapid GC headspace technique. See the latest revision of SOP S-GB-O-001 *Sample Screening Volatile Organics Prior to Preparation* for the specifics on the pre-screening of samples.

11.4 Dilute samples according to screen data.

11.4.1 Samples that screen clean can be analyzed directly from the original sample vials free of headspace.

11.4.2 Dilutions of higher concentration samples are made based on the results of the screen data.

11.5 If the samples are not to be analyzed immediately, store the set in the appropriate section of the sample refrigerator.

11.6 For each analytical batch, not to exceed 20 environmental samples, a method blank, matrix spike and matrix spike duplicate must be analyzed (the frequency of the spikes may be different for different monitoring programs but no less than 1 per 20 samples). The blank and spiked samples must be carried through all stages of the sample preparation and measurement steps. (This can be superseded by Accrediting or Project Specific Requirements.) The sample used for the MS/D pair is either determined by the client or selected at random from client samples, as sample volume allows. See latest revisions of SOPs S-GB-O-006 *Modified Method for Determination of Gasoline Range Organics*, S-GB-O-008 *Total Petroleum Hydrocarbons – Gasoline by Gas Chromatography Using Flame-ionization Detectors*, and S-GB-O-009 *Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors* for spike concentration and instructions.

11.7 A Laboratory Control Spike and Duplicate (LCS/LCSD) pair is also included with each analytical batch. The pair consists of an aliquot of reverse osmosis water. The pair is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, these results are used to verify that the instrument was in control. See latest revisions of SOPs S-GB-O-006 *Modified Method for Determination of Gasoline Range Organics,* S-GB-O-008 *Total Petroleum Hydrocarbons – Gasoline by Gas Chromatography Using Flame-ionization Detectors,* and S-GB-O-009 *Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors* for spike concentration and instructions.

11.8 All diluted samples, the blank, laboratory control spike, laboratory control spike duplicate, matrix spike and matrix spike duplicate must be free of headspace.

11.9 Print a VOA Water Prep Sheet by going "System" > "Submit Job" > "F9" > select "VOA Water Prep Sheet" > "Enter" or "OK", key in "GCV" for Queue and the Batch number > "F10". Print using EpicPro "Print" in the toolbar.

11.10 Fill in the dilution factors from the screen data and tape this sheet into the Water Prep Logbook.

11.11 Sign over the face of the printout and page to insure removal of page will be noticeable. Date the top of the page of the notebook and sign and date the bottom of the notebook.

12. QUALITY CONTROL

12.1 A maximum of 20 samples define a QC set. One method blank, laboratory control spike, laboratory control spike duplicate, matrix spike and matrix spike duplicate are necessary per 20 samples or per extraction batch whichever is more frequent.

12.2 After reception in the laboratory, samples are to be stored at $4^{o}C \pm 2°C$ at all times when not being prepared.

12.3 All water samples that are preserved should be analyzed within 14 days of collection or 7 days for unpreserved water samples.

12.4 The size of any bubble caused by degassing upon cooling the sample should not exceed 5-6 mm. When a bubble is present, also observe the cap and septum to ensure that a proper seal was made at the time of sampling. If the sample was improperly sealed or if the bubble exceeds 5-6mm, the sample should not be used for analysis. If insufficient sample vials are received, the sample is analyzed and the appropriate flag is entered into LIMS.

13. METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDOC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDOC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

15. REFERENCES

SW-846 5030B Purge and Trap for Aqueous Samples, Revision 2, December 1996

Modified GRO, Method for Determining Gasoline Range Organics, Wisconsin DNR, WI-PUB-SW-141, September 1995

Pace Analytical Services, Inc – Green Bay, S-GB-O-006 *Modified Method for Determination of Gasoline Range Organics*

Pace Analytical Services, Inc – Green Bay, S-GB-O-008 *Total Petroleum Hydrocarbons – Gasoline by Gas Chromatography Using Flame-ionization Detectors*

Pace Analytical Services, Inc – Green Bay, S-GB-O-009 *Aromatic Volatiles by Gas Chromatography Using Photo-ionization Detectors*

Pace Analytical Services, Inc – Green Bay, S-GB-O-001 *Sample Screening Volatile Organics Prior to Preparation*

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

16.1 Not applicable

17. REVISIONS

Document Number	Reason for Change	Date
G3-GRO-7 Rev. 2	Reformatted SOP body to Pace analytical format	12Apr2005
S-GB-O-010-Rev.1	Updated cover page to new format Section 8 - Replaced instances of EnChem to Pace Analytical Services, Inc. – Green Bay Laboratory Section 12.1.5 - Updated Procedure section Section 12 and 13 - Replaced BS/BSD with LCS/LCSD throughout document as necessary	27Nov2007
S-GB-O-010-Rev.02	Updated signature page Deleted Section 7. Renumbered document Section 11.1, 11.2, 11.9, & 11.10 – Updated with EpicPro procedure. Section 11.6 – Added SOPs Section 11.7 – Added SOPs Section 13 - Updated	19Nov2008
S-GB-O-010-Rev.03	Updated Signature Page	16Apr2010

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STANDARD OPERATING PROCEDURE

TOTAL PETROLEUM HYDROCARBONS

Reference Method: SW-846 Method 8015B

SOP NUMBER:	S-GB-O-023-REV.05
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-023-REV.04

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 07/07/11

Kate Grams, Laboratory Quality Manager — Date: 7/7/11

Chris Haase, Department Manager — Date: 07/07/11

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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17. Revisions....15

1. PURPOSE

1.1 The purpose of this Standard Operating Procedure (SOP) is to provide a consistent format for the determination Total Petroleum Hydrocarbons in Water and Soil. This includes #2 Fuel Oil. This covers a boiling point range of approximately 170^0C - 430^0C.

2. SCOPE AND APPLICATION

2.1 The policies and procedures contained in this SOP are applicable to all personnel involved in the planning, coordination, preparation, use and revision of SOPs.

2.2 The Practical Quantitation Limit (PQL) of this method for Total Petroleum Hydrocarbons is approximately 1.7 mg/kg for soils and 50 ug/L for groundwater.

2.3 This method is based on a solvent extraction, Gas Chromatography (GC) procedure. This method should be used by, or under supervision of, analysts experienced in solvent extraction and the use of gas chromatographs. The analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

2.4 The method is designed to measure mid-range petroleum products. Components greater than C28 present in products such as motor oils or lubricating oils are detectable under the conditions of the method. If, based on a review of the chromatograph the presence of these product types is suspected, additional analyses may be performed.

3. SUMMARY OF METHOD

3.1 This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as diesel, fuel oil #2, or kerosene. Samples are analyzed utilizing extraction to dissolve the organic constituents. The extract is dried, concentrated and injected into a capillary column gas chromatograph. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. Detection and quantitation is based on FID detector response compared to a TPH component standard.

3.2 This method is suitable for the analysis of waters, soils, or wastes.

3.3 This method is based in part on 1) USEPA SW-846: the 3rd edition of methods 8000 and 8100; 2) Method OA-2; 3) work by the EPA Total Petroleum Hydrocarbons Methods Committee; and 4) work by the Wisconsin Ad-Hoc Committee on LUST Program Analytical Requirements and Wisconsin State Laboratory of Hygiene.

3.4 Detection Limits

3.4.1 See Table A in Section 16.

4. INTERFERENCES

4.1 Raw GC data from all blanks, samples, and spikes must be evaluated for interferences. Determination of the source of the contamination is in the preparation and/or cleanup of the samples and takes the appropriate action to resolve these matters.

4.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with a solvent. Whenever a highly concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Reduce exposure by use of gloves, lab coats and safety glasses. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training.

6. DEFINITIONS

6.1 Refer to Section 10 of the Pace Quality Manual.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Aqueous samples are collected in 1 Liter amber bottles that are equipped with Teflon lined caps and are preserved with 5mL hydrochloric acid (HCl). All samples are transported on ice and stored at a temperature of 4 ± 2°C. Extraction must be performed within 7 days of the collection date.

7.2 Soil samples are collected in 2 oz. clear jars with Teflon lined caps. They are filled completely to meet the requirement of zero-headspace. The samples are then transported on ice and stored at 4 ± 2°C. Extraction must be performed within 14 days of the collection date.

7.3 Sample extracts are stored below 0 degrees Celsius. All extracts are to be analyzed within 40 days of the extraction date. The extracts are then stored for a minimum of 40 days after the date of sample analysis.

7.4 See Table F for a summary.

8. EQUIPMENT AND SUPPLIES

8.1 See Table B and Table C for a summary

9. REAGENTS AND STANDARDS

9.1 Reagent Water: Barnstead Nanopure Infinity Ultra pure water system.

9.2 Solvents: Burdick and Jackson methylene chloride pesticide grade or equivalent.

9.3 Stock Standards: Stock standard for the #2 diesel components in methylene chloride Diesel Fuel #2 Composite Standard (50000 µg/ml) obtained from Restek.

9.3.1 See Table E for a summary.

9.3.2 Transfer the stock standard solution into a Teflon-sealed screw-cap/crimp cap bottle. Store, with minimal headspace, at -10 C to -20 C and protect from light.

9.3.3 Standards must be replaced after 1 year, or sooner if comparison with check standards indicates a problem.

9.4 Calibration Standards: Prepare Calibration standards at a minimum of five concentration levels in solvent from the Diesel Fuel #2 Standard (20000 µg/ml) obtained from Supelco. One of the concentration levels should be around the MDL. The remaining concentration levels should correspond to the working range of the GC.

9.5 See Tables D and E for a summary.

10. **CALIBRATION**

10.1 TPH calibration

10.1.1 Run the Fuel Standard at least five concentration levels at and above the EQL and covering the linear range of the instrument. These levels are 50, 100, 250, 500, 1000 and 2000 ug/ml. Calibration standards are prepared by diluting the standard stock solution (20000 µg/ml) from Supelco with methylene chloride in 1, 2 and 10mL volumetric flasks. The stock standard is injected with a microsyringe below the surface of the methylene chloride in the volumetric and then the volumetric is diluted to the mark. The calibration standards are transferred to 2 mL vials with disposable pipettes. The vials are sealed with Teflon-lined aluminum crimp seals.

10.1.2 Inject each calibration standard. Tabulate peak area for the fuel range against the mass injected. The results are used to prepare a calibration curve by linear regression. The correlation coefficient must be >0.99.

10.2 Initial calibration accuracy must be evaluated before any samples are analyzed through the analysis of an Initial Calibration Verification standard (ICV) which includes all compounds of interest. The ICV should be at or near the midpoint of the calibration range, derived from a source independent of the calibration standard, (i.e., second source), and must quantitate within +/- 20%of the expected value. The ICV concentration is at 500 ug/l for the TPH-diesel fraction using 50 µl of Diesel Fuel #2 Standard (50,000 µg/mL).

10.3 The working calibration curve must be verified every 12 hours or less. The 12-hour period begins with the injection of the calibration verification standard. The period ends after the completion of the analysis of the final standard that can be injected within 12 hours of the beginning of the shift. If the response for the CVS varies from the predicted response by more than 15%, a new calibration curve must be prepared. The working range of the calibration curve is from 0-2000 ug/mL. The calibration check is at a concentration of 500 mg/L.

10.4 Reporting Limit Verification Standard (RLVS) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

11. **PROCEDURE**

11.1 Samples are analyzed by GC/FID. Waters are extracted using a separatory funnel technique (see SOP S-GB-O-053 *Sep Funnel Extraction)*. Soils are extracted using microwave extraction (see SOP S-GB-O-045 *Microwave Extraction for PAH, BNA and TPH Solids)*. After the extracts are concentrated, a volume is injected directly onto the GC.

11.2 Gas Chromatography

11.2.1 Conditions: Set column temperature to 50 °C for 1 minutes, then 250 °C C/min. to 320 °C and hold for 2 minutes. Set FID Detector to 280 °C and injector to 280°C.

NOTE: Other conditions can be used.

11.3 Retention Time Window and Quantitation: The retention time range is defined during initial calibration. The range is established from the retention times of the C_{10} and C_{28} alkanes. The retention time range is then calculated based on the lower limit of the RT window for the first eluting component and the upper limit of the RT window for the last eluting component. The retention time for the quantitative window is updated to the time of the daily CVS.

11.4 Calculations

11.4.1 The concentration of specific fuels in the sample is determined from a summation of peak area for all chromatographic peaks corresponding to a particular fuel in a window established by the calibration standards. From linear regression of calibration standard GC response (R) against their known concentrations (C in ug/mL) derive the following linear equation:

$$C = mR + b$$

Using the slope (m) and the intercept (b) from this equation the concentration of the sample can be calculated from the following equations:

Water samples

$$Cs = [(mRs + b) (Ve) (D)] / Vs$$

Soil samples

$$Cs = [(mRs + b) (Ve) (D)] / W$$

Where:

Cs = Concentration of sample in ug/L for waters and mg/kg on a dry weight basis for soils

m = slope of the calibration curve

Rs = GC response of sample in the DRO retention time window

b = intercept of calibration curve

Ve = total volume of sample extract (after concentration) in mL.

Vs = volume of water sample in liters

D = dilution factor of water or soil extract as diluted

W = total dry weight of soil sample in gm

11.5 Pace Analytical Services, Inc. has automated data processing. The integrated results are automatically processed against the linear regression curve, transferred to LIMS and then corrected for extraction volume, sample weight or sample volume and percent solids for soil samples. Assign the samples to run/runs and mark analyzed. If necessary, enter correct sample weights and percent solids. Review prep and analysis dates to be sure they are correct.

11.5.1 The initial result off the GC, the total solids, dilution factor and the weight of the sample are sent to LIMS through the autoprogram. The final volume of 1.0 mL is preset in LIMS. The initial volume for waters is preset at 1.0 L. If a sample is not blown down to 1.0 mL or a water sample is not 1.0 L, these parameters must be changed manually in LIMS.

The LIMS calculation.

IA=Initial amount - Value off the GC-transferred automatically in mg/L units.

D=Dilution factor - Transferred automatically.

V=Final volume - Set at 1.0 mL (Must be changed manually if different.)

W=Weight - Soil weight in grams is transferred automatically. For waters initial volume is set at 1.0L. (Must be changed manually if not 1.0L.)

S=SOL% - Total solids is transferred automatically for soils. SOL% is set at 100% for waters

TPH water result = (IA*D*V)/W Results are in ug/L with reporting to 2.0 significant figures.

$$\frac{(mg/L)}{mL} = \frac{(mg * mL)}{L^2} * \frac{L}{1000ml} * \frac{1000ug}{mg} = \frac{ug}{L}$$

TPH soil result = (IA*D*V)/(W*S) Results are in mg/kg with reporting to 2.0 significant figures.

$$\frac{mg}{L} * \frac{1.0}{g} * \frac{mL}{1.0} * \frac{1.0\ L}{1000mL} * \frac{1000g}{1.0\ kg} = \frac{mg}{kg}$$

11.5.2 Peak areas measured from blanks may not be subtracted from sample peak areas. Peaks from blank samples, which interfere in the window, and are associated with column bleed, are subtracted.

11.5.3 Comments on the chromatogram and/or other problems regarding the sample should be entered into the LIMS comment section and the reports.

12. QUALITY CONTROL

12.1 The analyst must make an initial demonstration of the ability to generate acceptable accuracy and precision with this method by successful analysis of the following:

12.1.1 Replicate Water spikes. Analysis of 4 replicates water samples at a concentration of 500 ug/L with recoveries between 70%-130% of the known concentration. The RSD must be <30%.

12.1.2 Replicate Soil spikes. Analysis of 4 replicates at a concentration of 33.3 mg/kg with all recoveries between 70% and 130% of the known concentration. The RSD must be <30%.

12.2 With every batch of 20 samples or less the lab must analyze the following:

12.2.1 Laboratory Control Spike - The RPD and replicate spikes must be within limits established by the laboratory using past recoveries. If insufficient sample is received to perform a MS/MSD, a LSCD must be extracted for precision criteria.

12.2.2 Matrix Spike and Matrix Spike Duplicate. The spike recovery and RPD must be within the limits established by the lab using past recoveries. Matrix spike samples are spiked in exactly the same way as LABORATORY-CONTROL SPIKE samples except environmental samples are used. The sample used for the MS/D pair is either determined by the client or selected at random from client samples as sample volume allows

12.2.3 Calibration Standard. - The peak area must fall within 15% of the value predicted by the calibration curve. A 500 ug/mL standard is run before any samples or QC.

12.2.4 Solvent Blank – A Methylene Chloride blank is used to determine column bleed.

12.2.5 Method Blank - Water: Purified water is processed through the method in the same manner as a sample. If the concentration exceeds the reporting limit, the samples associated with batch must be rerun or flagged.

12.2.6 Method Blank - Soil: Ottowa sand is processed through the entire extraction procedure. If the concentration exceeds the reporting limits the sample associated with that batch must be flagged or rerun.

12.2.7 Surrogates: All surrogate recoveries for CVS's, QC samples, and client samples must be within the laboratory's QC control limits. These limits are calculated by the laboratory using past surrogate recoveries. If surrogate recoveries are not within control limits in the CVS, the samples must be rerun after appropriate corrective action has taken place. If surrogate recoveries are not within control limits in the QC samples or samples, the QC samples and samples must be re-extracted and re-analyzed. If there is insufficient sample volume to re-extract, the data must be appropriately qualified.

12.2.8 The correlation coefficient of the calibration curve used to quantitate samples must be at least 0.9900.

12.2.9 If any of the criteria above are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those that have fallen out should be rerun. If this is not possible, that data must be flagged.

12.2.10 Solvent blanks should be run after samples suspected of being highly concentrated to prevent carryover.

12.2.11 Reporting Limit Verification Standard (RLVS) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

12.2.12 See Tables G and H for a summary.

13. METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-20, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.2.1 Analysis of four (4) replicates of Ottawa sand spiked with TPH Spiking Solution.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

13.2 A series of 4 spikes in 1.0L of water are extracted and analyzed before a method is put online. This is also the case whenever a new analyst is running this method. Method performances must be done at least once a year.

13.3 Method Modifications:

Method Modifications for SW846 8015B are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- 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.
- Procedures identified as ”Best Practices” by PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of ALL-S-002, *Waste Handling*.

15. REFERENCES

Modified DRO, Method for Determining Diesel Range Organics

Wisconsin DNR, WI-PUB-SW-141, September 1995

Method OA-2: Extractable petroleum in Products, Revision 3 January 10, 1990; University Hygienic Laboratory, Iowa City, Iowa

Method SW846 8000B Revision 2 December 1996

Method SW846 8015B Revision 2 December 1996

Method SW846 3544 Revision 0 December 1996

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16. **TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.**

Table A
Reporting Limits

	Reporting Limit
Diesel/ Mod 8015B/OA-2 Diesel - Aqueous	50 ug/L
Diesel/ Mod 8015B/OA-2 Diesel – Solids	1.7 mg/kg

Table B
EQUIPMENT

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
Gas chromatograph	Hewlett Packard	5890 Series I & II	Various	June 1994
Controller	Hewlett Packard	18594B	Various	June 1994
Injector	Hewlett Packard	G1513A	Various	June 1994
Autosampler tray	Hewlett Packard	18596M	Various	June 1994
Computer	Various Make	486 or better speed	Various	Various
Mach Door Unit	HP/Agilent	NA	NA	March 2008

Table C
SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #
2 mL autosampler vials	VWR	VWR	66020-953
Aluminum crimp seals	VWR	VWR	66010-847
Rubber pipette bulb	NJ Rubber	VWR	R5002-2
N-Dex Nitrile Glove Powder free (medium)	Best	Fisher Scientific	6005 PFM
N-Dex Nitrile Glove Powder free (large)	Best	Fisher Scientific	7005L
N-Dex Nitrile Glove (extra large)	Best	Fisher Scientific	7005XL
5.75” disposable pipettes	Fisher Scientific	Fisher Scientific	13-678-20-A
Disposable culture tubes	VWR	VWR	60825-618
25 mL adjustable Repipettor	VWR	VWR	40000-066
Lab notebooks	VWR	VWR	51280-108
600 mL beakers	Kimble	Fisher Scientific	13910-145
250 mL beakers	Kimble	Fisher Scientific	14005-250
150 mL beakers	Kimble	Fisher Scientific	02539J
60 degree powder funnel	Pyrex	VWR	30240-047
2.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	20-814-B
5.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	29623-300
25.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-A
50.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-B
9 oz Amber jars	QEC	QEC	2124-0009
33 ml stainless steel cells	Dionex	Dionex	049562
Cellulose filters	Dionex	Dionex	049458
60 mL collection vials	I-Chem	VWR	405531
Stainless Steel Spatula	VWR	VWR	
25 uL gastight syringe	Hamilton	Fisher Scientific	24559
50 uL gastight syringe	Hamilton	Fisher Scientific	111111
100 uL gastight syringe	Hamilton	Fisher Scientific	13-684-100
250 uL gastight syringe	Hamilton	Fisher Scientific	81100
500 uL gastight syringe	Hamilton	Fisher Scientific	81217
1000 uL gastight syringe	Hamilton	Fisher Scientific	1482425
Pyrex baking dish	Corning	-----	6001078

Table D
REAGENTS

Reagent	Purity	Manufacturer	Vendor	Catalog #
Methylene Chloride	Pesticide Quality	Burdick & Jackson	Fisher Scientific	CS300-200
Di-ionized Water	Type I ASTM	----	----	----
Nitrogen	99.99%	Whatman	----	----

Table E
STANDARDS

Standard - Fuel Oil	Acronym	Concentration
No. 2 Fuel Oil (Supelco)	TPHDRO CAL STD	20,000ug/ml In Methanol
No. 2 Fuel Oil (Restek)	TPHDRO 2nd Reference STD	50,000ug/ml In Methylene Chloride
TPHDRO CHK	500ppm	50ul of 50,000ppm #2 Fuel & 5ul of 10,000ppm o-terphenyl diluted to 1.0mls IN Methylene Chloride
Calibration STDS TPHDRO Pt #6	2000ppm	625ul of 20,000ppm #2 Fuel stock STD to 5.0mls in methylene chloride
Pt#1 and PRLS	50ppm	20ul of 2000ppm Pt#6 diluted to 1.0ml in Methylene Chloride
Pt#2	100ppm	40ul of 2000ppm Pt#6 diluted to 1.0ml in Methylene Chloride
Pt#3	250ppm	100ul of 2000ppm Pt#6 diluted to 1.0ml in Methylene Chloride
Pt#4	500ppm	200ul of 2000ppm Pt#6 diluted to 1.0ml in Methylene Chloride
Pt#5	1000ppm	400ul of 2000ppm Pt#6 diluted to 1.0ml in Methylene Chloride

Standard - Surrogates	Concentration	Amount Stock Used
O-Terphenyl STD (Restek)	O- Terphenyl STND	10,000ug/ml In Methylene Chloride

Table F
SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
WATER	3510C	1L Amber jar with Teflon lined lid	HCl	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
SOIL	3545	Amber jar with Teflon lined lid	none	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius

Table G
Quality Control

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 8015**
Initial Calibration	Minimum of five levels; lowest level near but above MDL.
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Calibration Verification Standard (CVS)	One at the beginning of a 12 hour time clock or more frequent.
Laboratory Control Spike	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Method Validation	Annually
MDL	Annually
Surrogate Standards	Added to every sample.
Reporting Limit Verification Standard (RLVS)	After every calibration and monthly thereafter.

Table H
CALIBRATION AND STANDARDIZATION

Analytical Method Acceptance Criteria⇨ Calibration Measure ⇩	**Frequency**	**Acceptance Criteria**
Initial Calibration	• Installation of new column • Creation of new analytical method • CVS fails criteria	• Use Linear Regression and Correlation Coefficient is > 0.9900.
Continuing Calibration Verification	• At the beginning of every 12 hour shift or more frequent	• 85-115% Recovery.
Laboratory Control Spike and Duplicate	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	• In house limits determined.
Matrix Spike and Duplicate	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	• In house limits determined.
MDL's	• Annually	• 1-10 times the MDL should be equal to the spike concentration that was used to determine the MDL's
Method Validation	• Annually	• 70-130%
Surrogate Standards	• Added to every sample.	• In house limits determined.
Reporting Limit Verification Standard (RLVS)	• After every initial calibration and monthly thereafter	• 60-140%

17. REVISIONS

Document Number	Reason for Change	Date
G3-SVO-03 Rev. 6	New Green Bay Laboratory document changed into Pace SOP format and updated.	25May2005
GB-O-023-Rev.1	Sec 6.1 changed definitions reference to Section 10 of the Quality Manual. Sec 8.1 changed preservation requirement from unpreserved to preserved to be consistent with Pace Network. Sec 12.4.2 added requirement to pH adjust QC samples. Sec 14.1.2 updated SOP number. Sec 15.2 updated SOP number and title.	01Feb2006
GB-O-023-Rev.2	Sec 10.5.3 Changed Standard expiration date to 1 year from 6 months. Sec 10.6.3 Changed Standard expiration date to 1 year from 6 months. Sec 12.4.2 Remove requirement to pH adjust samples if not received preserved. Sec 12.4.4 Added requirement to allow sample extract to separate for 10 minutes. Sec 12.4.7 changed from TurboVap to Water Bath for concentration procedure. Sec 13.2.7 Added Surrogate Recovery Info Added water bath to Table B Added KD flask and Separatory funnel to Table C	27Jun2006
S-GB-O-023-Rev.3	Updated Section 12.5 for Microwave digestion Updated Section 16 - References.	21Sept2007
S-GB-O-023-REV.04	Updated signature page. Section 2.2 – Changed PQL from 10 and 100 to 5 and 50 respectively. Deleted Section 7. Renumbered document. Section 10.1.1 – Updated Standard concentrations. Section 10.2 – Added ICV requirements. Section 10.3 – Eliminated 250 mg/L CCV. Section 11.3 – Clarified retention time window setting. Section 11.9 – Changed final volume from 2 mL to 1 mL. Section 12.1.2 – Updated Sol Spike Concentration for IDOC. Section 12.2.3 – Updated CCV concentration. Section 13 – Updated SOP references. Sections 10.3, 12.1.11, Table E, Table G, and Table H – added PRLS. Table A – Updated PRLs	01Oct2008
S-GB-O-023-Rev.05	Deleted all fuel references except Diesel fuel #2 and updated Table E Deleted extraction procedures and updatedTable B,C & D Section 11.2.1 – Update to include Mach conditions	17Mar2010

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STANDARD OPERATING PROCEDURE

Analysis of Organochlorine Pesticides by Gas Chromatography

Reference Methods: SW-846 8081A

SOP NUMBER	S-GB-O-027-Rev.05
EFFECTIVE DATE	Date of Final Signature
SUPERSEDES	S-GB-O-027-Rev.04

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 07/13/11

Kate Grams, Laboratory Quality Manager — Date: 7/13/11

Chris Haase, Department Manager — Date: 7/13/11

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the analysis of various organochlorine pesticides in water, soils, sediments, biological tissue, and solid waste compliant with SW-846 Method 8081A.

2. SCOPE AND APPLICATION

2.1 This method is used to determine the concentration of the various organochlorine pesticides in water, soil, sediment, biological tissue, and solid waste extracts using a gas chromatograph equipped with dual electron capture detectors listed in Appendix A: Analyte List and Reporting Limits.

2.2 The policies and procedures contained in this SOP are applicable to all personnel involved in the analysis of organochlorine pesticides by gas chromatography.

2.3 Sample extracts analyzed by this SOP were prepared by latest revisions of SOPs S-GB-O-053, *Separatory Funnel Extraction by SW846 3510C*; S-GB-O-054, *Ultrasonic Extraction by SW846 3550B*; S-GB-O-031, *Extraction of Biological Samples for Organochlorine Pesticides/PCBs*; and S-GB-O-043, *Extraction of Toxaphene Using Automated Soxhlet*.

3. SUMMARY OF METHOD

3.1 A volume of sample extract in hexane is injected into a gas chromatograph (GC) and the compounds in the GC effluent are detected by an electron capture detector (ECD) and then analyzed.

4. INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware. These interferences lead to discrete artifacts or elevated baselines in gas chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences by running reagent blanks and method blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates, which are easily extracted during laboratory operations. Avoiding the use of such plastics in the laboratory can best minimize interferences from phthalates.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled. Cleanup procedures such as Gel Permeation Chromatography (GPC), Florisil cartridge cleanup, Silica Gel Separation, and sulfur removal are available for the most common interference's encountered. These cleanup procedures are described in separate SOPs.

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. Standard solutions should be prepared in a hood.

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.

5.3 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training.

5.4 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent.

5.5 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management, consult the current version of S-GB-S-006, *Waste Management and Handling*.

5.6 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution.

6. DEFINITIONS

6.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Extracts should be stored at 4°±2ºC in the dark in Teflon-sealed containers until analysis is complete. The sample extracts must be analyzed within 40 days from extraction.

8. EQUIPMENT AND SUPPLIES

8.1 Gas Chromatograph: HP5890 or HP6890 GC with dual electron capture detectors (or equivalent)

Recommended Gas Chromatograph Operating Conditions

The following are the gas chromatographic analytical conditions. The conditions are recommended unless otherwise noted.

Carrier Gas:	Helium 2.0 mL/min flow rate
Make-up Gas:	Nitrogen 65 mL/min flow rate
Detector Temperature:	300°C
Injector Temperature:	200°C
Injection:	Splitless
Injection Volume:	1 µL
Initial Temperature:	150°C
Initial Hold Time:	0.0 min
Temperature Ramp:	12°C/min to 210°C, hold 1.0 min., 3°C/min to 225°C , hold 1.0 min, 12°C/min to 270°C, hold 8.0 min
Final Temperature:	270°C
Final Hold Time:	8 min

8.2 GC Autosampler: HP7673A or HP7683

8.3 Detector: ECD

8.4 Gas Chromatograph Columns: RTX-CLP, 30 m x 0.32 mm ID, .25µm film thickness (Restek or equivalent) and a RTX-CLP2, 30 m x 0.32 mm ID, .25 µm film thickness, (Restek, or equivalent). Other analytical columns may be used based on projects specific requirements. Columns are mounted in a dual GC/ECD with a single injection port/guard column connected to a glass Y-splitter.

8.5 Data Processor: ChemStation/HP Target

8.6 Syringes: 10-1000 µL Gastight syringes

8.7 Autosampler Vials: 2 ml glass vials with crimp top caps.

8.8 Helium gas

8.9 Nitrogen gas

9. REAGENTS AND STANDARDS

9.1 Hexane, Acetone, and Isooctane, pesticide grade

9.2 Stock Standard Solutions: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf life of standard solutions is 1 year from the date of preparation or the expiration date on the vendor label; whichever is earliest. (See Table 1 for standard preparation.)

9.3 Calibration Standards: Single component standards are prepared from stock standard solutions at 5 concentration levels. One of the concentration levels should be at a concentration near, but above the method detection limit. Separate calibration standards are required for each multi-component target analyte (e.g. Toxaphene and Technical Chlordane). Shelf life of the calibration standards is 6 months from the date of preparation or the expiration date of the stock standard solution; whichever is earliest. (See Table 2 for standard preparation.)

9.4 Surrogate Standards: Surrogate standards are used to monitor the performance of the method. They are added to all calibration standards. Shelf life of the surrogate standards is 6 months from the date of preparation or the expiration date of the stock standard solution; whichever is earliest. (See Tables 1 and 2 for standard preparation.)

9.5 Performance Evaluation Mixture (PEM): A standard comprised of DDT and Endrin, which is analyzed at the beginning of every 12 hours to determine compound breakdown. (See Table 3 for standard preparation.)

Table 1

Stock Standards

Standard	Stock Standard	Conc.	Amount Used	Final Volume	Solvent Used	Final Conc.	Expiration Date
TMX/DCB Stock	Restek Pesticide Surrogate Mix	200 µg/mL	0.500 mL	10 mL	Hexane	10 µg/mL	1 year from date of preparation
Pesticide A/B Stock	Restek Organochlorine A/B Mix	8-80 µg/mL	1.0	10 ml	-	0.8-8.0µg/mL	1 year from date of preparation
Pesticide C-Mix Stock	Absolute Stds	100µg/mL	1.0	50 ml	-	2.0µg/mL	1 year from date of preparation
Tech. Chlordane Stock Solution	Restek Chlordane Mix	1000µg/mL	1.0	10 ml	-	100 µg/mL	1 year from date of preparation
Toxaphene Stock	Restek Toxaphene Mix	1000µg/mL	1.0 mL	10 mL	Hexane	100 µg/mL	1 year from date of preparation
Pesticide A/B ICV Stock	Supelco Mix A/Mix B	5-50µg/mL	1.0 mL	12.5 mL	Hexane	0.4-4.0 µg/mL	1 year from date of preparation
Absolute Stds Different Lot than Calibration Std	Absolute Stds	100 µg/mL	1.0 ml	50 mL	Hexane	2.0 µg/mL	1 year from date of preparation
Different Lot # than Calibration Std	Restek Toxaphene Mix	1000µg/mL	1.0 mL	10 mL	Hexane	100 µg/mL	1 year from date of preparation

Supelco Pesticide Mix A contains alpha-BHC, gamma-BHC, Heptachlor, Endosulfan I, Dieldrin, Endrin, 4,4'-DDD, 4,4'-DDT, and Methoxychlor.

Supelco Pesticide Mix B contains beta-BHC, delta-BHC, Aldrin, Heptachlor epoxide, alpha-chlordane, gamma-chlordane, 4,4'DDE, Endosulfan II, Endrin aldehyde, Endosulfan sulfate, and Endrin ketone.

Absolute Stds Pesticide Mix C contains 2,4-DDT, 2,4-DDD, 2,4-DDE, Hexachlorobenzene, Pentachloroanisole, Oxychlordane, Trans-nonachlor, Cis-nonachlor, Mirex and Isodrin. Isodrin is not a reported compound.

Table 2

Calibration Standards

Standard	Pesticide Stock Standard	Amount Used (µL)	Final Conc. (µg/mL)	Surrogate Stock Standard	Amount Used (µL)	Final Conc. (µg/mL)	Solvent Used	Final Volume (mL)	Expiration Date
PEST-1 & RLVS	Pesticide A/B Stock	200	0.0050-0.050	TMX/DCB Stock	32	0.010	Hexane	32	6 months from date of preparation
PEST-2	Pesticide A/B Stock	250	0.010-0.10	TMX/DCB Stock	40	0.020	Hexane	20	6 months from date of preparation
PEST-3	Pesticide A/B Stock	500	0.020-0.20	TMX/DCB Stock	100	0.050	Hexane	20	6 months from date of preparation
PEST-4	Pesticide A/B Stock	1000	0.040-0.40	TMX/DCB Stock	200	0.10	Hexane	20	6 months from date of preparation
PEST-5	Pesticide A/B Stock	1000	0.080-0.80	TMX/DCB Stock	120	0.12	Hexane	10	6 months from date of preparation
PEST-4 ICV	Pesticide A/B ICV Stock	1000	0.040-0.40	TMX/DCB Stock	100	0.1	Hexane	10	6 months from date of preparation
INDC-1 & RLVS	Pesticide C-Mix Stock	50	0.005	TMX/DCB Stock	20	0.010	Hexane	20	6 months from date of preparation
INDC-2	Pesticide C-Mix Stock	100	0.010	TMX/DCB Stock	40	0.020	Hexane	20	6 months from date of preparation
INDC-3	Pesticide C-Mix Stock	200	0.020	TMX/DCB Stock	100	0.050	Hexane	20	6 months from date of preparation
INDC-4	Pesticide C-Mix Stock	800	0.004	TMX/DCB Stock	400	0.10	Hexane	40	6 months from date of preparation
INDC-5	Pesticide C-Mix Stock	800	0.080	TMX/DCB Stock	240	0.12	Hexane	20	6 months from date of preparation
INDC-4 ICV	Pesticide C-Mix ICV Stock	800	0.040	TMX/DCB Stock	400	0.1	Hexane	40	6 months from date of preparation
TOX-1 & RLVS	Toxaphene Stock	75	0.3	TMX/DCB Stock	125	0.005	Hexane	25	6 months from date of preparation
TOX-2	Toxaphene Stock	125	0.5	TMX/DCB Stock	25	0.01	Hexane	25	6 months from date of preparation
TOX-3	Toxaphene Stock	375	1.5	TMX/DCB Stock	50	0.02	Hexane	25	6 months from date of preparation
*TOX-4	Toxaphene Stock	1500	3.0	TMX/DCB Stock	250	0.05	Hexane	50	6 months from date of preparation
TOX-5	Toxaphene Stock	1000	4.0	TMX/DCB Stock	250	0.10	Hexane	25	6 months from date of preparation
TOX-6	Toxaphene Stock	1250	5.0	TMX/DCB Stock	375	0.15	Hexane	25	6 months from date of preparation
TOX-4 ICV	Toxaphene ICV Stock	750	3.0	TMX/DCB Stock	125	0.050	Hexane	25	6 months from date of preparation
TCHLOR-1 & RLVS	Technical Chlordane Stock	10	0.10	TMX/DCB Stock	10	0.010	Hexane	10	6 months from date of preparation
TCHLOR-2	Technical Chlordane Stock	20	0.20	TMX/DCB Stock	20	0.020	Hexane	10	6 months from date of preparation
TCHLOR-3	Technical Chlordane Stock	40	0.40	TMX/DCB Stock	50	0.050	Hexane	10	6 months from date of preparation
*TCHLOR-4	Technical Chlordane Stock	80	0.80	TMX/DCB Stock	100	0.10	Hexane	10	6 months from date of preparation
TCHLOR-5	Technical Chlordane Stock	100	1.0	TMX/DCB Stock	120	0.12	Hexane	10	6 months from date of preparation

* Level used in 1 point calibration of multicomponent.

Table 3
PEM Standards

Standard	Stock Standard	Conc.	Amount Used	Final Volume	Solvent Used	Final Conc.
PEM Stock Solution	4,4'-DDT & Endrin	1000ug/ml	1.0 ml each	100 ml	Hexane	10µg/mL
PEM Working Standard	PEM Stock Solution	10µg/mL	0.6 mL	40 mL	Hexane	0.15µg/mL

10. CALIBRATION

10.1 Prime (or deactivate) the column by injecting a pesticide standard mixture at the high point in the calibration curve. Inject this standard mixture prior to beginning the initial calibration or calibration verification at the beginning of the analytical sequence.

10.2 A solvent blank may be run following the prime to verify there is no carryover or contamination introduced from the injection process. Solvent blanks may be placed into the analytical sequence at any position by the analyst to demonstrate that the analytical system is not contaminated. Solvent blanks, however, may not be routinely placed immediately prior to continuing calibration verification standards, PEM's, etc.

10.3 A Performance Evaluation Mixture (PEM), a standard comprised of DDT and Endrin, will be analyzed before samples are analyzed and at the beginning of every 12 hours. If the breakdown for either compound exceeds 15%, corrective action must be taken prior to calibration. Breakdown is calculated by the following equations:

$$\% \text{ Breakdown DDT} = \frac{\text{Response (DDD + DDE)}}{\text{Response (DDD + DDE + DDT)}} * 100$$

$$\% \text{ Breakdown Endrin} = \frac{\text{Response (Endrin aldehyde + Endrin Ketone)}}{\text{Response (Endrin + Endrin aldehyde + Endrin Ketone)}} * 100$$

10.3.1 The PEM standard is not analyzed when Toxaphene is the only analyte being quantified and reported from the analysis.

10.4 Initial Calibration: The initial calibration includes the analysis of five concentrations of each single component pesticide and surrogate compound and a single point calibration for each multi-component analyte (unless otherwise necessary for a specific project). See SW-846 Method 8000B for additional guidelines on proper initial calibration and calibration verification.

10.4.1 The initial calibration sequence contains the following injections that must be analyzed before any samples:

1. PEM
2. Toxaphene – LVL 4
3. T-Chlordane – LVL 4
2. Pesticide A/B Mix #5
3. Pesticide A/B Mix #4
4. Pesticide A/B Mix #3
5. Pesticide A/B Mix #2
6. Pesticide A/B Mix #1
7. Pesticide C Mix #1
8. Pesticide C Mix #2
9. Pesticide C Mix #3
10. Pesticide C Mix #4
11. Pesticide C Mix #5
12. Hexane
13. PEM
11. Initial Calibration Verification- A/B Mix
12. Initial Calibration Verification- C Mix

(This sequence can change based upon the analytes of interest and project specific requirements.)

10.5 Initial Calibration Acceptance Criteria

10.5.1 Linear Calibration using Average Response Factors: Calculate the response factor (RF) for each analyte at each concentration, the mean response factor, and the relative standard deviation (RSD) of the response factors using the formulas below.

$$\text{Response Factor (RF)} = \frac{\text{Peak Area}}{\text{Standard concentration (µg/mL)}}$$

$$\%\text{RSD} = \frac{\text{Standard Deviation}}{\text{Average Response Factor}}$$

10.5.2 Any target analyte which has calibration factors with a relative standard deviation (RSD) of less than 20% is considered valid and the average response factor may be used for quantitation purposes. If the %RSD exceeds 20%, the analyst must use linear regression for determining analyte concentrations.

10.5.3 If linear regression is used the intercept should not be forced through the origin. The regression calculation will generate a correlation coefficient "r". In order to be used for quantitative purposes the r-value must be greater than 0.99.

10.5.4 All initial calibration and calibration verification criteria apply to both analytical columns.

10.5.5 The initial calibration may continue to be used as long as the analytical system meets acceptance criteria. If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. Perform instrument maintenance as necessary and recalibrate the instrument.

10.6 Initial Calibration Verification

10.6.1 To ensure the calibration standards are at the correct concentration, an initial calibration verification (ICV) will be analyzed against the initial calibration curve prior to the analysis of samples. The ICV is a midpoint of the Pesticide A/B Mix, (and Pesticide C Mix if included in the initial calibration) and must be a different lot number from the same vendor. The difference between the average of all compounds responses of the ICV and initial calibration standards must be within 15% of one another.

10.6.2 The injection of the first ICV begins the 12-hour clock in which samples may be analyzed.

10.7 Continuing Calibration Verification

10.7.1 Continuing Calibration Verification (CCV) will include the injection of the PEM standard, midpoint calibration checks of the Pesticide A/B Mix, and Pesticide C Mix if included in the initial calibration.

10.7.2 A PEM and CCV must be analyzed every 12 hours or 20 samples, whichever is more frequent. It is recommended that calibration verification be performed after every 10 samples to minimize reruns due to calibration verification failure.

10.7.3 Sample data is not acceptable unless bracketed by acceptable analyses of a CCV. Any samples not bracketed by an acceptable CCV must be reanalyzed. The ending PEM of a sequence may fail breakdown and samples can be reported.

10.8 Initial Calibration Verification and Continuing Calibration Verification Acceptance Criteria

10.8.1 The percent difference (%D) is determined for all analytes in the ICV/CCV. The average %D must be within $\pm$ 15% of the calibration curve for the average of all analytes.

$$\%D = \frac{R_2 - R_1}{R_2} * 100$$

Where: R_1 = Theoretical Value
R_2 = Calculated Amount

The analyst should verify that the software is using appropriate values for calculations.

10.8.2 If the acceptance criteria for the ICV/CCV are not met inspect the gas chromatographic system for problems. Reinject the standard; if acceptance criteria are not met then a new initial calibration must be performed.

10.8.3 Any analyte, for any reason, not meeting the ICV/CCV acceptance criteria must be qualified, if reported to the client.

10.9 Retention Time Windows

10.9.1 Retention time windows are determined for all single and multi-components analytes.

10.9.1.1 Make at least four injections of all analytes of interest over a 72-hour period.

10.9.1.2 Record the retention time (RT) for each analyte, and selected peaks for multi-component analytes, to three decimal places. Calculate the mean and standard deviation for each peak.

10.9.1.3 The width of the retention time window is defined as $\pm$ 3 standard deviations of the mean established. The minimum retention window will be $\pm$ 0.030 minutes.

10.9.1.4 Establish the center of the RT window for each analyte and surrogate using the absolute RT from the calibration verification standard at the beginning of the analytical shift. Optionally, the initial calibration retention time windows may continue to be used as long as the method criteria are met. For samples run during the same shift as an initial calibration, use the RT of the mid-point standard in the initial calibration.

10.9.1.5 RT windows must be calculated on each column and instrument when a new GC column is installed. Additional guidance is provided in SW846 8000B.

10.9.2 The retention time of each analyte must fall within its respective retention time window for all standards. If not, the gas chromatographic system must be evaluated. Reinject the standard; if not acceptable then a new calibration must be performed.

10.10 Reporting Limit Verification Standard (RVLS) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

11. PROCEDURE

11.1 Sample Analysis

11.1.1 A matrix spike/matrix spike duplicate must be prepared and analyzed at least once for each matrix type per every twenty samples. The sample use for the MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. If insufficient sample volume is available to perform a MS/MSD, a laboratory control spike and laboratory control spike duplicate will be used instead.

11.1.2 Analysis of a sample on both GC columns is required for all samples, blanks, matrix spikes, matrix spike duplicates and laboratory control samples.

11.1.3 The laboratory will identify and quantify peaks based on RT and the average calibration factor established during the initial calibration sequence.

11.1.4 ThePEM and continuing calibration are analyzed every 20 samples (not to exceed 12 hours) or less during an analytical sequence in order to monitor retention times, calibration factors, and column performance. (It is recommended that calibration verification be performed after every 10 samples to minimize reruns due to calibration verification failure.) Sample data can be collected only as long as the results for this standard fall within the defined limits. If two consecutive unacceptable standards are run, all extracts run since the previous acceptable standards must be reanalyzed. The ending PEM of the sequence is allowed to fail.

11.1.5 Failure to meet any of the criteria established in this method must be thoroughly documented, and technical justification for the validity of the data must be presented to the section supervisor.

11.2 Quantitation of Analytes

11.2.1 Analytes must be quantified with a laboratory data system. Peak area is the basis for quantitation.

11.2.2 Quantitation of *single response pesticides* and surrogate standards is performed using the column designated as the primary column. This column will generally be the RTX-CLP, however, the analyst may specify either column as the primary column. The primary column must be used unless there is a documented reason (i.e. interference) for using the confirmation column for quantitation.

11.2.2.1 The identification and quantification of peaks is based on RT and the average calibration factor established during the initial calibration sequence.

11.2.2.2 A tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window. Each tentative identification must be confirmed by a second GC column of dissimilar stationary phase.

11.2.3 Quantitation of *multi-response analytes* can be done this way several ways.

The analyst may choose 4 to 6 of the largest peaks for quantitation. The peaks chosen for quantitation should have minimal co-elution with other peaks in the chromatogram. Use each peak chosen in the standard to calculate a calibration factor for that peak. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 4 to 6 resulting concentrations are averaged to provide the final result for the multi-response analyte in the sample.

11.3 Calculations

Aqueous samples:

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(V_t)(D)}{(Cf)\,(V_i)}$$

Soil, Solid, Biota samples:

$$\text{Concentration } (\mu g/Kg) \text{ (Dry weight basis)} = \frac{(A_x)(V_t)(D)}{(Cf)(W)(S)}$$

Where:
A_x = Area or peak height for an analyte
V_t = Final volume of extract in mL
D = Dilution factor

Cf = Average calibration factor
W = Initial sample weight (Kg)
Vi = Initial sample volume (L)
S = %Solids/100

Biota samples are typically reported on an "as is" or wet weight basis. The dry weight correction portion of the formula is typically not utilized.

11.4 Data Evaluation

11.4.1 For samples where a second analysis is performed at a dilution, the results that are reported from the second analysis are qualified with a "D".

11.4.2 The analyst should compare the analyte concentrations between the two columns used for analysis. The RPD between the two columns should be < 40. If the RPD between the two results is greater than 40 but less than or equal to 100 the analyst should evaluate the chromatogram for interfering peaks that are co-eluting with the analyte. If an interference is determined to be present, report the lower of the two values and qualify the result with a "P" data qualifier. If no interference is determined to be present, report the higher of the two values and qualify the result with a "P" data qualifier.

11.4.3 Additional data qualifiers may be used dependant upon project specific requirement.

12. QUALITY CONTROL

12.1 Method Blanks

12.1.1 Method blanks are an analyte-free matrix spiked with surrogate solution, extracted, and analyzed by the same procedure that is used with the associated samples.

12.1.2 In order to be acceptable, a method blank analysis cannot contain any of the analytes listed in this method above the reporting limit. All samples associated with a contaminated method blank must be re-extracted and reanalyzed unless the analyte concentration in the sample is greater than 20 times the amount found in the method blank, the analyte is not detected in the associated sample, or other approval is given directly by the supervisor.

12.1.3 The surrogate retention times must be within the retention time windows for both tetrachloro-m-xylene and decachlorbiphenyl. The surrogate recovery limits are established annually and distributed in the analytical laboratory. When surrogate recoveries in method blank do not meet the recovery limits, the method blank is reanalyzed and/or the samples re-extracted.

12.2 Matrix Spike/Matrix Spike Duplicate

12.2.1 The analyte and surrogate retention times must be within the windows specified during the initial calibration or current continuing calibration.

12.2.2 The percent recovery and the relative percent difference between the recoveries of each of the compounds in the matrix spike samples will be calculated and reported by using the following equations:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = Spike sample result
SR = Sample result
SA = Spike added

$$RPD = \frac{|MSR - MSDR|}{1/2\,(MSR + MSDR)} \times 100$$

Where: RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

12.2.3 The limits for matrix spike/matrix spike duplicate compound recoveries and RPD limits are established and distributed in the analytical area. Failure to meet these limits requires corrective action by the laboratory in accordance with S-ALL-Q-012 *Corrective Action / Preventative Action Process*.

12.3 Laboratory Control Spike

12.3.1 The percent recoveries and the relative percent difference between the recoveries of each of the compounds in the LCS/LCSD will be calculated and reported using the following equation:

$$\text{LCS/LCSD Recovery} = \frac{LCSR}{SA} \times 100$$

Where: LCSR = Lab control spike/duplicate recovery
SA = Spike added

$$RPD = \frac{|LCSR - LCSDR|}{1/2\,(LCSR + LCSDR)} \times 100$$

Where: RPD = Relative percent difference
LCSR = Laboratory control spike recovery
LCSDR = Laboratory control spike duplicate recovery

12.3.2 The limits for LCS/LCSD compound recoveries and RPD limits are established and distributed in the analytical area. Due to number of analytes fortified into the LCS, a small percentage of sporadic marginal failures may be tolerated (i.e. will not trigger re-extraction and analysis of the entire batch.) If more than 10% of the analytes are outside of the control limits, the samples are re-extracted or re-analyzed.

12.4 Surrogate recoveries

12.4.1 Surrogate recoveries must be evaluated using laboratory established control limits. If both surrogate recoveries fail these criteria, re-extract the sample. One surrogate is allowed to be outside of the control limits.

12.4.2 Surrogates are not evaluated in samples where the surrogates are diluted below the low level standard concentration of the surrogates.

12.4.3 Surrogate recoveries will be calculated and reported using the following calculation:

$$\textbf{S}\text{urrogate Percent Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where: Q_d = Quantity determined by analysis
Q_a = Quantity added

13. METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures,* most current revision or replacement. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of the LOD and LOQ*, most current revision or replacement, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, most current revision or replacement to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Management and Handling*, most current revision or replacement.

14.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention..

15. REFERENCES

15.1 USEPA, SW-846, Method 8000B, Revision 2, December 1996.

15.2 USEPA, SW-846, Method 8081A, Revision 1, December 1996.

15.3 S-GB-O-053, Separatory Funnel Extraction by SW846 3510C

15.4 S-GB-O-054, Ultrasonic Extraction by SW846 3550B

15.5 S-GB-O-031, Extraction of Biological Samples for Organochlorine Pesticides/PCBs

15.6 S-GB-O-043, Extraction of Toxaphene Using Automated Soxhlet

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA, ETC.

Appendix A
Analyte List and Reporting Limits

	Water	Soil	Biota
Compound	[1]PRL (µg/L)	[1]PRL (µg/Kg)	[1]PRL (µg/Kg)
2,4'-DDD (o,p-DDD)	0.05	1.7	2.5
2,4'-DDE (o,p-DDE)	0.05	1.7	2.5
2,4'-DDT (o,p-DDT)	0.05	1.7	2.5
4,4'-DDD	0.10	3.3	5.0
4,4'-DDE	0.10	3.3	5.0
4,4'-DDT	0.10	3.3	5.0
Aldrin	0.050	1.7	2.5
alpha-BHC	0.050	1.7	2.5
alpha-Chlordane	0.050	1.7	2.5
beta-BHC	0.050	1.7	2.5
cis-Nonachlor	0.05	1.7	2.5
Delta-BHC	0.050	1.7	2.5
Dieldrin	0.10	3.3	5.0
Endosulfan I	0.050	1.7	2.5
Endosulfan II	0.10	3.3	5.0
Endosulfan Sulfate	0.10	3.3	5.0
Endrin	0.10	3.3	5.0
Endrin Aldehyde	0.10	3.3	5.0
Endrin Ketone	0.10	3.3	5.0
gamma-BHC	0.050	1.7	2.5
gamma-Chlordane	0.050	1.7	2.5
Heptachlor	0.050	1.7	2.5
Heptachlor Epoxide	0.050	1.7	2.5
Hexachlorobenzene	0.050	1.7	2.5
Methoxychlor	0.50	17	25
Mirex	0.05	1.7	2.5
Oxychlordane	0.05	1.7	2.5
Pentachloroanisole	0.050	1.7	2.5
trans-Nonachlor	0.05	1.7	2.5
Toxaphene	3.0	100	150
Technical chlordane	1.0	33	50

ND = Not determined

PRL = Pace Reporting Limit

1 = Reporting limit is based on lowest calibration standard.

17. REVISIONS

SOP Number	Revision	Date
GB-O-027-Rev.0	First Issue	May 23, 2006
S-GB-O-027-Rev.1	Section 2.3 – added SOPs references Section 5 – updated with current information Section 6 – Updated with current information Section 7.2 – changed Assistant General Manager to General Manager Section 13.3.2 – added South Carolina requirement as to acceptability of sporadic failures as applied to the LCS Section 16 – updated references	August 23, 2007
S-GB-O-027-Rev.2	SOP Review Section 13.3.2 & 13.4.1 – added South Carolina control limit requirements. Updated Signature Page Updated Section 14	January 23, 2008
S-GB-O-027-Rev.03	Updated Signature Page. Updated Scope to refer to Appendix A. Section 2.3 – updated SOP references. Section 7 – Deleted. Renumbered document accordingly. Section 9 – Table 2 – Added Report Limit Verification Standard (RLVS). Section 10.10 – Added RLVS Definition and criteria Section 12.2.3 – Updated SOP reference. Re-numbered document-	January 19, 2009
S-GB-O-027-Rev.03	Updated Signature Page. Section 9.3 & 9.4 – Update expiration dates. Section 9 – Table 1 – Updated with current information. Section 9 – Table 2 – Updated with current information. Section 9 – Table 3 – Updated with current information. Updated Section 10. Section 11.1.4 – Added PEM information. Section 11.4.3 – Deleted. Section 16 – Appendix A updated.	April 30, 2009
S-GB-O-027-Rev.05	Throughout Document: Updated Waste Handling and Management SOP to S-GB-S-006 Throughout Document: Removed all references to State of South Carolina criteria. Section 10.6.1 and 10.8.1: Added language of the acceptance criteria being the average of all compounds reported must be <15%. Section 11.2.3.2: Deleted Section 11.3 Calculations: Removed GPC From Calculation, it is a 1:1 dilution.	13Jul2011

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STANDARD OPERATING PROCEDURE

Extraction of PCBs Using the Automated Soxhlet

Reference Methods: SW-846 Method 3541

SOP NUMBER:	S-GB-O-041-REV.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-041-REV.02

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 4/20/10

Kate Grams, Laboratory Quality Manager — Date: 4/19/10

Glen Coder, Department Manager — Date: 4/19/10

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the extraction of soil and sediment samples compliant with SW-846 Method 3541 prior to PCB analysis.

2. SCOPE AND APPLICATION

2.1 This procedure is applicable to extraction and concentration of PCBs from soil or sediment samples. Extracts may be prepared by this method for analysis by SW-846 Method 8082/8082A as per the latest revision of Pace Analytical Services, Inc.'s SOP S-GB-O-026, *Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography* and S-GB-O-047, *Analysis of Polychlorinated Biphenyls (PCB's) by Gas Chromatography by 8082A*.

2.2 The policies and procedures contained in this SOP are applicable to all personnel involved in the preparation of extracts for chromatographic analysis.

3. SUMMARY OF METHODS

3.1 A measured mass of sample, typically 10 grams, is mixed with sodium sulfate until it is free flowing. The sample mixture is transferred into a cellulose extraction thimble. The thimbles are placed in extraction beakers and solvent is added. The beakers are loaded into the automated soxhlet unit. The samples are subjected to a pre-programmed heat and pressure extraction cycle, which isolates the organic components from the sample mixture by contact with the solvent. The extract is concentrated to a final volume and subjected to necessary cleanups prior to analysis, as needed.

4. INTERFERENCES

4.1 Interferences may be introduced into sample extracts by contaminants in solvents, reagents, glassware, and any other material that comes in contact with the sample or extract during extract preparation. These interferences must be closely monitored by analyzing Method Blank samples and taking corrective action as required.

4.2 Interferences co-extracted from samples will vary considerably depending on the source of the material. Contaminants that may interfere with the analysis may be removed from the extracts using any combination of cleanups including, but not limited to Florisil column cleanup, Mercury cleanup, Copper cleanup, and Sulfuric Acid cleanup. These cleanup procedures are described in separate SOPs.

5. SAFETY

5.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent.

5.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-O-006, *Waste Handling and Management*.

5.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution.

6. DEFINITIONS

6.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Samples should be stored at 4°±2°C and extracted within 14 days of collection.

7.2 Extracts should be stored at 4°±2ºC in the dark in Teflon-sealed containers until analysis is complete. The sample extracts must be analyzed within 40 days from extraction.

8. EQUIPMENT AND SUPPLIES

8.1 Soxtherm extractors with controllers

8.2 Cellulose Extraction Thimbles (Whatman 2800338 or equivalent)

8.3 Wire Thimble holders

8.4 Extraction Beakers 54x130

8.5 TurboVap II

8.6 200 mL TurboVap tubes

8.7 Inert Rack for extraction beakers

8.8 Culture tubes: 15 ml and 9 ml with Teflon-lined screw cap

8.9 Boiling chips: Teflon or pre-rinsed silicone carbide

8.10 Copper chips precleaned

8.11 Syringes: 250-1000µL Gastight syringes (Hamilton 1000 series or equivalent)

8.12 Stainless steel spatulas

8.13 Analytical balance: Capable of weighing 300g $\pm$ 0.01g

8.14 Disposable Pasteur pipettes

9. REAGENTS AND STANDARDS

9.1 Acetone, pesticide grade

9.2 Hexane, pesticide grade

9.3 Sodium Sulfate (Na_2SO_4): Preheated at 400°C for 4 hours in a crucible to remove contaminants. See SOP: S-GB-O-028, *Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes*, most current revision.

9.4 Surrogate spiking solution: See Table 1 for standard preparation.

9.5 Matrix spiking solution: Dependent upon analysis requested. See Table 1 for standard preparation.

9.6 Ottowa Sand: Preheated at 400°C for 4 hours in a crucible to remove contaminants. See SOP: S-GB-O-028, *Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes*, most current revision.

Table 1

Standard	Stock Standard	Conc.	Amount Used	Final Volume	Solvent Used	Final Conc.
Surrogate spiking solution	TMX (Tetrachloro-m-xylene) and DCB (Decachlorobiphenyl)	200µg/mL	10 mL	1000 mL	Acetone	2.0µg/mL
PCB Matrix Spike	One of either Aroclor 1016, 1242, 1248, 1254, or 1260 or any combination*	1000 µg/ml	1000 µL	200 mL	Acetone	5.0µg/mL

Historical data or requirements of specific projects may determine the analytes and concentrations added to the sample spikes.
*South Carolina State Requirement – Both 1016 and 1260 must be spiked in all LCS, MS and MSD samples.

10. CALIBRATION

10.1 Refer to the most current version of S-ALL-Q-013 *Support Equipment* for the proper procedure to calibrate the analytical balance.

11. PROCEDURE

11.1 Extraction

11.1.1 Rinse the soxhlet extraction beakers with 4:1 Hexane/Acetone. Label with sample identification. Add boiling chips or copper chips to the bottom and set aside.

11.1.2 Weigh approximately 10g of sample into a 250 mL beaker. Record the mass of the sample in the extraction log to the nearest tenth of a gram. Repeat the process for all samples and quality control samples. Ottowa sand is used as the matrix for the quality control samples.

11.1.3 Add enough anhydrous sodium sulfate to each beaker while mixing to create a dry free flowing mixture. The sample should appear granular.

11.1.4 Transfer the mixture from the 250 ml beaker to a cellulose extraction thimble. Place the thimble in an automated soxhlet extraction beaker equipped with a wire thimble holder. Transfer all samples and quality control samples using the same process. Label each extraction beaker with the LIMs number or quality control measure identifier. **Retain the 250 ml beaker for solvent addition**.

11.1.5 Spike each thimble with 500 µL of 2.0 µg/mL surrogate spiking solution. Apply directly to dried sample in thimble.

11.1.6 Spike each laboratory control spike (LCS) and matrix spike (MS/MSD) with 1 mL of 5.0 µg/mL PCB Matrix Spike solution. The volume and concentration of the matrix spiking solution may vary depending on the project requirements.

11.1.7 Pour approximately 140 mL of 4:1 hexane/acetone into the retained 250 mL weighing beaker. Swirl gently to remove any sample mixture adhering to the weighing beaker. Pour the solvent into each cellulose thimble slowly.

11.1.8 Push the wire thimble basket down into the extraction beaker, which will immerse the samples in solvent. Load the extraction beakers onto the automated soxhlet unit.

11.1.9 Verify the automated soxhlet extraction settings (program 02) as summarized here.

Extraction temperature	180°C
Boil Time	45 min
Solvent Reduction	2 x 15 mL
Extraction Time	45 min
Cycle Time	1 hour 38 minutes
Solvent	4:1 Hexane/Acetone

11.1.10 Start the extraction process. Rotate the extraction beakers slightly to insure seal of top o-ring. The process will produce approximately 90 mL of extract.

11.1.11 Turn on the power to the TurboVap. Turn on the nitrogen flow to 16 psi. Allow the TurboVap to warm to 55°C.

11.1.12 Transfer the extracts from the extraction beakers to labeled TurboVap tubes. Rinse the extraction beaker with hexane and add the rinse to the TurboVap tubes. Concentrate the extracts to less than 10 mL. Remove from the TurboVap.

11.1.13 Quantitatively transfer the extracts to labeled vials. Adjust the final volume to 10 mL using hexane.

11.2 Extract Cleanups

11.2.1 Extracts are screened and additional cleanups such as Mercury cleanup, Copper cleanup, or Sulfuric Acid cleanup may be performed if determined necessary.

12. QUALITY CONTROL

12.1 One method blank is extracted with each extraction batch of 20 or fewer samples of the same matrix. .

12.2 A laboratory control spike is extracted with each extraction batch of 20 or fewer samples of the same matrix.

12.3 A matrix spike and a matrix spike duplicate must be performed with each extraction batch when appropriate sample volume is present, otherwise a laboratory control spike duplicate will be performed. Matrix spikes are used to indicate matrix effects on the analysis of the analytes of interest. The sample used for the MS/D pair is either determined by the client or selected at random from client samples as sample volume allows.

12.4 Surrogate standards must be added to all samples, laboratory control spikes, matrix spikes, and method blanks prior to extraction. Surrogates are used to monitor the efficiency of the method on each sample and possible matrix related effects.

12.5 All quality control samples (MB, LCS, MS, MSD, and duplicate samples) must undergo the same preparation and cleanup methods as the samples in the batch. The acceptance criteria and corrective actions are described in the determinative method SOPs.

13. METHOD PERFORMANCE

13.1 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 Quality Manual.

13.2 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.3 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.4 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.5 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the quality assurance office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

14.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

15. REFERENCES

15.1 USEPA, SW-846, Method 3541, "Automated Soxhlet Extraction", September 1994.

15.2 Pace analytical, Inc – Green Bay SOP S-GB-O-026, Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography

15.3 Pace Analytical, Inc – Green Bay SOP S-GB-O-047, Analysis o f Polychlorinated Biphenyls (PCB's) by Gas Chromatography by 8082A

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ETC.

Not Applicable

17. REVISIONS

Document Number	Reason for Change	Date
KM-O-017-Rev.0	First Issue	August 23, 2005
KM-O-017-Rev.1	Weigh into beaker and mix with sodium sulfate prior to transferring to thimble. Matrix for QC samples is Ottawa sand	December 29, 2005
GB-O-041-Rev.0	Converted to Green Bay SOP	January 22, 2007
S-GB-O-041-Rev.1	Section 2 and 16 – added Analytical SOP reference Section 7.2 – updated Assistant General Manager to General Manager Section 10 Table 1 – added South Carolina requirement to spike both Aroclor 1016 and 1260 into the LCS, MS, and MSD.	August 23, 2007
S-GB-O-041-REV.02	Updated signature page. Deleted Section 7. Renumbered document. Section 13 – Updated SOP references	October 3, 2008
S-GB-O-041-REV.03	Updated signature page Sections 1 and 15: Added Reference to new SOP	09Apr2010

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STANDARD OPERATING PROCEDURE

Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons, Base/Neutral/Acids and Total Petroleum Hydrocarbons in Solid Matrices

Reference Method(s):SW-846 Method 3546

SOP NUMBER:	S-GB-O-045-REV.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-045-rev.02

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 5/6/10

Kate Grams, Laboratory Quality Manager — Date: 5/5/10

Glen Coder, Department Manager — Date: 5/5/10

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

The Standard Operating Procedure (SOP) is used to prepare solid samples, based on SW846 Method 3546, for analysis of semi-volatile compounds. Extracts prepared by this method are analyzed by Pace SOPs S-GB-O-049 *Determination of Semivolatile Organics by GC/MS,* S-GB-O-050, *Determination of Semi-volatile Organics by GC/MS (Selective Ion Monitoring,* S-GB-O-044, *Determination of Low Level PAHs by GC/MS-SIM in Solid and Biological Matrices* and S-GB-O-023 *Total Petroleum Hydrocarbons*.

2. SCOPE AND APPLICATION

2.1 This procedure is applicable to extraction and concentration of nonvolatile and semivolatile organics from solids. Extracts may be prepared by this method for analysis by several chromatographic methods including, but not limited to, SW-846 8270C and SW-846 8015B.

2.2 This SOP provides procedures for extracting both low and high concentration samples.

2.3 Appropriate cleanup procedures that may be required for some sample extracts are described in separate SOPs.

2.4 Determinative methods used for extracts prepared by this SOP are described in separate SOPs.

2.5 This procedure is restricted to use by, or under the supervision of, analysts experienced in preparation of extracts for chromatographic analysis. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3. SUMMARY OF METHOD

3.1 An aliquot of the solid sample (30.0-g for low concentration samples and 2.0-g for high concentration samples) is extracted using the microwave extraction unit.

3.2 The extract is concentrated (if necessary) and exchanged into the solvent required for the cleanup or determinative method (if necessary).

4. INTERFERENCES

Interferences may be introduced into sample extracts by contaminants in solvents, reagents, glassware, and any other material that comes in contact with the sample or extract during extract preparation. These interferences must be closely monitored by analyzing Method Blank samples and taking corrective action as required.

5. SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training.

5.3 Samples should be considered biological contact hazards. Use appropriate protective equipment (lab coat, gloves and lab glasses) and aseptic techniques when handling these samples. Wash hands with soap and water upon completion of the procedure and clean the work surface with a disinfectant.

6. DEFINITIONS

Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Samples are to be collected in glass jars with Teflon lined lids, demonstrated to be free of contamination at or below the reporting limit.

7.2 Samples are to be chilled to 4 ± 2°C immediately after collection or to a specified temperature range governed by accredit ting authorities to preserve integrity. Samples collected in the State of Wisconsin that are hand delivered to the laboratory immediately after collection may be accepted if there is evidence, such as arrival on ice, that the chilling process has begun.

7.3 Shipments to the laboratory must be timely to avoid preparation holding time issues. Shipment to the laboratory may be accomplished by hand delivery, courier services or common carrier during normal business hours (8:00 am – 5:00 pm central time). Arrangements to accept samples outside normal business hours must be made with personnel prior to shipment.

7.4 Upon receipt and after processing requests for analysis, samples are to be refrigerated at 4 ± 2°C until preparation. After preparation the extracts are held at $<-10^0$ C until analysis.

7.5 See Appendix A Table F for a summary.

7.6 Holding Times

7.6.1 Soil Samples – Samples must be extracted within 14 days of collection.

7.6.2 Extracts – Extracts must be analyzed within 40 days of preparation. The extracts then must be stored for a minimum of 40 days after the date of the sample analysis.

8. EQUIPMENT AND SUPPLIES

See Appendix A Table B and Table C for a summary.

9. REAGENTS AND STANDARDS

9.1 Where the purity of reagents and standards are not specified analytical reagent grade will be used. A grade no less than those specified by the references will not be used.

9.2 All samples, MB, LCS, LCSD, MS, and MSD must be spiked with the appropriate surrogate standard spiking solutions.

9.3 The LCS/LCSD, MS/MSD must be spiked with the appropriate standard spike.

9.4 Analytical Standard Storage Conditions.

Surrogate Standards:

Standard Name	Components	Concentration	Manufacturer	Catalog #	Storage
Restek B/N Surrogate Mix for PAH and BNAs	Nitrobenzene-d5 2-Fluorobiphenyl p-Terphenyl-d14	5000 ug/mL	Restek	31082	Refrigerator 4 ±2°C
Restek Acid Surrogate Mix for BNAs	2-Fluorophenol Phenol-$d_{(6)}$ 2,4,6-Tribromophenol	7500 ug/mL	Restek	31083	Refrigerator 4 ±2°C
O-Terphenyl Surrogate	O-Terphenyl	10000 ug/mL	Restek	31097	Refrigerator 4 ±2°C

LCS and MS Standards

Standard	Components	Concentration	Manufacturer	Catalog #	Storage
PAH Standard	Naphthalene 2-Methylnaphthalene 1-Methylnaphthalene Acenaphthylene Acenapthene Fluorene Phenanthrene Anthracene Fluoranthene Pyrene Benzo(a)anthracene Chrysene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenz[a,h]anthracene Benzo(g,h,i)perylene	500 ug/mL	Accustandard	M-610-FL-R-5X	Refrigerator 4 ±2°C
BNA Standard - 70 Component Custom LCS Mix	70 Component Custom	200 ug/mL	Supelco	2008436	Freezer <-10ºC
BNA Standard - n-nitrosodiphenylamine	n-Nitrosodiphenylamine	5000 ug/mL	Supelco	46702-U	Freezer <-10ºC
Diesel Fuel #2	Diesel Fuel #2	50000µg/mL	Restek	32158	Refrigerator 4 ±2°C

9.5 Standard Preparation Procedures

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration of Standard	Amount Spiked into a Sample Aliquot	Concentration of Spike in Sample Aliquot
PAH Working Spike Solution	PAH Standard – 500ug/mL	NA	NA	500µg/mL	20µL	10 ug/mL
Working PAH Surrogate Stock Solution	Restek BN Mix Catalog #31082 – 5000 ug/mL	3000 uL	100mL of methylene chloride	150µg/mL	1000 µL	15 ug/mL
BNA Working Spike Solution	BNA Standard - 70 Component Custom LCS Mix – 200 ug/mL	NA	NA	200 ug/mL	250 uL	50 ug/mL
	BNA Standard - n-nitrosodiphenylamine 5000 ug/mL	NA	NA	5000 ug/mL	10 uL	
BNA Surrogate Spike Solution	Restek Acid Surrogate Mix for BNAs – 7500 ug/mL	5 mL	500mL of Methylene chloride	75 ug/mL	1000 uL	75 ug/mL
	Restek B/N Surrogate Mix for BNAs – 5000 ug/mL	5 mL		50 ug/mL		50 ug/mL
TPH Spike Standard Solution	Restek Diesel fuel #2 – 50,000 ug/mL	NA	NA	50,000 ug/mL	5 uL	1000 ug/mL
TPH Surrogate Spike Solution	Restek O-Terphenyl – 10000 ug/mL	NA	NA	10,000 ug/mL	5 uL	50 ug/mL

10. CALIBRATION

10.1 Analytical Balance Calibration

10.1.1 Annual Calibration - The balance must be calibrated at least annually by an outside agency and checked daily before each use using Class 1 or 2 weights. Refer to Pace SOP S-ALL-Q-013 *Support Equipment*.

10.2 Daily Calibration Check

10.2.1 Clean the balance and surrounding area prior to starting the daily calibration check.

10.2.2 Check the sight level on the balance. If it needs adjusting, level the balance.

10.2.3 The weight set ID indicated in the logbook is used as the primary set. If an alternate weight set ID is used, that ID must be recorded in the comment section of the balance calibration logbook for that day.

10.2.4 Tare the balance before weighing the NIST certified weights.

10.2.5 Use forceps or other means to lift each weight (Do not touch the weights with fingertips as the residue may artificially adjust the true value of the weights). Record the date of the calibration check, the true value of the weight, and the actual measured weight in the logbook. Repeat this procedure for the other certified weights. If calibration weights differ from the certified weights by more than specified in the balance calibration logbook, corrective action must be taken (see 10.3).

10.3 Corrective Action

10.3.1 Clean the balance and balance pan. Check the sight level on the balance and adjust if necessary. Re-tare and reweigh all the certified weights.

10.3.2 The internal calibration function (if available) of the balance may be used as a means of corrective action.

10.3.3 Utilize the internal calibration function and diagnostics. Refer to instrument manual.

10.3.4 Contact the QA office for assistance if the balance does not meet the calibration tolerances.

10.3.5 If the above action does not correct the problem, the balance should be taken out of service and appropriately labeled to avoid improper usage. A service technician should be contacted.

10.3.6 Record any corrective action. Initial and date all entries in the logbook.

10.4 Microwave calibration expressed as Power in Watts.

10.4.1 Use the following procedure to determine actual power output at any of the following levels: 400, 800 and 1600 Watts. Record all measurements in the instrument maintenance logbook.

10.4.2 Ensure that the solvent sensor assembly is disconnected prior to performing any power test on the MARS system.

10.4.3 Install the turntable in the microwave cavity.

10.4.4 With the main menu displayed, highlight “Load Method. “ Press the “SELECT” key. The directory menu screen will appear.

10.4.5 Use the arrow keys to highlight the CEM Directory and again press the "SELECT" key and the CEM Menu screen will appear.

10.4.6 Use the arrow keys to highlight one of the power tests 400W, 800W or 1600W Power test and press the "SELECT" key to return to the main menu.

10.4.7 Place 1000 mL of ambient temperature (18-22 ºC) deionized water in a 1000-mL PTFE or polypropylene beaker.

10.4.8 Using a thermometer with 0.1 ºC gradations, measure and record the initial temperature, T_i. Ensure that the thermometer is immersed to its indicated immersion line prior to reading the temperature.

10.4.9 Remove the thermometer from the beaker. Carefully place the beaker in Vessel #1 position on the microwave turntable. Gently close the door to avoid spilling any of the water.

10.4.10 Press "START".

10.4.11 At the end of the programming time (2 minutes), remove the beaker from the microwave cavity. Stir the water thoroughly for 30 seconds, measure and record the peak temperature reading. This is the final temperature, T_f.

10.4.12 The microwave output is calculated as follows: Power in Watts = $47(T_f-T_i)$.

10.4.13 At the 400W power test the calculated power must be > 340Watts, at the 800W power test the calculated power must be $\geq$ 680 Watts and at the 1600W power test the calculated power must be $\geq$ 1360 Watts.

10.4.14 The microwave power test must be performed weekly at one of the setting. It is recommended to use the 1600W setting since this is the closest to the settings used for our extraction method.

11. PROCEDURE

11.1 Extraction – Low Concentration Samples

11.1.1 Inspect the 75-mL microwave extraction vessels to ensure they are clean and dry. Pre-rinse vessels with appropriate solvent and discard the rinsate as waste.

11.1.2 Weigh a 30.0-g aliquot of each sample into a vessel, removing twigs, rocks and other foreign material (refer to SOP S-ALL-Q-021, *Sub-Sampling (Sample Homogenization)*).

11.1.3 For each batch of 20 samples, or less, prepare two additional sample aliquots. One will serve as a MS and the second as a MSD. Also prepare 2 aliquots of reagent sand to serve as the Method Blank and LCS.

11.1.4 Spiking of Samples.

11.1.4.1 PAH Samples

11.1.4.1.1 Add 100uL of the150 ug/mL surrogate spike solution to each sample aliquot, LCS, MB, MS, and MSD. In addition, add 20uL of the PAH spike mix at 500ug/mL to the LCS, MS and MSD samples.

11.1.4.2 BNA Samples

11.1.4.2.1 Add 1 mL of the 75/50 ug/mL surrogate spike solution to each sample aliquot, LCS, MB, MS, and MSD. In addition, add 250 uL of the LCS/Matrix spike mix @ 200ug/mL and 10 uL of N-nitrosodiphenylamine @ 5000 ug/mL to the LCS, MS and MSD samples.

11.1.4.3 TPH Samples

11.1.4.3.1 Add 5 µL of the 10,000 µg/mL surrogate standard to each sample aliquot, LCS, MB, MS and MSD. In addition, add 20 µl of Diesel fuel #2 standard at 50,000 µg.mL to the LCS, MS and MSD samples

11.1.5 Immediately add 25 mL of CH_2Cl_2 to each TPH aliquot or CH_2Cl_2/Acetone at a 4 to 1 ratio to each PAH/BNA aliquot, add the vessel plug and screw on the vent cap. Tighten the cap by either using the capping station or hand tightening. Note: (1) When hand-tightening, finger tighten then turn approximately 30 degrees more. (2) When using the Capping station, turn on the capping station and insert the vessel with cap while holding onto the vessel. Keep the vessel in the station until the motor stops turning and remove. The Capping station has a preset torque and doesn't require adjusting. DO NOT re-torque vessels without completely loosening the covers first.

Note: Other solvent blends may be used based on project requirements. A MDL and IDC study must be performed if different solvent blends are used.

11.1.6 Insert each vessel into a high-pressure microwave sleeve. Make sure the sleeve is pushed up tight against the bottom of the vessel cap; otherwise the vessel may explode during the extraction process.

11.1.7 Add vessels to microwave carousel. Make sure vessels are evenly spaced around the carousel in order for the heating sensors to work properly. This only becomes an issue if you have less than a full circle of vessels on the carousel.

11.1.8 Turn on the microwave extractor and set the microwave extractor to method 3546 Large and press "START". This method is set as follows:

11.1.8.1 Power setting: 1600 w at 100%

11.1.8.2 Ramp rate: 10 minutes to a final temperature of 115 ºc.

11.1.8.3 Hold at 115 ºc for 10 minutes

11.1.8.4 Note: if less than 12 samples are extracted within the batch, use method 3546 small. The method is set as follows:

11.1.8.5 Power setting: 800 w at 100%

11.1.8.6 Ramp rate: 10 minutes to a final temperature of 115 ºc.

11.1.8.7 Hold at 115 ºc for 10 minutes

11.1.9 Allow the vessels to cool to room temperature (~ 30 minutes). Empty the entire contents of the microwave vessel into a filtering funnel pre-assembled with a glass wool plug and sodium sulfate. The sample extract is collected in a 60 mL disposable vial set underneath the funnel. Rinse the microwave vessel with approximately 15 mL of $CH_2Cl_{2\ to}$ collect any remaining sample residue and pour into the filter funnel. Once the remaining sample residue is poured into the funnel, rinse the sodium sulfate with $CH_2Cl_{2\ and}$ collect gravimetrically into the 60 mL vessel. After all sample extract has been filtered, proceed to concentration.

11.2 Extraction – High Concentration Samples

11.2.1 The criterion is the same as Low Concentration sample extraction procedure, except 2g of sample volume is used instead of 30g.

11.3 Concentration

11.3.1 Water Bath Concentration

11.3.1.1 Add 1-2 boiling chips to each 60 mL disposable vial containing sample extract and attach a three-ball micro-Snyder column.

11.3.1.2 Place the sample on the water bath with the bottom of the 60 mL vial partially immersed in water. The water bath temperature should range between 75° to 85° C. After heating to temperature, the balls in the micro-Snyder column should actively chatter but not flood with solvent.

11.3.1.3 Remove the 60 mL vial from the water bath when the extract volume has concentrated to approximately 500 uL. After the unit has cooled, rinse the snyder column with CH_2Cl_2, collect solvent into the vial and remove the snyder column. Transfer the sample aliquot into a 2 mL autosampler vial marked and verified with 0.5, 1 and 1.5 mL volume markings and adjust the final volume to 1 mL.

11.3.2 If GPC cleanup is required as listed in section 12.4, the same concentration technique listed above should be followed but the sample aliquot volume should be taken to approximately 7mL, filtered with a glass syringe and accudisc filter and brought to a 10mL final volume. The sample is now ready to be cleaned by GPC

11.4 Extract Cleanup and Concentration

11.4.1 GPC Cleanup of Extracts: Sample extracts may have contaminants removed using Gel Permeation Chromatography following the latest version of S-GB-O-032 *Gel Permeation Chromatography*. All QC samples must be subjected to the same cleanup criteria as the samples. GPC cleanup is performed on a project basis.

11.4.2 Once all samples and QC have been subjected to GPC cleanup, assemble a K-D concentrator apparatus by attaching a 10-mL concentrator tube to a 300 ml evaporative flask. Quantitatively transfer GPC'd extract into K-D concentrators with two or three rinses of methylene chloride. Add one or two boiling chips to concentrator apparatus and attach a three-ball macro-snyder column.

11.4.3 Place the K-D apparatus on a warm water bath (75° to 85° C) so that the concentrator tube is totally immersed in the warm water and the entire lower rounded surface of the flask is bathed with water. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of the liquid reaches 3 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

11.4.4 Rinse the Snyder column and the evaporating flask with a small amount of methylene chloride. Remove Snyder column and evaporating flask.

11.4.5 Final concentration of the sample extract can be performed by either the Nitrogen Blowdown method or the Micro-snyder column method as follows:

11.4.6 Nitrogen Blowdown Concentration Method: Concentrate each sample to approximately 0.3 ml with nitrogen. Rinse the internal wall of the concentrator tube several times with methylene chloride during the nitrogen blowdown. DO NOT ALLOW THE EXTRACT TO BECOME DRY, OR NEARLY DRY AS THIS WILL RESULT IN LOSS OF ANALYTES. Adjust to a final volume of 0.5 ml using methylene chloride by rinsing down the walls of the concentrator tube. Transfer 0.5 ml of the extract to a prelabelled injection vial and cap with an injection cover.

11.4.7 Micro-Snyder Column Concentration Method: Add a clean boiling chip and attach a two-ball micro-Snyder column to the concentrator tube. Place the apparatus in the small water bath (80° to 85° C). At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the liquid reaches an apparent volume of 2 ml, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. DO NOT ALLOW THE EXTRACT TO BECOME DRY, OR NEARLY DRY AS THIS WILL RESULT IN LOSS OF ANALYTES. Remove the Snyder column and rinse its lower joint with approximately 0.2 ml of methylene chloride into the concentrator tube. Use the Nitrogen Blowdown Concentration Method to further concentrate the extract to a final volume of 0.3 ml. Adjust to a final volume of 0.5 ml using methylene chloride by rinsing down the walls of the concentrator tube. Transfer 0.5 ml of the extract to a prelabelled injection vial and cap with an injection cover.

12 QUALITY CONTROL

12.1 All Quality Control measures shall be subjected to exactly the same preparation procedures as those used on actual samples.

12.2 Quality Control measures shall be assessed on an on going basis to determine the usability of the data. The controls measure contamination and statistical measures of accuracy and bias.

12.3 Method blanks shall be performed at a frequency of one per extraction batch, not to exceed 20 environmental samples. This is a negative control used to assess contamination during the preparation process.

12.4 Laboratory Control Spike (LCS) is performed at a frequency of one LCS per extraction batch, not to exceed 20 environmental samples. This is a positive control used to assess the manner in which the samples are prepared.

12.5 A matrix spike (MS) and a matrix spike duplicate (MSD) must be performed for every 20 samples when appropriate sample volume is present; otherwise a laboratory control spike duplicate will be performed. Matrix spikes are used to indicate matrix effects on the analysis of the analytes of interest. The sample used for the MS/D pair is either determined by the client or selected at random from client samples as sample volume allows.

12.6 Surrogate standards must be added to all samples, laboratory control spikes, matrix spikes, and method blanks prior to extraction. Surrogates are used to monitor the efficiency of the method on each sample and possible matrix related effects.

12.7 All quality control samples (MB, LCS, MS, MSD, and duplicate samples) must be analyzed by the same determinative methods as the samples in the batch. The acceptance criteria and corrective actions are described in the determinative method SOPs.

12.8 See Appendix A Table G for a summary.

13 METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA Office.

14 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

14.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

15 REFERENCES

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 3546, "Microwave Extraction", Draft Update IVB, 11/2000.

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 3500B, "Organic Extraction and Sample Preparation", Update III, 12/1996.

15.3 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-044, *Determination of Low Level PAHs by GC/MS-SIM in Solid and Biological Matrices*.

15.4 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-032 *Gel Permeation Chromatography*.

15.5 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-049 *Determination of Semi-volatile Organics by GC/MS*

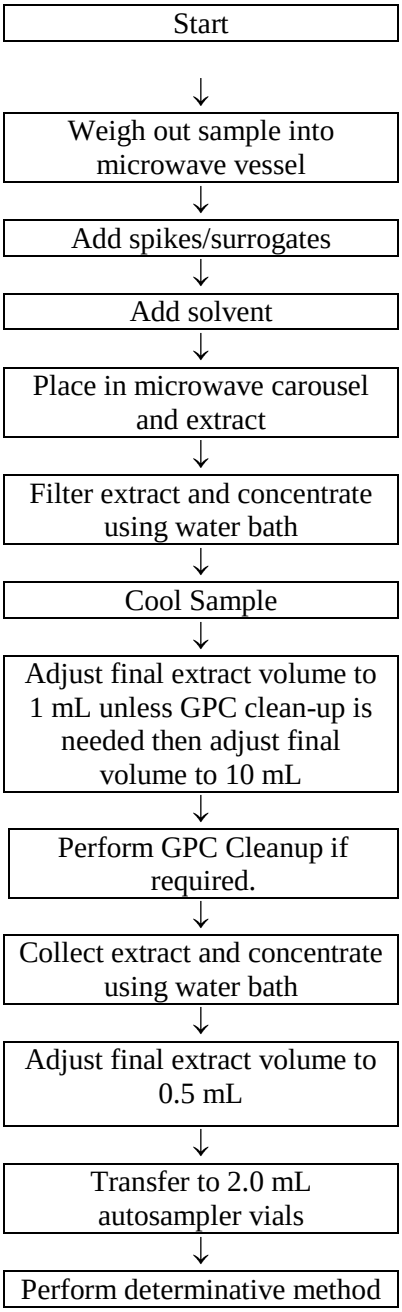
15.6 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-050 *Determination of Semi-volatile Organics by GC/MS (Selective Ion Monitoring).*.

15.7 Pace Analytical Services, Inc. – Green Bay, SOP S-GB-O-023 *Total Petroleum Hydrocarbon.*

15.8 Pace Analytical Services, Inc – Green Bay, SOP S-ALL-Q-021, *Sub-Sampling (Sample Homogenization)*

16 TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

FLOWCHART



Appendix A

Tables

Table A **Required Safety Equipment**

Table B **Equipment**

Table C **Supplies**

Table D **Reagents**

Table E **Standards**

Table F **Sample Collection**

Table G **Quality Control**

Table A

REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses	x	
2	Lab Coat	x	
3	Gloves	x	
4	Face Shield		x

Table B
EQUIPMENT

Equipment	**Manufacturer**	**Model(s)**	**Serial #**	**Date In Service**
Microwave Extractor	CEM	907501	MD9414	July 2007
Heated Water Bath	Organimation	N-EVAP	N/A	March 1992
Pressure Filter Apparatus	N/A	N/A	N/A	July 2007

Table C
SUPPLIES

Supplies	Manufacturer*	Vendor*	Catalog #
2 mL autosampler vials	VWR	VWR	66020-953
Aluminum crimp seals	VWR	VWR	66010-847
Rubber pipette bulb	NJ Rubber	VWR	R5002-2
N-Dex Nitrile Glove Powder free (medium)	Best	Fisher Scientific	6005 PFM
N-Dex Nitrile Glove Powder free (large)	Best	Fisher Scientific	7005L
N-Dex Nitrile Glove (extra large)	Best	Fisher Scientific	7005XL
Labtone glassware detergent	VWR	VWR	21851-006
Tube and bottle brush	VWR	VWR	PK-5-17041-002
5.75" disposable pipets	Fisher Scientific	Fisher Scientific	13-678-20-A
Disposable culture tubes	VWR	VWR	60825-618
25 mL adjustable repipetor	VWR	VWR	40000-066
Lab notebooks	VWR	VWR	51280-108
600 mL beakers	Kimble	Fisher Scientific	13910-145
250 mL beakers	Kimble	Fisher Scientific	14005-250
150 mL beakers	Kimble	Fisher Scientific	02539J
60 degree powder funnel	Pyrex	VWR	30240-047
2.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	20-814-B
5.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	29623-300
25.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-A
50.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-B
9 oz Amber jars	QEC	QEC	2124-0009
33 ml stainless steel cells	Dionex	Dionex	049562
Cellulose filters	Dionex	Dionex	049458
60 mL collection vials	I-Chem	VWR	405531
250 mL K-D flasks	HGF	HGF	
25 mL Concentrator Tubes	HGF	HGF	
10 mL Concentrator Tubes	HGF	HGF	
Micro distilling columns	Kontes	VWR	569261-0324
Stainless Steel Spatula	VWR	VWR	
25 uL gastight syringe	Hamilton	Fisher Scientific	24559
50 uL gastight syringe	Hamilton	Fisher Scientific	111111
100 uL gastight syringe	Hamilton	Fisher Scientific	13-684-100
250 uL gastight syringe	Hamilton	Fisher Scientific	81100
500 uL gastight syringe	Hamilton	Fisher Scientific	81217
1000 uL gastight syringe	Hamilton	Fisher Scientific	1482425
Pyrex baking dish	Corning	---	6001078

*** Equivalent supplies may be used.**

Table D

REAGENTS

Reagent	Purity	Manufacturer	Vendor	Catalog #
Methylene Chloride	Pesticide Quality	Burdick & Jackson	Fisher Scientific	CS300-200
Acetone	Pesticide Quality	Burdick & Jackson	Fisher Scientific	010-4
Di-ionized Water	Type I ASTM	----	----	----
Ottawa Sand	-----	Fisher Scientific	Fisher Scientific	S23-3
Sodium sulfate (anhydrous; 10-60 mesh)	ASC grade	Fisher Scientific	Fisher Scientific	MK802406
Nitrogen	99.99%	Whatman	----	----

Table E
STANDARDS

Standard	Acronym	Concentration	Direction found in Section:	Alias
SV Internal Standard	Stock	4000 ppm	Reagents & Standards	Int. Std. Stock
B/N Surrogate Mix	Stock	5000 PPM	Reagents & Standards	Surr. Stock
PAH Standard Mix	Stock	100 PPM	Reagents & Standards	Cal. Stock
PAH Standard Mix	Stock	500 PPM	Reagents & Standards	Spike Stock
PAH Internal Standard (Soil)	Int. Std. Inter.	2000 PPM	Reagents & Standards	Int. Std. Inter.
B/N Surrogate Standard	Surr. Inter.	150 PPM	Reagents & Standards	Surr Spike
SV Acid Surrogate Mix	Stock	7500 ug/mL	Reagents & Standards	Surr Stock
BNA Custom Standard Mix	Stock	200 ug/mL	Reagents & Standards	Spike Stock
N-Nitrosodiphenylamine	Stock	5000 ug/mL	Reagents & Standards	Spike Stock
O-Terphenyl	Stock	10000 ug/mL	Reagent & Standards	Surr Stock
Diesel Fuel #2	Stock	50000 ug/mL	Reagent & Standards	Spike Stock

Table F
SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
SOIL	SW 846 3546	Amber jar with teflon lined lid	None	On ice 4 ± 2° Celsius	Refrigerate 4 ± 2° Celsius

Table G
QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 8270C**
Method Blank	One per batch of samples, up to 20 enviromental samples, whichever is more frequent.
Laboratory Control Spike	One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Method Validation	Annually
MDL	Annually
Surrogate Standards	Added to every sample.
Pace Reporting Limit Standard (PRLS)	After every calibration and monthly thereafter

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 8015B**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Laboratory Control Spike	One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Method Validation	Annually
MDL	Annually
Surrogate Standards	Added to every sample.
Pace Reporting Limit Standard (PRLS)	After every calibration and monthly thereafter

17 REVISIONS

Document Number	Reason for Change	Date
S-GB-O-045-Rev. 0	First Issue	21Sept2007
S-GB-O-045-Rev.1	Added Base/Neutral/Acid Information to SOP Section 12.1.9 – Added filtering can be done gravimetrically.	24Mar2008
S-GB-O-045-Rev.02	Deleted Section 7 Responsibilities Added TPH information to SOP Updated Flowchart. Section 11.1.5 – Added CH_2Cl_2/Acetone at a 4 to 1 ratio for PAH/BNA. Section 11.1.9 – Removed pressure filter working. Table G – Added Pace reporting limit standard working.	17Mar2010
S-GB-O-045-Rev.03	Updated Signature Page Section 9.5: Changed LCS Mix in standard and final concentration of standard to 200ug/mL Section 11.1.4.2.1: Changed LCS/Matrix spike mix@2000ug/mL to 200ug/mL	28Apr2010

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STANDARD OPERATING PROCEDURE

Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by SW846-8082A

Reference Methods: SW-846 Method 8082A

SOP NUMBER:	S-GB-O-047-REV.02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-047-REV.01

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 9/15/10

Kate Grams, Laboratory Quality Manager — Date: 9/14/10

Glen Coder, Department Manager — Date: 9/15/10

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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17. REVISIONS **17**

1. PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to determine the concentration of PCBs in water, soil, sediment, waste, and biological samples in accordance with SW846 Method 8082A. Samples for analysis are prepared by SW846 Method 3510C, 3540C, 3541, and 3580A. See Section 15 for list of reference SOPs.

2. SCOPE AND APPLICATION

2.1 This method is used to determine the concentration of PCBs in extracts prepared from water, soil, sediment, waste, and biological samples. A list of the Aroclors routinely analyzed, their CAS numbers and Pace Reporting Levels (PRLs) are shown in Section 16, Table A. PRLs are subject to change based on current analytical system performance and actual sample matrices.

2.2 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/electron capture detection (GC/ECD) systems and interpretation of complex chromatograms. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results

3. SUMMARY OF METHOD

3.1 Sample extracts are prepared for analysis by the appropriate sample preparation method. The procedures for extract preparation are described in separate SOPs. A volume of sample extract is injected into a GC and compounds in the effluent are detected by an ECD based on an operating program set up to achieve optimum separation and quantitation of target analytes.

3.2 Retention time windows, in combination with characteristic elution patterns from a dual-column analysis, are used in the identification of PCBs as Aroclors.

3.3 PCBs are quantified as Aroclor mixtures by comparison of their ECD response on a single column with a calibration curve(s) constructed from the response(s) of authentic standards.

3.4 Results are reported in parts per billion (µg/kg or µg/L). Soil and sediment sample results are corrected for moisture and reported on a dry weight basis. Biological results are reported based on wet weight, or “as is” basis.

4. INTERFERENCES

4.1 Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that leads to discrete artifacts and/or elevated baselines. Phthalate esters are common contaminants that result from contact with flexible plastics. Contact with common plastics or rubber products must be avoided. Lab ware should be constructed of glass, stainless steel, or PTFE, must be thoroughly cleaned and dried prior to use, and should be rinsed with the appropriate solvent immediately before use.

4.2 Elemental sulfur is a common environmental contaminant in many soil, sediment and leachate samples, producing a broad peak that will confound analysis of early eluting analytes. Sulfur may be removed from extracts by treatment with elemental mercury, copper powder, or similar procedure described in a separate SOP.

4.3 Waxes, lipids, and other similar high molecular weight materials may be co-extracted from samples typically resulting in baseline elevation during GC analysis. These interferences may be removed by sulfuric acid clean up and/or column chromatography cleanup using Florisil or gel permeation chromatography (GPC), all of which are described in separate SOPs. Other halogenated pesticides and similar industrial chemicals, which can interfere with analytes of interest, may be removed by these procedures as well.

4.4 All solvents, reagents, glassware, and sample processing hardware must be routinely demonstrated to be free from interferences under the conditions of the analysis by monitoring method blanks and taking corrective action as required.

5. SAFETY

5.1 The toxicity, or carcinogenicity, of many chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each analyst is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. 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.

5.2 Take precautions when handling samples. Samples should always be treated as potentially hazardous. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.

5.3 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training.

5.4 PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Primary standards of these toxic compounds should be prepared in a hood.

6. DEFINITIONS

6.1 All applicable definitions can be found in Section 10.0 of the PASI Quality Manual.

6.2 **Extract** – A solution of contaminants extracted and concentrated from a sample.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 **General Procedures** – Procedures for sample collection, preservation, and handling are described in the separate sample preparation SOPs.

7.2 **Holding Times**

7.2.1 **Water Samples** – Samples must be extracted within 7 days of collection.

7.2.2 **Soil, Solid, and Other Waste Samples** – Samples must be extracted within 14 days of collection.

7.2.3 **Biological Samples** – Samples remain frozen at ≤-10ºC for up to one year or longer per client request prior to extraction; typical extraction hold times do not apply.

7.2.4 **Extracts** – Extracts must be analyzed within 40 days of preparation.

7.3 **Sample Storage** – Store all samples and extracts in the dark at 4 ± 2°C. Biological samples must be stored at or below -10°C until extracted.

8. REAGENTS AND STANDARDS

8.1 **Solvents** – Hexane and acetone, pesticide grade (Section 16, Table B). All solvents are stored at room temperature and environmental conditions.

8.2 **Analytical Standards** – Prepared from stock standard solutions and are required for initial calibration and continuing calibration checks (Section 16, Table C). The following describes the contents of each type of solution:

8.2.1 **Calibration and Calibration Check Standards** – Five concentration levels of calibration solutions are prepared containing equal amounts of Aroclors 1016 and 1260 (combined in the same solution named AR1660 throughout this document), as well as the surrogates decachlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TCMX). A single point calibration standard is required for the other Aroclor mixtures preferably at the mid-point level of the AR1016/1260 curve. A calibration check solution (ICV) is also prepared at the mid-level concentration of AR1016/1260 from second source materials.

8.2.2 **Surrogate Standard Spiking Solution** – contains decachlorobiphenyl (DCB) and 2,4,5,6-tetrachloro-m-xylene (TCMX) and is spiked into all samples prior to extraction.

8.2.3 **Matrix Spiking Solutions** – contain an Aroclor mixture that is spiked into all appropriate QC samples (LCS, MS, and MSD) prior to extraction. The Aroclor(s) spiked and/or spike amounts may be adjusted when prior knowledge of the type or concentration of Aroclor(s) present in the sample matrix is known, or to comply with project requirements.

8.3 **Preparation of Analytical Standard Solutions** – Standards are prepared from commercially available stock solutions. The sources of the stock solutions, recipes for preparing dilutions and working standards, and concentrations in all solutions are shown in Section 16, Table D. All standards are prepared in hexane and stored in amber vials with PTFE-lined screw caps at 4 ºC or colder.

8.4 **Stability of Analytical Standards** – Stock solutions of Aroclor mixtures must be replaced within 1 year of preparation. All dilutions and working standard solutions must be replaced within 6 months of preparation or sooner if the standards show signs of degradation. As each standard from the vendor is opened, record all pertinent information in the stock standard logbook. Record all standard preparations in the working standard logbook

9. EQUIPMENT AND SUPPLIES

9.1 Instrumentation

9.1.1 **GC** – Hewlett Packard (HP) 5890 equipped with dual ECDs or HP 6890 equipped with dual µECDs.

9.1.2 **GC Autosampler** – HP 7673A (5890) or HP 7863 (6890).

9.1.3 **GC Columns** – Two of the following capillary columns may be used:

9.1.3.1 RTX CLPesticides I, 30m x 0.32mm I.D. (Restek)

9.1.3.2 RTX CLPesticides II, 30m x 0.32mm I.D. (Restek)

9.1.3.3 DB-1701, 30m x 0.32mm I.D. (J&W Scientific)

9.1.3.4 DB-5, 30m x 0.32mm I.D. (J&W Scientific)

9.1.4 **Data Processor** – TurboChrom IV or HP ChemStation.

9.1.5 **Printer** – HP LaserJet 5Si or equivalent.

9.2 Glassware and Materials

9.2.1 **Gastight Syringes** – any size ranging from 10µL to 1000µL (Hamilton series 1000 or equivalent)

9.2.2 **Autosampler Vials** – 1.8mL with crimp caps

10. CALIBRATION

10.1 Initial Calibration (ICAL)

10.1.1 Analysis of Standards

10.1.1.1 The initial calibration includes analysis of a five-point calibration curve of AR1660 at concentrations of 0.1, 0.3, 0.5, 0.8, and 1.0µg/mL, which includes TCMX and DCB at concentrations of 0.01, 0.02, 0.05, 0.1, and 0.15µg/mL respectively. Inject a single point standard of Aroclors 1221, 1232, 1242, 1248, 1254, and 1268 at 0.5µg/mL.

10.1.1.2 Other calibration ranges may be substituted to meet expected concentrations of samples to be analyzed. If historical data indicates a specific Aroclor is present, a five point initial calibration may be performed for the Aroclor of concern instead of using the AR1660 mixture.

10.1.1.3 A minimum of five (preferably seven) peaks must be selected for each Aroclor. The peaks chosen for quantitation should be at least 25% of the height of the largest peak in each Aroclor and should have minimal co-elution with the peaks of other Aroclors.

10.1.2 **Retention Time (RT)** – Retention time windows are used for compound identifications in samples. The RT for all components in all standards must be within the windows specified for both columns.

10.1.2.1 Make at least three injections of all analytes of interest over a 72-hour period.

10.1.2.2 Record the retention time for each selected peak for each Aroclor mixture, to three decimal places. Calculate the mean and standard deviation for each peak.

10.1.2.3 The width of the retention time window is defined as ± 3 standard deviations of the mean established. The minimum retention window will be ± 0.03 minutes.

10.1.2.4 Establish the center of the RT window for each Aroclor mixture and surrogate using the absolute RT from the calibration verification standard at the beginning of the analytical shift. Optionally, the Initial Calibration RT windows may continue to be used as long as method criteria are met. For samples run during the same shift as an initial calibration, use the RT of the mid-point standard in the Initial calibration as the center of the RT window.

10.1.2.5 When conducting Aroclor analysis, it is important to determine that common single-component pesticides such as DDT, DDD, and DDE do not elute at the same retention times as the target Arolcors. In conjunction with determining the retention time windows, the analyst should analyze a standard containing the DDT analogs. The standard only needs to be analyzed when the retention time windows are being determined. It is not part of the routine initial calibration or calibration verification steps in the method, nor are there any performance criteria with the analysis of the standard. If it is determined that any of the DDT analogs elute at the same retention time as an Arolcor peak that was chosen for quantitation, then the analyst must either adjust the GC conditions to achieve better resolution, or choose another peak that is characteristic of that Arolcor and does not correspond to a peak from a DDT analog.

10.1.3 **Response Factors (RF)** – Individually tabulate the area responses for each of the five or more peaks selected for each Aroclor versus concentration of the five-point calibration standards for each GC column. Calculate RF for each peak using the following equation:

$$RF = \frac{A_x}{C_x}$$

Where:
A_x = Total area of analyte response.
C_x = Concentration of the analyte in the solution (µg/mL).

10.1.4 Acceptance Criteria – The percent relative standard deviation (%RSD) of the five calibration factors for each peak of each Aroclor, (1016 and 1260) along with the surrogates must be ≤ 20%. If this is the case, linearity can be assumed, and the average RF can be used for quantitation. If the %RSD is >20%, a linear calibration curve may be used if the correlation coefficient is ≥ 0.99. **The results for both columns must meet calibration acceptance criteria.Calibration Verification**

10.2 **Initial Calibration Verification (ICV)** – In order to consider the initial calibration acceptable, an ICV standard must be analyzed. The ICV standard must be from a second source stock and meet the same criteria as the continuing calibration verification standard before the initial calibration may be considered valid.

10.2.1 **Continuing Calibration Verification (CCV)** – A midpoint calibration check standard must be injected at the beginning and end of each 12-hour analysis period, and at intervals of not less than once every 20 samples, for calibration verification. If the response factor (area/concentration) of the check standard deviates by more than 20% from the initial average response factor, the calibration is considered out of control and analysis must be stopped.

10.2.2 **Acceptance Criteria** – The percent difference (%D) is determined for every analyte and must be within ±20% of the calibration curve. Calculate %D for each peak using the following equation:

$$\%D = \left(\frac{R_1 - R_2}{R_1} \right) X 100$$

Where:
R_1 = Mean Response factor from the ICAL
R_2 = RF calculated from the CCV

10.2.2.1 First determine whether the average %D for all of the peaks for each specific Aroclor with a five-point calibration is ≤ 20%. Each individual Aroclor must be evaluated separately. For example, the average %D for all of the peaks used for quantitation of AR1016 must be ≤ 20%. If the Aroclors themselves are acceptable, evaluate the %D for each surrogate. If the %D is ≤ 20% for each individual Aroclor and surrogate, the continuing meets the acceptance criteria.

10.2.2.2 If the ending calibration verification standard exceeds 20%D criteria on the high side (i.e., an increase in sensitivity) samples that had no Aroclors detected do not need to be reanalyzed. If the continuing calibration standard criterion is exceeded on the low side (i.e., a drop in sensitivity), all samples analyzed since the last acceptable CCV must be re-analyzed.

10.2.3 **All samples must be bracketed by acceptable calibration verifications on both columns.** Perform corrective action such as injection port or column maintenance. Prior to the analysis of any subsequent samples acceptable calibration verification must be established. In the event that this cannot be achieved, a new initial calibration must be performed.

10.2.4 Reporting Limit Verification Standard (RLVS) – For every five point Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) must also be analyzed. The RLVS is analyzed prior to any samples being analyzed, and monthly thereafter. The limits are +/- 40% of the true concentration. The analysis of this standard demonstrates the instruments ability to report down to the reporting limit with known accuracy. If outside the limits, reevaluate the low level standards. If still outside the limits, recalibrate.

11 PROCEDURE

11.1 Sample Preparation – All sample extracts and standard solutions must be allowed to warm to room temperature before analysis.

11.2 GC/ECD System Preparation – Verify instrument parameters as set up for current operating conditions.

11.2.1 GC Column Conditions

Carrier Gas	UHP Helium
Flow Rate	3.4 mL/min.
Make-up Gas	UHP Nitrogen
Flow Rate	35.0 mL/min.
Detector Temp.	300°C
Injector Temp.	205°C
Injection	Splitless

11.2.2 GC Temperature Program

Initial Temp.	110°C
Initial Time	1.5 min.
Rate 1	20.00°C/min.
Final Temp. 1	140°C
Final Time 1	0.00 min.
Rate 2	11.00°C/min.
Final Temp. 2	280°C
Final Time 2	5.00 min.
Rate 3	20.00°C/min.
Final Temp. 3	300°C
Final Time 3	3.00 min.

11.3 **Batch Sequence** – Generate a sequence to run a batch of samples and the associated quality control samples.

11.3.1 **Initial Calibration** – For example, the batch for initial calibration should include the following:

Series of 2-3 Primes
Solvent Blank (Hexane)
AR1660-1 (0.1µg/mL)
AR1660-2 (0.3µg/mL)
AR1660-3 (0.5µg/mL)
AR1660-4 (0.8µg/mL)
AR1660-5 (1.0µg/mL)
Solvent Blank (Hexane)
AR1660-3 ICV
AR1660-1 (0.1µg/mL) (PRLS)

11.3.2 **Sample Analysis** – For example, the typical batch for analysis of PCBs should include the following:

AR1660-301 CCV (0.5µg/mL)
(20 samples or 12-hour period)
Method Blank
Laboratory Control Spike
Samples
Matrix Spike/Matrix Spike Duplicate
Duplicate Sample(s)
AR1660-302 CCV (0.5µg/mL)

11.4 **Load Autosampler** – Load the autosampler with the appropriate primes, solvent blanks, standards and samples for the batch as it was created.

11.5 **Analyze Samples** – Analyze all standards, quality control samples, and environmental samples.

11.5.1 The method blank and LCS extracted along with the samples should be analyzed on the same instrument as the samples.

11.5.2 If the analyst determines that interferences could be removed by sulfuric acid cleanup and/or sulfur removal, then the analyst will perform the necessary cleanups and re-analyze the samples. The blank and LCS will also undergo the same cleanups and be re-analyzed.

11.6 Qualitative Analysis of Results

11.6.1 **Identification**

11.6.1.1 To be identified as an Aroclor, peaks present in a sample extract must fall within the established retention time window for a specific Aroclor. Once the Aroclor pattern has been tentatively identified, compare the responses of 3 to 10 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. Overlay comparison of sample chromatograms with standard chromatograms may be required to clearly identify patterns.

11.6.1.2 Since the chromatograms for many Aroclor mixtures overlap, the presence of multiple mixtures may complicate their quantitation. Also, environmental "weathering" of PCBs may complicate reliable identification and quantitation.

11.6.2 **Confirmation**

11.6.2.1 Confirmation is generally required using a second GC column of dissimilar stationary phase. When dual-column analysis is performed for confirmation, the same initial and continuing calibration criteria apply to both columns. If analyte concentrations are sufficient, identifications may be confirmed by GC/MS by analyzing the same extract by SW846 Method 8270.

11.6.2.2 Since Aroclors provide distinct multiple peak patterns which may be identified by an experienced analyst, confirmation on the second column may be based upon pattern recognition.

11.7 Calculate Results

11.7.1 The amount of Aroclor is calculated using the individual response factor (single point) for each of the 7 characteristic peaks chosen for quantitation of that specific Aroclor. If Aroclor 1016 and/or 1260 is being quantified use the average response factor from the AR1660 curve. Use the single point response factor from the initial calibration for all other Aroclors. Surrogates are quantified based on the average response factors for TCMX and DCB analyzed with the AR1660 curve. A concentration is determined using each of the characteristic peaks and then those 7 concentrations are averaged to determine the on-column concentration of that Aroclor.

11.7.2 If the initial on-column result of a sample extract exceeds the calibration range, the extract must be diluted and re-analyzed. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve. The GC data system will calculate concentration of each parameter as µg/mL on-column in the extract. Concentrations in samples are then calculated based on sample size, total volume of the final extract, any dilution factor, and any correction factor.

11.7.2.1 Water and Water-Miscible Waste Samples

$$\text{Final Concentration } (\mu\text{g/L}) = \frac{(C_x)(DF)(U_f)(V_t)}{(V_i)(V_o)}$$

Where:
C_x = On-column concentration in extract (µg/mL).
DF = Dilution factor.
U_f = Correction factor.
V_t = Volume of final extract (µL).
V_i = Volume injected (µL).
V_o = Volume of water sample extracted (mL).

11.7.2.2 Soil/Solid, Waste and Biological Samples

$$\text{Final Concentration } (\mu\text{g/Kg}) = \frac{(C_x)(DF)(U_f)(V_t)}{(V_i)(W_s)(S)}$$

Where:
C_x = On-column concentration in extract (µg/mL).
DF = Dilution factor.
U_f = Correction factor.
V_t = Volume of final extract (µL).
V_i = Volume injected (µL).
W_s = Weight of sample extracted (g).
S = Percent Solids (biological samples not corrected for percent solids).

11.8 **Quality Control Results** – Calculate recoveries for the surrogates in all samples; spiked analytes in LCS and MS/MSD samples; and Relative Percent Differences (RPD) for duplicate and MS/MSD samples.

12 QUALITY CONTROL

12.1 Calibration Checks

12.1.1 **ICAL** – If initial calibration criteria are not met, check standards preparation procedure for errors. Prepare new standards as required and re-run the calibration.

12.1.2 **Continuing Calibration Verification** – If the CCV criteria are not met, check system parameters, identify and correct likely causes, and re-run the check. An acceptable check is required to report sample results for the applicable batch.

12.1.3 **Reporting Limit Verification Standard (RLVS)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and monthly thereafter, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate. The AR1660 0.1µg/mL initial calibration standard is used as the PRLS

12.2 **Surrogate Recoveries** – Surrogate compound(s) must be added to all samples, spikes, control samples and method blanks, prior to analysis as indicators of method accuracy. Laboratory-based accuracy limits should be used for acceptance criteria. For Biota samples, if laboratory limits chart to narrow, limits will be set to 60-130%. If these criteria are not met, check system parameters, identify and correct likely causes, and re-run the samples.

12.2.1 If **both** surrogate recoveries fail this criterion, re-extraction of the sample may be necessary. If surrogate recoveries are higher than the acceptance criteria and target compounds are less than the reporting limit, the results may be reported with an appropriate footnote. If recoveries appear out of control due to sample matrix, report the results with an appropriate footnote.

12.2.2 One surrogate is allowed to be outside of the control limits. For instance, if an interfering peak obscures one surrogate, then that one surrogate may be excluded. The surrogate is considered diluted out and not evaluated when the dilution performed brings the theoretical on-column concentration below the concentration of the low standard in the initial calibration curve.

12.2.3 For samples run from the state of South Carolina, both surrogates must recover between 70-130%.

12.3 **Method Blank** –The method blank must not contain analyte responses at or above the reporting limit. If the results are not acceptable, re-analyze the method blank. If the problem persists, conduct maintenance to clean the analytical system. An acceptable method blank is required to report sample results for the applicable batch.

12.3.1 One surrogate is allowed to be outside of the control limits. For instance, if an interfering peak obscures one surrogate, then that one surrogate may be excluded. The surrogate is considered diluted out and not evaluated when the dilution performed brings the theoretical on-column concentration below the concentration of the low standard in the initial calibration curve.

12.3.2 If the blank contains any analyte of interest above the reporting limit, all of the associated samples, matrix spikes, and laboratory control spikes **must** be re-extracted unless the sample concentration is greater than 20X the amount found in the blank or the analyte is not detected in an associated sample. For Wisconsin projects this criteria will be “Above the LOD”.

12.4 **LCS Recoveries** – One LCS must be analyzed with each batch of 20 samples. Laboratory-based accuracy limits should be used to for acceptance criteria. For Biota samples, if laboratory limits chart to narrow, limits will be set to 60-130%. An acceptable LCS is required to report sample results for the applicable batch.

12.4.1 If the laboratory control spike does not meet the recovery criteria, the results of all QC performed with the batch will be evaluated by the analyst. Corrective actions include re-extraction of the samples or reanalysis of the extracts.

12.4.2 For samples run from the state of South Carolina, Ar1016 and 1260 must recover between 70-130%.

12.5 One LCSD must be analyzed with each batch of 20 samples if inadequate sample is available to perform a MS/MSD. Laboratory-based accuracy limits should be used to for acceptance criteria. An acceptable LCSD is required to report sample results for the applicable batch.

12.6 **MS/MSD Recoveries** – One MS/MSD pair should be analyzed with each batch of 20 samples. Laboratory-based accuracy limits should be used to for acceptance criteria. The sample use for the MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows.

12.6.1 If a matrix spike recovery fails this criterion, the recovery of the other spiked sample in the MS/MSD pair should be evaluated. If recovery failures are duplicated then the sample matrix is suspected as the problem and the data should be flagged and the failures discussed in the sample narrative.

12.7 **Duplicate and MS/MSD RPDs** – Five percent of all environmental samples should be analyzed in duplicate. A MS/MSD pair is also an acceptable duplicate analysis. If results are not acceptable, check for possible sample preparation problems and re-analyze if needed. Report the results with an appropriate data qualifier.

13 METHOD PERFORMANCE

13.1 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.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-ALL-Q-004, *Method Detection Limit Studies*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

14 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management*.

14.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

15 REFERENCES

15.1 USEPA, SW-846, Method 8082A, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography", February 2007.

15.2 USEPA, SW-846, Method 8000B, "Determinative Chromatographic Separations", December 1996.

15.3 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-053, *"Separatory Funnel Extraction.*

15.4 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-031, "*Extraction of Biological Samples for Organochlorine Pesticides/PCBs*".

15.5 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-040, "*Extraction of Wipes and Oil for PCB Analysis".*

15.6 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-041, "*Extraction of PCBs Using the Automated Soxhlet*".

15.7 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-032, "*Gel Permeation Chromatography*".

15.8 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-034 *Sulfuric Acid Cleanup"*.

15.9 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-035, "*Mercury Cleanup for the Removal of Sulfur from PCB Samples*".

15.10 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-036, "*Florisil Cleanup for PCBs*".

15.11 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-038, "*Silica Gel Cleanup of Organochlorine Pesticides and PCBs*".

15.12 Pace Analytical Services, Inc – Green Bay, SOP S-GB-O-039, "*Copper Cleanup for the Removal of Sulfur from PCB Samples*".

16 TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

16.1 Table A. Reporting Limits for PCBs.

Aroclor	CAS #	Water PRL (µg/L)	Soil PRL (µg/Kg)	Biota PRL (µg/Kg as is)
AR1016	12674-11-2	1.0	100	50
AR1221	11104-28-2	1.0	100	50
AR1232	11141-16-5	1.0	100	50
AR1242	53469-21-9	1.0	100	50
AR1248	12672-29-6	1.0	100	50
AR1254	11097-69-1	1.0	100	50
AR1260	11096-82-5	1.0	100	50
AR1262	37324-23-5	1.0	100	50
AR1268	11100-14-4	1.0	100	50

***Note: Aroclor 1262 and 1268 only analyzed upon client request**

16.2 Table B. Solvents.

Reagent	Purity	Manufacturer	Vendor	Catalog #
Hexane	NS Grade	Burdick & Jackson	MG Scientific	B&J-217-4
Acetone	Pesticide Grade	Burdick & Jackson	MG Scientific	B&J-010-4

16.3 Table C. Standard Stock Solutions.

Standard	Concentration	Manufacturer	Catalog #
Pesticide Surrogate Mix	200µg/mL each in Acetone	Restek Corporation or equivalent	32000
Aroclor 1016 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32006
Aroclor 1221 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32007
Aroclor 1232 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32008
Aroclor 1242 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32009
Aroclor 1248 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32010
Aroclor 1254 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32011
Aroclor 1260 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32012
Aroclor 1262 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32409
Aroclor 1268 Mix	1000µg/mL in Hexane	Restek Corporation or equivalent	32410
Aroclor 1016	1000µg/mL in Isooctane	Supelco or equivalent	4-8097
Aroclor 1260	1000µg/mL in Isooctane	Supelco or equivalent	4-4809

***Note: Aroclor 1262 and 1268 only analyzed upon client request**

16.4 Table D. Preparation of Analytical Standard Solutions.

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
TCMX/DCB Stock Solution	Pesticide Surrogate Mix	1000µL	20mL of Hexane	10µg/mL	1 year from date of preparation
AR1221 Stock Solution	Aroclor 1221 Mix	1000µL	10mL of Hexane	100µg/mL	1 year from date of preparation
AR1232 Stock Solution	Aroclor 1232 Mix	1000µL	10mL of Hexane	100µg/mL	1 year from date of preparation
AR1242 Stock Solution	Aroclor 1242 Mix	1000µL	10mL of Hexane	100µg/mL	1 year from date of preparation
AR1248 Stock Solution	Aroclor 1248 Mix	1000µL	10mL of Hexane	100µg/mL	1 year from date of preparation
AR1254 Stock Solution	Aroclor 1254 Mix	1000µL	10mL of Hexane	100µg/mL	1 year from date of preparation
AR1262 Stock Solution	Aroclor 1262 Mix	1000µL	10mL of Hexane	100µg/mL	1 year from date of preparation
AR1268 Stock Solution	Aroclor 1268 Mix	1000µL	10mL of Hexane	100µg/mL	1 year from date of preparation
AR1660 Stock Solution	Aroclor 1016 Mix Aroclor 1260 Mix	1000µL each	10mL of Hexane	100µg/mL each	1 year from date of preparation
AR1660 ICV Stock Solution	Aroclor 1016 Aroclor 1260	1000µL each	10mL of Hexane	100µg/mL each	1 year from date of preparation
AR1221-3 Calibration Standard	AR1221 Stock Solution TCMX/DCB Stock Solution	AR1221 500µL TCMX/DCB 500µL	100mL of Hexane	AR1221 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation
AR1232-3 Calibration Standard	AR1232 Stock Solution TCMX/DCB Stock Solution	AR1232 500µL TCMX/DCB 500µL	100mL of Hexane	AR1232 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation
AR1242-3 Calibration Standard	AR1242 Stock Solution TCMX/DCB Stock Solution	AR1242 500µL TCMX/DCB 500µL	100mL of Hexane	AR1242 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation
AR1248-3 Calibration Standard	AR1248 Stock Solution TCMX/DCB Stock Solution	AR1248 500µL TCMX/DCB 500µL	100mL of Hexane	AR1248 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation
AR1254-3 Calibration Standard	AR1254 Stock Solution TCMX/DCB Stock Solution	AR1254 500µL TCMX/DCB 500µL	100mL of Hexane	AR1254 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation
AR1262-3 Calibration Standard	AR1262 Stock Solution TCMX/DCB Stock Solution	AR1262 500µL TCMX/DCB 500µL	100mL of Hexane	AR1262 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation
AR1268-3 Calibration Standard	AR1268 Stock Solution TCMX/DCB Stock Solution	AR1268 500µL TCMX/DCB 500µL	100mL of Hexane	AR1268 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation
AR1660-1 Calibration Standard and PRLS	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 100µL TCMX/DCB 100µL	100mL of Hexane	AR1660 0.1µg/mL TCMX/DCB 0.01µg/mL	6 mo. from date of preparation
AR1660-2 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 300µL TCMX/DCB 200µL	100mL of Hexane	AR1660 0.3µg/mL TCMX/DCB 0.02µg/mL	6 mo. from date of preparation
AR1660-3 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 1000µL TCMX/DCB 1000µL	200mL of Hexane	AR1660 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation

***Note: Aroclor 1262 and 1268 only analyzed upon client request**

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
AR1660-4 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 800µL TCMX/DCB 1000µL	100mL of Hexane	AR1660 0.8µg/mL TCMX/DCB 0.10µg/mL	6 mo. from date of preparation
AR1660-5 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 1000µL TCMX/DCB 1500µL	100mL of Hexane	AR1660 1.0µg/mL TCMX/DCB 0.15µg/mL	6 mo. from date of preparation
AR1660-3 ICV Calibration Standard	AR1660 ICV Stock Solution TCMX/DCB Stock Solution	AR1660 500µL TCMX/DCB 500µL	100mL of Hexane	AR1660 0.5µg/mL TCMX/DCB 0.05µg/mL	6 mo. from date of preparation

17 REVISIONS

Revision Number	Reason for Change	Date
S-GB-O-047-REV.00	First Issue.	11Sept2008
S-GB-O-047-REV.01	Updated signature page Changed all references to TMX to TCMX Added a note to tables including Aloclor 1262 and 1268 stating they will only be analyzed upon request Section 10.1.1.1: Deleted Aloclor 1262 Section 10.2.3.1: Changed %RSD to %D for surrogate recoveries Section 10.2.5: Added that a low level standard is to be evaluated for each 5 point calibration that is analyzed	09Apr2010
S-GB-O-047-Rev.02	Section 10.1.2.5: Added requirement to verify DDT analogs to not co-elute with Arolcors of interest. Section 12.2 and 12.4: Included language that biota recovery limits should be broadened to 60-130% if laboratory charted limits are narrower than that.	13Sept2010

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STANDARD OPERATING PROCEDURE

DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS

REFERENCE METHODS: Methods SW-846 8270C / EPA 625

LOCAL SOP NUMBER:	S-GB-O-049-Rev.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-049-Rev.02, S-ALL-GB-O-001-Rev.00
SOP TEMPLATE NUMBER:	SOT-ALL-O-001-Rev.00

LOCAL APPROVAL

Nils Melberg, Laboratory General Manager — Date: 7/9/10

Kate Grams, Laboratory Quality Manager — Date: 7/9/10

Glen Coder, Department Manager — Date: 7/9/10

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Green Bay to determine the concentration of Semi-Volatile Organic Compounds (SVOCs) in environmental samples. The laboratory utilizes GC/MS and bases these documented procedures on those listed Methods SW-846 8270C / EPA 625. The Green Bay laboratory currently processes water samples by automated separatory funnel using Method SW846 3510C, soil samples by Microwave Extraction using Method SW846 3546 and biota samples by soxhlet extractor using Method SW846 3540C. The latest revision of Pace's SOPs S-GB-O-053 *Separatory Funnel Extraction of Water Samples for Semivolatile Analysis*, S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic hydrocarbon, Base/Neutral/Acids, and Total Petroleum Hydrocarbons on Solid Matrices*, and GB-O-033 *Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis* for these extraction techniques are available from the quality office.

2. SCOPE AND APPLICATION

2.1 This procedure may be used to determine concentrations of neutral, acidic, and basic semivolatile organic compounds in extracts prepared from many types of water samples, soil samples and wastes. Analytes must be soluble in dichloromethane and amenable to capillary gas chromatography. Specific compound classes include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols. A list of applicable compounds is shown herein. Pace Reporting Levels (PRLs) are also shown for water and soil samples. PRLs are subject to change based on current analytical system performance and actual sample matrices.

2.2 This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interference with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data

2.3 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of semi-volatile configured GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

2.4 This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

3. SUMMARY OF METHOD

Sample extracts are prepared for analysis by an appropriate sample preparation method. The semivolatile organic compounds are introduced into the gas chromatograph (GC) by injecting an aliquot of the sample extract. The GC conditions are programmed to separate the analytes. The GC effluent is directly introduced to a mass spectrometer (MS) for both identification and quantification of analytes. Analytes are identified by comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected major (quantitation) ion relative to an internal standard using a multi-point calibration curve.

4. INTERFERENCES

4.1 Interferences may be introduced into sample extracts by contaminants in solvents, reagents, glassware, and any other material that comes in contact with the sample or extract during extract preparation. These interferences must be closely monitored by analyzing Method Blank samples and taking corrective action as required.

4.2 Matrix interferences may result from materials co-extracted from some samples.

4.3 Significant phthalate contamination may result at any time if consistent quality control is not practiced. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

4.4 Contamination by carryover can occur when high concentration extracts are analyzed prior to low concentration extracts. The contamination may also cause degradation of labile analytes. Whenever carryover is suspected, the affected extracts should be re-analyzed. If significant degradation of the GC/MS systems is suspected, system performances samples should be analyzed and corrective action taken as needed.

4.5 The extent of matrix interference's will vary considerably from source to source. The laboratory utilizes the Gel Permeation Chromatography (GPC) clean-up procedure to clean up the sample extract following the latest revision of Pace's SOP S-GB-O-032 *Gel Permeation Chromatography*. The extraction personnel initially determine the need for GPC cleanup. The analyst can request that the sample extracts have GPC cleanup prior to the addition of Internal Standards. This is usually determined by visual inspection of the sample extract or by historical data. Tissue samples must be cleaned by GPC and florisil column procedures.

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.

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.

5.3 Equipment

Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones.

The GC pneumatic system uses gas under high pressure. These high pressures introduce the risk of injury due to flying objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of

equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

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 **Run Sequence Log** – A logbook that lists all injections and analyses performed on a particular piece of equipment regardless of the use of the data collected from each analysis.

6.3 **Toxicity Characteristic Leaching Procedure (TCLP)** – An extraction procedure used to determine if a sample is acceptable for upland disposal. The extraction procedure is meant to simulate the leaching of contaminants under the environmental conditions typically found in a landfill.

6.4 **Tune Period –** The period after the DFTPP instrument tune check within which analyses may be performed.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	One (1) 1-Liter Amber Glass Samples to be analyzed by EPA 625 must be checked for residual chlorine. If residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well.	None	4 ± 2°C	7 days
Soil/Solid (non-aqueous)	One (1) 8-oz Glass Jar	None	4 ± 2°C	14 days
Biota	-	None	≤ -10°C until extraction	1 year when frozen
TCLP	One (1) 1-Liter Amber Glass	None	4 ± 2°C	TCLP Leachates must be solvent extracted within 7 days of the completion of the tumbling process.
Extracts	2-5mL Glass vials, same as used for standard storage	None	≤-10°C	40 days

8. EQUIPMENT AND SUPPLIES

8.1 Instrumentation

Analytical Instrument/Peripherals	EPIC Pro Name	Serial Number
HP 5890 Series II GC	40MSS1	3336A57925
HP 5972 Mass Selective Detector	40MSS1	3501A02320
HP 7673 AutoSampler Tray	40MSS1	3526A39072
HP 7673 Injector	40MSS1	3009A20936
HP Controller	40MSS1	3526A02233
Alcatel 2005 Rough Pump	40MSS1	265402
HP 5890 Series II GC	40MSS5	3022A29174
HP 5972 Mass Selective Detector	40MSS5	3501A02209
HP 7673 AutoSampler Tray	40MSS5	3319A32067
HP 6890 Injector	40MSS5	DE64502673
HP Controller	40MSS5	3018A22346
Varian DS302 Rough Pump	40MSS5	244968
HP 5890E GC	40MSS6	3310A49571
HP 5972A Mass Selective Detector	40MSS6	3524A03107
HP 18596A AutoSampler Tray	40MSS6	2920A10670
HP 6890 Injector	40MSS6	US0000692
HP 7673 Controller	40MSS6	3113A25880
Edwards E2M2 Rough Pump	40MSS6	53747

8.2 Chromatography Supplies

Item	Vendor	Model / ID	Catalog #	Description
Analytical Column	Restek	XTI-5 w/ Integraguard	12223-124	30 m, 0.25 mm ID, 0.25 df
Analytical Column	Restek	Rxi-1 MS		20m, 0.18 mm ID, 0.18 df
Fluorocarbon O-rings	Restek		20377	
Vespel/Graphite Ferrules	Restek		20229	1/16” x 0.4 mm ID
Gooseneck Splitless Liner	Restek		20800	4 mm x 6.5 x 78.5 for Aligent GCs
Guard Column	Restek		20231	0.5 MID
Ferrules	Fisher Scientific		5-5	

8.3 Glassware

Glassware	Description	Vendor / Item # / Description
Volumetric Flasks	10mL, 25mL, 50mL	Class A
Glass Storage Vials	5mL, 10mL, 12mL, with Teflon-lined screw caps	MG Scientific / T102-3-INV, T102-1-INV V138-19, B510-1
Glass Autosampler Vials	2.0 mL with Teflon-lined crimp caps	MG Scientific / V300-3 / V300-20N

8.4 General Supplies

Glassware	Description	Vendor/ Item # / Description
Gas tight syringes	10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL, as needed, Hamilton or equivalent.	Fisher Scientific / Various
Pipettes	Borosilicate Glass 9” Pipette	MG Scientific / D200-9

9. REAGENTS AND STANDARDS

9.1 Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Dichloromethane		Pesticide Grade or equivalent

9.2 Analytical Standards

9.2.1 Definitions

9.2.1.1 Standards are required for mass spectrometer tuning, initial calibration, calibration verification standards, second source verification, internal standards, surrogates, and for preparing LCS, MS, and MSD samples. Table 9.1 describes the standards used.

Table 9.1 Standard Definitions and vendors

Standard	Description	Comments
Tune Standard	Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, pentachlorophenol, and benzidine solution in dichloromethane used to verify ion response ratios and system inertness prior to analysis.	Must inject no more than 50 ng on column
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the accuracy of the calibration curve.	
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	1,4 Dichlorobenzene-d4 Naphthalene-d_8 Acenaphthene-d_{10} Phenanthrene-d_{10} Chrysene-d_{12} Perylene-d_{12}
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to extraction.	Nitrobenzene-d_5 2-Fluorobiphenyl Terphenyl-d_{14} Phenol-d_6 2-Fluorophenol 2,4,6-Tribromophenol
Spiking Standard	This solution contains 70 target analytes and should not be prepared from the same standards as the calibration standards.	

9.2.2 Storage Conditions

Table 9.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	▪ Concentrated reference solution purchased directly from approved vendor	▪ Manufacturer's recommended expiration date for unopened ampulated standards. ▪ All other stock standards must be replaced 1 year after ampule is opened or on expiration date, whichever is sooner.	▪ 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	▪ 1 year from preparation or the expiration date listed for the stock source, whichever is sooner. ▪ Working solutions must be checked frequently and replaced if degradation or evaporation is suspected.	▪ Store in amber vials with Teflon lined caps ▪ Manufacturer's recommended storage conditions for stock source solution. ▪ If stock source conditions conflict, store according to method requirements.

9.2.3 Standard Sources

Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat materials, recipes for preparing dilutions and working standards, and concentrations for all compounds are presented in Section 9.2.4. All intermediate standards are prepared using dichloromethane and stored in glass vials with Teflon lined caps or Mininert valves or as recommended by the standard manufacturer.

9.2.4 Preparation Procedures

9.2.4.1 Internal Standard Stock solution: Restek brand Internal Standard Mix, 4000ug/mL, catalog # 31006 (contains six internal standards: acenaphthene-d10; chrysene-d12; 1,4-dichlorobenzene-d4; naphthalene-d8; perylene-d12; and phenanthrene-d10), or equivalent. Add 1uL of internal standard solution to 100 uL of every standard, sample, and QC sample injected.

9.2.4.2 Surrogate Standard stock solutions: Restek brand Base Neutral surrogate mix, 5000ug/mL, catalog # 31082 (contains four surrogate standards: 2-fluorobiphenyl; nitrobenzene-d5; p-terphenyl-d14; 1,2-Dichlorobenzene-d4 (advisory). Restek brand Acid surrogate mix, 7500ug/mL, catalog # 31083 (contains four surrogate standards 2-fluorophenol; phenol-d6; 2,4,6-tribromophenol; 2-chlorophenol-d4(advisory)), or equivalent.

9.2.4.3 Surrogate Standard working solution (for extractions): dilute 5.0mL of both the Restek Base Neutral stock surrogate solution (# 31082) and the Restek Acid stock surrogate solution (#31083) to 50mL with Dichloromethane, or equivalent. This gives a final concentration of 500ug/mL per Base Neutral surrogate compound and 750ug/mL per Acid surrogate compound. The extraction analyst spikes each water and soil sample with 100uL of this working solution.

9.2.4.4 **DFTPP tuning solution**: dilute 500uL of Supelco DFTPP stock standard (catalog # 47548-U; 1000ug/mL) to a total volume of 10mL with methylene chloride for a final concentration of 50ug/mL, or equivalent. The stock standard also contains 4,4'-DDT, benzidine, and pentachlorophenol for assessing column degradation. Information for the standards preparation and expiration dates are affixed to the outside of the vial, and is easily accessible through the standard preparation logbook. The standard material will be kept in a freezer at -10°C.

9.2.4.5 **Initial Calibration curve standards**: the following three stock standards, or equivalent, are used to prepare the initial calibration curve:

9.2.4.5.1 8270 Custom Mix 1, Supelco, 400ug/mL, Quote# 20447738

9.2.4.5.2 n-Nitrosodiphenylamine, Supelco, 5000ug/mL, catalog # 46702-U

9.2.4.5.3 1,4-Dioxane, Restek, 2000ug/mL, catalog # 30287

9.2.4.5.4 2,3,4,6-Tetrachlorophenol, Accustandard, 2000ug/mL, catalog # A-029S-D-10X

9.2.4.5.5 1,2,4,5-Tetrachlorobenzene, Absolute Standards, 1000ug/mL, catalog # 70274.

9.2.4.5.6 **Initial Calibration Intermediate Standard**: Dilute 2.5mL of 400ug/mL Supelco 8270 Custom Mix 1, 200uL of the 5000ug/mL n-Nitrosodiphenylamine solution, 500uL of the 2000ug/mL 1,4-Dioxane solution, 500 uL of the 2000 ug/mL 2,3,4,6-Tetrachlorophenol solution, and 1000 uL of 1000ug/mL 1,2,4,5-Tetrachlorobenzene to 5.0mL with dichloromethane, or equivalent. The resulting intermediate standard has a concentration of 200mg/L for each compound.

9.2.4.5.7 **Initial Calibration Working Standards**: The following working calibration standards are prepared by using the intermediate mix listed above (9.2.4.5.6):

Standard Concentration*	Amount of Intermediate Standard added	Amount of Stock internal standard added	Dilution Solvent Used	Final Volume
5ppm	25uL	10uL	Dichloromethane	1010uL
10ppm	50uL	10uL	Dichloromethane	1010uL
25ppm	125uL	10uL	Dichloromethane	1010uL
50ppm	250uL	10uL	Dichloromethane	1010uL
80ppm	400uL	10uL	Dichloromethane	1010uL
100ppm	500uL	10uL	Dichloromethane	1010uL
120ppm	600uL	10uL	Dichloromethane	1010uL

9.2.4.6 Initial Calibration Verification stock standards (second-source):

9.2.4.6.1 Restek, custom 8270,catalog # 52939, 200ug/mL per compound.

9.2.4.6.2 Supelco 1,4-Dioxane, catalog #48367, 2000ug/mL.

9.2.4.6.3 Restek 2,3,4,6-tetrachlorophenol, catalog #560028, 2000ug/mL, or equivalent

9.2.4.6.4 Supelco, 1,2,4,5-Tetrachlorobenzene, catalog # 40177, 1000ug/mL.

9.2.4.6.5 Initial Calibration Verification working standard (second source): Dilute 250uL of the custom 8270 second source standard #52939, 25ul of 2,3,4,6-Tetrachlorophenol #560028, 25uL of 1,4-Dioxane #48367, 50uL of 1,2,4,5-Tetrachlorobenzene #40177, and 10uL of the stock internal standard solution (9.2.4.1) to 1mL with dichloromethane, or equivalent. This gives a final concentration of 50ppm.

9.2.4.7 LCS/MS Standard working solution: Supelco 70 Component Custom LCS Mix catalog # 861389-U, 200ug/mL. Supelco n-Nitrosodiphenylamine, catalog # 46702-U, 5000ug/mL, or equivalent. The extraction analyst spikes each LCS/LCSD and matrix spike sample with 250uL of the LCS mix and 10uL of the n-NDPA solution. This produces a concentration of 50ug/mL.

9.2.4.8 Other calibrations: Other compounds are analyzed per client requests. Curves are prepared at levels similar to those of the standards above. The calibration standards and the second source standards are as follows: Calibration Standards, Benzidine, Supelco, catalog # 40005, 5000ug/mL; EPA CLP SOW OLM4 mix, Supelco. catalog # 47514-U, 2000ug/mL (contains Acetophenone, Atrazine, Benzaldehyde, Biphenyl and Caprolactam). Second Source standards, Benzidine, Restek, catalog# 31441, 1000ug/mL; EPA CLP SOW OLM4 mix, Absolute Standards, catalog #19253, 2000ug/mL, or equivalent. Minnesota Phenols Samples required Calibration Standard Supelco, catalog #20858688, 500 ppm for initial calibration and Accustandard, catalog # 5-8126-R3, 500 ppm for second source standard.

9.2.5 Store at -10°C or less in amber Teflon-sealed containers. The solutions should be checked frequently for stability.

9.3 Traceability of Calibration Standards – The calibration standards purchased from vendors have been manufactured according to the following guidelines

9.3.1 Identity of neat material verified by GC/MS

9.3.2 Purity of neat material determined by GC/FID or GC/ECD. Correction for impurities is made when purity is less than 97%. Standards are prepared gravimetrically to a precision of 0.5%. All weights are traceable to NIST.

9.3.3 Analyte concentration verified by capillary gas chromatography. Standards tested for stability and homogeneity.

9.3.4 Standards are expiration dated.

9.4 Standard Labeling – All working calibration standards will have a label attached to the bottle identifying the following:

9.4.1 Name of Solution

9.4.2 PASI, Inc. Standard ID Number

9.4.3 Preparation Date

9.4.4 Preparer's initials

9.4.5 Concentration

9.4.6 Expiration Date

9.5 The standards preparation logbook will contain date prepared, analyst, PASI ID Number, analyte description, analyte initial and final concentrations, aliquots used, and solvents for dilution.

10 CALIBRATION

10.1 Tune Verification

The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. If the current tune status does not meet the ion ratio criteria in the method (see section 11.2.1), take corrective actions as outlined in section 11.2.1. Otherwise, follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be re-verified after the tuning procedures.

Refer to section **11.2 Procedures** for details on the analysis and evaluation of this standard.

10.2 Initial Calibration (ICAL)

10.2.1 Analysis of Standards

10.2.1.1 An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to table 10.1 for compound concentrations.

10.2.1.2 An analyte must be present and calibration curve in control in order to be reported on the target analyte list. Analytes identified by mass spectral match but not present and in control in the calibration table may be reported as Tentatively Identified Compounds (TICs). Guidelines for identification are listed in Section 11.6.4. Results for these TICs should be reported only on a present/absent basis. However, quantitative results may be reported provided they are qualified as estimated values.

Table 10.1 – Calibration standard compound concentrations*

Analyte	CAS Number	PQL water (ug/L)	PQL soil (ug/kg)	PQL Biota (ug/kg)	Std 1 ug/ml	Std 2 ug/ml	Std 3 ug/ml	Std 4 ug/ml	Std 5 ug/ml	Std 6 ug/ml	Std 7 ug/ml
Acenaphthene	83-32-9	5.0	167	330	5.0	10	25	50	80	100	120
Acenaphthylene	208-96-8	5.0	167	330	5.0	10	25	50	80	100	120
Aniline	62.53.3	5.0	167	N/A	5.0	10	25	50	80	100	120
Anthracene	120.12.7	5.0	167	330	5.0	10	25	50	80	100	120
Benz(a)anthracene	56-55-3	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(a)pyrene	50-32.8	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(b)fluoranthene	205-99-2	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(g,h,i)perylene	191-24-2	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(k)fluoranthene	207-08-9	5.0	167	330	5.0	10	25	50	80	100	120
Benzoic acid	65-85-0	10	167	N/A	5.0	10	25	50	80	100	120
Benzyl alcohol	100.51-6	10	167	N/A	5.0	10	25	50	80	100	120
4-Bromophenylphenyl ether	101.55.3	5.0	167	330	5.0	10	25	50	80	100	120
Butylbenzylphthalate	85-68-7	5.0	167	330	5.0	10	25	50	80	100	120
Carbazole	86-74-8	5.0	167	330	5.0	10	25	50	80	100	120
4-Chloro-3-methylphenol	59-50-7	5.0	167	330	5.0	10	25	50	80	100	120
4-Chloroaniline	106-47-8	10	333	330	5.0	10	25	50	80	100	120
bis(2-Chloroethoxy)methane	111-91-1	5.0	167	330	5.0	10	25	50	80	100	120
Bis(2-Chloroethyl) ether	114-44-4	5.0	167	330	5.0	10	25	50	80	100	120
bis(2-Chloroisopropyl) ether	108-60-1	5.0	167	330	5.0	10	25	50	80	100	120
2-Chloronaphthalene	91-58-7	5.0	167	330	5.0	10	25	50	80	100	120
2-Chlorophenol	95-57-8	5.0	167	330	5.0	10	25	50	80	100	120
4-Chlorophenylphenyl ether	7005-72-3	5.0	167	330	5.0	10	25	50	80	100	120
1,2-Diphenylhydrazine	122-66-7	5.0	167	N/A	5.0	10	25	50	80	100	120

Chrysene	218-01-9	5.0	167	330	5.0	10	25	50	80	100	120
Dibenz(a,h)anthracene	53-70-3	5.0	167	330	5.0	10	25	50	80	100	120
Dibenzofuran	132-64-9	5.0	167	330	5.0	10	25	50	80	100	120
1,2-Dichlorobenzene	95-50-1	5.0	167	330	5.0	10	25	50	80	100	120
1,3-Dichlorobenzene	541-73-1	5.0	167	330	5.0	10	25	50	80	100	120
1,4-Dichlorobenzene	106-46-7	5.0	167	330	5.0	10	25	50	80	100	120
3,3'-Dichlorobenzidine	91-94-1	10	167	330	5.0	10	25	50	80	100	120
2,4-Dichlorophenol	120-83-2	5.0	167	330	5.0	10	25	50	80	100	120
Diethylphthalate	84-66-2	5.0	167	330	5.0	10	25	50	80	100	120
2,4-Dimethylphenol	105-67-9	5.0	167	330	5.0	10	25	50	80	100	120
Dimethylphthalate	131-11-3	5.0	167	330	5.0	10	25	50	80	100	120
Di-n-butylphthalate	84-74-2	5.0	167	330	5.0	10	25	50	80	100	120
4,6-Dinitro-2-methylphenol	534-52-1	5.0	333	670	5.0	10	25	50	80	100	120
2,4-Dinitrophenol	51-28-5	10	333	670	5.0	10	25	50	80	100	120
2,4-Dinitrotoluene	121-14-2	5.0	167	330	5.0	10	25	50	80	100	120
2,6-Dinitrotoluene	606-20-2	5.0	167	330	5.0	10	25	50	80	100	120
Di-n-octylphthalate	117-84-0	5.0	167	330	5.0	10	25	50	80	100	120
bis(2-Ethylhexyl)phthalate	117-81-7	5.0	167	330	5.0	10	25	50	80	100	120
Fluoranthene	206-44-0	5.0	167	330	5.0	10	25	50	80	100	120
Fluorene	86-73-1	5.0	167	330	5.0	10	25	50	80	100	120
Hexachloro-1,3-butadiene	87-68-3	10	333	330	5.0	10	25	50	80	100	120
Hexachlorobenzene	118-74-1	5.0	167	330	5.0	10	25	50	80	100	120
Hexachlorocyclopentadiene	77-47-4	5.0	167	330	5.0	10	25	50	80	100	120
Hexachloroethane	57-72-1	5.0	167	330	5.0	10	25	50	80	100	120
Indeno(1,2,3-cd)pyrene	193-39-5	5.0	167	330	5.0	10	25	50	80	100	120
Isophorone	78-59-1	5.0	167	330	5.0	10	25	50	80	100	120
2-Methylnaphthalene	91-57-6	5.0	167	330	5.0	10	25	50	80	100	120
2-Methylphenol	95-48-7	10	333	330	5.0	10	25	50	80	100	120
3&4-Methylphenol	106-44-5	5.0	167	330	5.0	10	25	50	80	100	120
Naphthalene	91-20-3	5.0	167	330	5.0	10	25	50	80	100	120
2-Nitroaniline	88-74-4	5.0	167	330	5.0	10	25	50	80	100	120
3-Nitroaniline	99-09-2	5.0	167	670	5.0	10	25	50	80	100	120
4-Nitroaniline	100-01-6	10	333	670	5.0	10	25	50	80	100	120
Nitrobenzene	98-95-3	5.0	167	330	5.0	10	25	50	80	100	120
2-Nitrophenol	88-75-5	5.0	167	330	5.0	10	25	50	80	100	120
4-Nitrophenol	100-02-7	10	333	670	5.0	10	25	50	80	100	120
N-Nitrosodimethylamine	62-75-9	5.0	333	330	5.0	10	25	50	80	100	120
N-Nitroso-di-n-propylamine	621-64-7	5.0	167	330	5.0	10	25	50	80	100	120
N-Nitrosodiphenylamine	86-30-6	10	333	330	5.0	10	25	50	80	100	120
Pentachlorophenol	87-86-5	5.0	167	670	5.0	10	25	50	80	100	120
Phenanthrene	85-01-8	5.0	167	330	5.0	10	25	50	80	100	120
Phenol	108-95-2	5.0	167	330	5.0	10	25	50	80	100	120
Pyrene	129-00-0	5.0	167	330	5.0	10	25	50	80	100	120
1,2,4-Trichlorobenzene	120-82-1	5.0	167	330	5.0	10	25	50	80	100	120
2,4,5-Trichlorophenol	95-95-4	5.0	167	670	5.0	10	25	50	80	100	120
2,4,6-Trichlorophenol	88-06-2	5.0	167	330	5.0	10	25	50	80	100	120
1,4-Dioxane	123-91-1	10	167	N/A	5.0	10	25	50	80	100	120
1,2,4,5-Tetrachlorobenzene	95-94-3	5.0	167	N/A	5.0	10	25	50	80	100	120
2,3,4,6-Tetrachlorophenol	58-90-2	10	167	N/A	5.0	10	25	50	80	100	120
Acetophenone	98-86-2	10	333	N/A	5.0	10	25	50	80	100	N/A
Atrazine	1912-24-9	10	333	N/A	5.0	10	25	50	80	100	N/A
Benzaldehyde	100-52-7	10	333	N/A	5.0	10	25	50	80	100	N/A
Benzidine	92-87-5	50	1670	N/A	5.0	10	25	50	80	100	N/A
Caprolactam	105-60-2	10	333	N/A	5.0	10	25	50	80	100	N/A
Biphenyl	92-52-4	10	333	N/A	5.0	10	25	50	80	100	N/A

*Additional compounds may be analyzed per client request. MDL and PQL will be determined at time of analysis

10.3 Calibration Response Factors

10.3.1 Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

10.3.2 To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:
A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L).
C_x = Concentration of the compound being measured (µg/L).

Most, if not all modern chromatography data systems are capable of calculating this factor and using it to quantify analyte concentrations. The 8270C method has minimum requirements that these response factors must meet in order to be considered valid. The method uses a subset of the target analyte list to evaluate the performance of the system. These compounds are referred to as the System Performance Check Compounds or the SPCCs. The SPCCs serve as an indicator of instrument sensitivity and, by meeting a minimum value, ensure that the laboratory has adequate sensitivity to analyze and reliably report data for environmental samples.

10.4 Calibration Curve Fit

10.4.1 The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the "goodness of fit".

10.4.1.1 Average Response Factor (ARF): The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the

interception of the y axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the ARF (see Table 10.2).

The % RSD is calculated as follows: %RSD = SD*100 / ARF

Where: SD = Standard deviation of the averaged RFs for a given compound

The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The ***Calibration Check Compounds (CCCs)*** are a subset of the target analyte list that must meet specific criteria (see Table 10.2) for the calibration to be acceptable. For the CCCs, the %RSD for each is compared to the method criteria. If that of any CCC exceeds the criteria, the system needs to be inspected for potential sources of errors and recalibrated.

10.4.1.2 Linear Regression: The linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of y=ax+b where "a" is the slope of the line and "b" is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y axis at the origin. However, because the linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. A calculation of the correlation coefficient "r" is used to determine the acceptability of a linear regressed curve (see Table 10.2).

10.4.1.3 Non-linear Regression: The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

A calculation of the coefficient of determination (COD) is used to determine the acceptability of a non-linear regressed curve (see Table 10.2). Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious problem is discovered and documented. The point must be dropped in its entirety or reanalyzed. Re-analysis should be within the same 12-hour time window and must occur within 8 hours of the original analysis.

10.5 Calibration Verification

10.5.1 Second Source Verification (SCV)

In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the "fit" of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

Therefore, to assess the accuracy relative to the purity of the standards, a single 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. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$\%\ \text{Difference} = [\text{Result}_{SCV} - \text{TrueValue}_{SCV}] / \text{TrueValue}_{SCV} * 100$

See Section 9.2.4.6 for details on the preparation of this standard. See Table 10.2 for control criteria

10.5.2 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 at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished 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 change calculated. The percent change must meet the method specified criteria for the analysis to proceed for an additional 12 hours.

The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$\%\ \text{Difference}: [RF_{CCV} - \text{AvgRF}_{CAL}] / \text{AvgRF}_{CALl} \times 100$

$\%\ \text{Drift}: [\text{Result}_{CCV} - \text{TrueValue}_{CCV}] / \text{TrueValue}_{CCV} \times 100$

Table 10.2 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor	%RSD ≤ 15%	If not met, try linear regression fit
	Linear Regression	r ≥ 0.99	If not met, try non-linear regression fit
	Non-linear Regression	COD ≥ 0.99	If not met, remake standards, or perform instrument maintenance and recalibrate
System Performance Check Compounds (SPCCs)	N-Nitroso-di-n-propylamine Hexachlorocyclopentadiene 2,4-Dinitrophenol 4-Nitrophenol	Avg RF ≥ 0.05 Avg RF ≥ 0.05 Avg RF ≥ 0.05 Avg RF ≥ 0.05	Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, poor purging efficiency, and active sites in the column or chromatographic system. Additional client specific requirements for the analysis of contract samples requires that all PAH's and surrogate compounds also be considered SPCCs and must meet the minimum RRF criterion of 0.05.
Calibration Check Compounds (CCCs)	Acenaphthene 1,4-Dichlorobenzene Hexachlorobutadiene N-Nitrosodiphenylamine Di-n-octylphthalate Fluoranthene Benzo[a]pyrene 4-Chloro-3-methylphenol 2,4-Dichlorophenol 2-Nitrophenol Phenol Pentachlorophenol 2,4,6-Trichlorphenol	%RSD < 30%	%RSD for the calibration check compounds (CCC's) must be ≤30% regardless of curve fit used. If the CCCs are not included on a list of analytes for a project, and therefore not included in the calibration standards, then all compounds of interest must meet a ≤15% RSD criterion. Additional client specific requirements for the analysis of contract samples requires that all PAH's and surrogate compounds also be considered CCC's and must meet the maximum RSD criterion of 30%.
Second Source Verification Standard	Immediately after each initial calibration	% Diff ±30%	Acceptance criteria are ±30% for all analytes, with allowances for 5% of compounds @ ±40%. See current revision of S-ALL-Q-025. Additional client specific requirements for the analysis of contract samples requires that all compounds must be within ±20%
Continuing Calibration Verification	Prior to the analysis of any samples and every 12 hours thereafter		If the requirements for continuing calibration are not met, these corrective actions must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back.
	SPCCs	Must meet response criteria listed above	
	Internal Standard RT	RT ± 30 sec	Use midpoint calibration standard as reference
	Internal Standard Response	50 – 200%	Use midpoint calibration standard as reference
	CCCs	RF ± 20% Diff.	Use for Avg RF calibration curves
		Result ± 20% Drift	Use for linear and non-linear calibration curves Additional client specific requirements for the analysis of contract samples requires that all PAH's and surrogate compounds also be considered CCCs and must meet the 20% CCV criterion.
	Non-CCC Targets	RF ± 50% Diff. Result ±50% Drift	Some programs may require control over non-CCC target analytes. In the absence of specified criteria, use those listed

10.6 Calibration Corrective Actions

10.6.1 Calibration Linearity Problems

10.6.1.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.

10.6.1.2 Perform another initial calibration.

10.6.1.3 No data can be reported.

10.6.1.4 Generate Non-Conformance Memo.

10.6.2 Secondary Verification Problems

10.6.2.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.

10.6.2.2 Perform another initial calibration.

10.6.2.3 No data can be reported.

10.6.2.4 Generate Non-Conformance Memo

10.6.3 Continuing Verification Problems

10.6.3.1 Reanalyze the original CCV standard to determine instrument consistency.

10.6.3.2 Prepare and analyze a new CCV standard to determine preparation consistency / standard integrity.

10.6.3.3 Document instrument maintenance.

10.6.3.4 Reanalyze CCV standard to determine if maintenance was effective in restoring performance.

10.6.3.5 Complete recalibration of instrument.

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 injection log with a thorough explanation for the deviation from policy.

Exceptions: If calibration verification is above the upper control limit, samples non-detected for those analytes may be reported without reanalysis.

10.7 General Comment: When constructing a linear initial calibration curve, the analyst can drop curve points as follows:

- The lowest curve point can be dropped as long as there is a standard that can meet the necessary reporting limits of the associated samples (or the reporting limits would have to be raised accordingly).
- The highest curve point, or points, can be dropped but this decreases the upper calibration range, thereby limiting the analyst to reporting data within this new calibration range (this may cause more dilutions).
- Mid point injections in the curve can be removed if it can be proven that it was a bad injection, instrument failure, etc. The same curve point is required to be reanalyzed and incorporated in the curve prior to sample analysis. A supervisor or the Quality Manager is required to approve any such instance

11 PROCEDURES

11.1 GC/MS System Preparation

11.1.1 Operating Parameters

Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration.

Table 11.1 –Instruments and Operating Parameters

GC/MS Instrument 40MSS1	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 40°C	Acquisition mode: SCAN
Temp Program: hold 1.0 min at 40°C, ramp at 18°C/min to 100°C, then ramp at 15°C/min to 290°C, hold 5.95min, then ramp at 40°C/min to 320°C and hold for 1 min	Mass Range: 35-500
Final Temp: 320°C	
Transfer Line Temp: 300°C	
Column: Restek XTI-5 (30m; 0.25mm ID and 0.25um film thickness)w/Integraguard	
Purge Flow: 40mL/min	

GC/MS Instrument 40MSS5 – Screen Instrument only	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 75°C	Acquisition mode: SCAN
Temp Program: hold 0.3 min at 75°C, ramp at 34°C/min to 320°C, then ramp at 40°C/min to 340°C, hold 1.20min	Mass Range: 35-500
Final Temp: 340°C	
Transfer Line Temp: 300°C	
Column: Restek XTI-5 (30m; 0.25mm ID and 0.25um film thickness)w/Integraguard	
Purge Flow: 40mL/min	

GC/MS Instrument 40MSS6 used for Minnesota Phenols Samples	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 5°C	Acquisition mode: SCAN
Temp Program 50°C, ramp at 18°C/min to 150°C, then ramp at 3°C/min to 167°C , then ramp at 40°C/min to 320°C and hold for 2.5 min	Mass Range: 35-500
Final Temp: 320°C	
Transfer Line Temp: 300°C	
Column: Restek Rxi-1MS (20m; 0.18um ID, 0.18 df)	
Split Flow: 100mL/min	

GC/MS Instrument 40MSS6 used for Biota Samples	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 40°C	Acquisition mode: SCAN
Temp Program: hold 1.0 min at 40°C, ramp at 18°C/min to 100°C, then ramp at 15°C/min to 290°C, hold 5.95min, then ramp at 40°C/min to 320°C and hold for 1 min	Mass Range: 35-500
Final Temp: 320°C	
Transfer Line Temp: 300°C	
Column: Restek XTI-5 (30m; 0.25mm ID and 0.25um film thickness)w/Integraguard	
Purge Flow: 40mL/min	

11.2 Tune Verification

At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified. This is done by analyzing a standard containing DFTPP (refer to table 9.3). The tune verification standard can be combined with the CCV standard provided that the amount of DFTPP introduced into the system meets the criteria in Table 11.2. For semi-volatile analysis, the system must also be verified for inertness. This is done simultaneously by the inclusion of DDT, benzidine and pentachlorophenol. DDT is used to verify breakdown conditions; benzidine and pentachlorophenol are used to check for tailing due to system activity.

11.2.1 Ion Ratio Verification

After the analysis of this standard, the mass spectrum of DFTPP must be evaluated against the following criteria.

Table 11.2

Mass (m/z)	Ion Abundance criteria
51	30.0-80.0% of m/z 198
68	<2.0% of m/z 69
69	Present
70	<2.0% of m/z 69
127	25.0-75.0% of m/z 198
197	<1.0% of m/z 198
198	Base peak, 100% relative abundance
199	5.0-9.0% of m/z 198
275	10.0-30.0% of m/z 198
365	>0.75% of m/z 198
441	Present, but less than m/z 443
442	40.0-110.0% of m/z 198
443	15.0-24.0% of m/z 442

To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for DFTPP. If the software has a program or macro for automatically selecting the spectra and evaluating the response ratios, use this option. Otherwise, the spectra must be obtained in one the following manners, in the listed order.

1. **Take one scan for DFTPP; or**
2. **Using an average of three scans, centered on the apex of the peak; or,**
3. **Using an average of all scans across the width of the peak, taken at half height; or,**

4. Using an average of all scans taken across the width of the peak from baseline to baseline.

A background scan taken immediately before but not including the peak must be subtracted.

Once obtained, evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, then analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date / time for the DFTPP tune verification. After that, the tune must be verified again to establish a new analytical window. The same Ion Abundance Criteria used for the DFTPP tune coupled with the initial calibration must be used for all subsequent analyses associated with that initial calibration.

If the ratios do not meet the criteria, refer to the following corrective actions to address the problem. Any changes made to the system must be followed with the reanalysis of a tune verification standard. Any maintenance performed on the physical mass spec components requires recalibration. "Autotunes" may be performed as long as the following CCV meets all criteria for response, retention time and sensitivity.

11.2.2 Tailing Factor Verification

Benzidine and Pentachlorophenol should be present at their normal responses, and peak tailing should not be to an excess.

11.2.3 Breakdown Verification

The GC/MS system must be sufficiently inert such that DDT will not breakdown excessively while in the injection port. The inertness is assessed by calculating the percent breakdown of DDT into the products DDD and DDE. The calculation is performed as follows:

% DDT Breakdown = (Sum of responses for DDD and DDE) / (Sum of responses for DDT, DDD and DDE) *100

The % breakdown must not exceed 20% unless all required compounds are within 20% on the CCV. If the breakdown of DDT exceeds this amount, samples may be analyzed if all requested analytes are within +/- 20% in the CCV (documented on an out-of-control form), otherwise corrective action must be taken prior to analysis of samples. The breakdown must be verified by the analysis of another breakdown standard after corrective action is taken. Follow the following steps to return the system to an acceptable operating condition.

- Perform front-end maintenance on the GCMS System.
- Begin the run again by re-analyzing the DFTPP tune solution.

11.3 Calibration Verification

After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. If it has already been determined that the instrument needs to be recalibrated, follow the procedures listed in section 10.2.1 (Analysis of Standards). Otherwise, analyze a Continuing Calibration Verification Standard to determine the current calibration status.

If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed section 10.6.3 (Continuing Verification Problems). If the tune verification has been combined with the CCV, the 12 hour analysis window begins from the analysis date / time of the CCV.

Note: In situations where the instrument will run unattended (i.e. overnight), the analyst may load sequential (back-to-back) CCVs in anticipation of that the first in the series may fail due to carry over from a previous sample. If so, both CCVs must be evaluated according to the protocol set forth in the Quality Assurance Manual within Section 6 – Equipment and Measurement Traceability.

11.4 Sample Preparation

11.4.1 Samples

11.4.1.1 Water Samples

Aqueous samples are prepared according to EPA Method 3510C. These procedures are contained in a separate standard operating procedure. Refer to S-GB-O-053 *Separatory Funnel Extraction of Water Samples for Semivolatile Analysis* for details on the preparation of aqueous samples.

Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10uL of the internal standard solution (9.2.4.1).

11.4.1.2 Soil Samples

Solid samples are prepared according to EPA Method 3546. These procedures are contained in a separate standard operating procedure. Refer to S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons and Base/Neutral/Acids in Solid Matrices* for details on the preparation of soil or solid samples.

Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10uL of the internal standard solution (9.2.4.1).

11.4.1.3 Biota Samples

Biota samples are prepared according to EPA Method 3540C. These procedures are contained in a separate standard operating procedure. Refer to S-GB-O-033 *Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis* for details on the preparation of biota samples.

Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 5uL of the internal standard solution (9.2.4.1).

11.4.2 Dilutions

Dilutions on sample extracts must be prepared in a volumetric fashion. Sample aliquots should be taken in volumetric syringes and brought to volume by the addition of solvent via an appropriate syringe. In the event a dilution is made to bring a target analyte into calibration range, the analyst should make a dilution such that the target analyte is roughly the equivalent of the mid calibration point whenever possible.

If dilutions are made on extracts that already contain internal standards, a proportional aliquot of internal standard solution must be added to the diluted extract based on the volume of diluent used.

11.5 Sample Analysis

11.5.1 GC/MS System Preparation

11.5.1.1 Operating Parameters – Set up the instrument parameters shown in Table 11.1.

11.5.1.2 System Tuning and GC Performance Checks – Analyze the Tuning Solution and tune the mass spectrometer to meet the criteria shown in Section 11.2. Verify acceptable GC system performance as described in Section 11.2. Print out a tune report.

11.5.2 Batch Sequence – Generate a sequence to run a batch of samples.

Initial Calibration – The typical batch for initial calibration should include:

Tune Standard
Calibration Level 1
Calibration Level 2
Calibration Level 3
Calibration Level 4
Calibration Level 5
Calibration and System Performance Solution

Sample Analysis – The typical batch for sample analysis should include the following. Preparation of LCS, MS, MSD, and Duplicate sample extracts is described in the appropriate sample preparation SOP.

Tune Standard
Calibration and System Performance Solution
Instrument Blank
Method Blank
Laboratory Control Sample
Laboratory Control Sample Duplicate
20 samples
Matrix Spike
Matrix Spike Duplicate

11.5.3 Autosampler – Load the autosampler with standards and samples for the batch created above.

11.5.4 Analyze Samples – Analyze all standards, quality control samples, and environmental samples.

11.5.5 Process all runs with Target software

11.5.6 View sample chromatograms and verify analyte identifications (Section 11.6).

11.5.7 Post data to EPIC Pro.

11.6 Data Reduction

11.6.1 Qualitative Analysis

11.6.1.1 Retention Time Comparison: The relative retention time (RRT) of the sample component must be within ±0.06 RRT units of the component in the calibration verification standard. Extracted Ion Current Plots (EICPs) may be used to provide a more reliable assignment of RT in the presence of co eluting components.

11.6.1.2 Mass Spectrum Comparison: The characteristic ions from the reference mass spectrum are defined as 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.

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times.

- Additional client specific requirements for the analysis of contract samples requires all ions present in the reference mass spectrum at a relative intensity > 10% must be present in the sample spectrum.
- Due to limitations of the "Target" software, analyst discretion is advised.

Table 11.6 Primary and Secondary quantitation ions for target compounds[2]

Analyte	Primary Ion	Secondary Ions
Phenol	94	65, 66
Bis (2-Chloroethyl) ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	46	148, 111
Benzyl Alcohol	108	79, 107
1,2-Dichlorobenzene	146	148, 111
2-Methylphenol	108	107, 79
Bis (2-Chloroisopropyl)ether	45	77, 121
3&4-Methylphenol	108	107, 79
N-Nitroso-di-n-propylamine	70	43, 101
Hexachloroethane	117	200.8, 198.8
1,4-Dioxane	88	58, 43
Benzaldehyde	77	106, 105
N-Nitrosodimethylamine	42	74
Aniline	93	66, 65
Acetophenone	105	77, 51
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	107	122, 121
Benzoic acid	122	105, 77
Bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129
4-Chloroaniline	127	129
Hexachlorobutadiene	224.9	118, 190
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Caprolactam	113	56, 55
Hexachlorocyclopentadiene	237	234.9
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	153
2,6-Dinitrotoluene	165	63, 89
3-Nitroaniline	138	108, 92
Acenaphthene	154	153, 152
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 89
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167

4-Nitroaniline	138	108, 92
1,2,4,5-Tetrachlorobenzene	216	214, 179
2,3,4,6-Tetrachlorophenol	232	131, 230
4,6-Dinitro-2-methylphenol	198	51, 105
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	283.8	142, 248.8
Atrazine	200	173, 215
Pentachlorophenol	265.8	263.8, 267.8
Phenanthrene	178	179, 176
Anthracene	178	176, 179
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 203
Benzidine	184	185, 92
Pyrene	202	200, 203
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
Chrysene	228	226, 229
Bis(2-ethylhexyl)phthalate	149	167, 279
Di-n-octylphthalate	149	43
Benzo(b)fluoranthene	251.99	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 25
Indeno(1,2,3-cd)pyrene	276	138, 277
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Carbazole	167	

[2] The information in this table was taken from Method 8270C. Please refer to the method for additional compounds and their applicable ions.

Analyte	Primary Ion	Secondary Ions
Internal Standards		
1,4-Dichlorobenzene-d_4	152	150, 115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265
Surrogates		
2-Fluorophenol (acid)	112	64
Phenol-d_6 (acid)	99	42, 71
Nitrobenzene-d_5 (BN)	82	128, 54
2-Fluorobiphenyl (BN)	172	171
2,4,6-Tribromophenol (acid)	330	332, 141
Terphenyl-d_{14} (BN)	244	122, 212

[2] The information in this table was taken from Method 8270C. Please refer to the method for additional compounds and their applicable ions.

11.6.2 Internal Standard Assignment List (from Method SW-846 8270C- Table 5): this section lists the internal standard compounds and all of the target compounds that are assigned to each internal for quantitation:

1,4-Dichlorobenzene-d4
Aniline
Benzyl alcohol
Bis(2-chloroethyl)ether
Bis(2-chloroisopropyl)ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
2-Fluorophenol (surrogate)
Hexachloroethane
2-Methylphenol
4-Methylphenol
N-Nitroso-dimethylamine
N-Nitroso-di-n-propylamine
Phenol
Phenol-d6 (surrogate)
1,4-Dioxane
Pyridine
2-Chlorophenol-d4(advisory surrogate)
Benzaldehyde
1,2-Dichlorobenzene-d4(advisory surrogate)

Naphthalene-d8
Acetophenone
Benzoic acid
Bis(2-chloroethoxy)methane
4-Chloroaniline
4-Chloro-3-methylphenol
2,4-Dichlorophenol
2,6-Dichlorophenol
2,4-Dimethylphenol
Hexachlorobutadiene
Isophorone
2-Methylnaphthalene
Naphthalene
Nitrobenzene
Nitrobenzene-d8 (surrogate)
2-Nitrophenol
1-Methylnaphthalene
1,2,4-Trichlorobenzene

Acenaphthene-d10
Acenaphthene
Acenaphthylene
1,2-Diphenylhydrazine
2-Chloronaphthalene
4-Chlorophenyl phenyl ether
Dibenzofuran
Diethyl phthalate
Dimethyl phthalate
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Fluorene
2-Fluorobiphenyl (surrogate)
Hexachlorocyclopentadiene
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
4-Nitrophenol
Biphenyl
2,4,6-Tribromophenol (surrogate)
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
1,2,4,5-Tetrachlorobenzene

Phenanthrene-d10
Atrazine
Anthracene
4-Bromophenyl phenyl ether
Di-n-butyl phthalate
4,6-Dinitro-2-methylphenol
Carbazole
Fluoranthene
Hexachlorobenzene
N-Nitroso-diphenylamine
Pentachlorophenol
Phenanthrene

Chrysene-d12
Benzidine
Benzo(a)anthracene
Bis(2-ethylhexyl)phthalate
Butyl benzyl phthalate
Chrysene
3,3'-Dichlorobenzidine
Pyrene
Terphenyl-d6 (surrogate)
Di-n-octyl phthalate
Indeno(123,cd)pyrene

Perylene-d12
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Dibenz(a,h)anthracene

11.6.3 Quantitative Analysis – Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

11.6.3.1 Raw Data Results: The GC/MS data system will calculate the concentration of each analyte in the sample extract. If supplied with the preparation parameters, the system may be able to calculate the results back to the original matrix. The calculation for the concentration of the target analyte in the original matrix is listed below and is based on the calibration table in units of ppm (ug/mL).

If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents near the middle of the linear range of the curve.

11.6.3.1.1 Results Calculation – Aqueous Sample:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(C_x)(V_x)(DF)}{(V_s)}$$

Where:

C_x = Concentration in extract (µg/mL).
V_x = Volume of final extract (mL).
DF = Dilution factor.
V_s = Volume of water sample extracted (mL).

11.6.3.1.2 Results Calculation – Soil / Solid calculations:

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(C_x)(V_x)(1000)(DF)}{(W_s)}$$

Where:

C_x = Concentration in extract (µg/mL).
V_x = Volume of final extract (mL).
DF = Dilution factor.
W_s = Weight of soil sample extracted (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

11.6.3.1.3 Results Calculation – Biota calculations:

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(C_x)(V_x)(1000)(DF)}{(W_s)}$$

Where:

C_x = Concentration in extract (µg/mL).
V_x = Volume of final extract (mL).
DF = Dilution factor.
W_s = Weight of biota sample extracted (g).

11.6.4 Tentatively Identified Compounds (TICs) – For some samples, identification may be desired for non-target compounds. A mass spectral library search may be conducted to attempt assignment of tentative identifications. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12 QUALITY CONTROL

12.1 Instrument Quality Control: Refer to Table 10.2 for initial and continuing calibration criteria and corrective actions.

12.1.1 South Carolina requires the use of a linear calibration model. Either the %RSD is less than 15% and the average RF is used for quantitation or a linear regression with a correlation coefficient greater than or equal to 0.99 must be used for all analytes listed in SW 846 Method 8270C.

12.2 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One (1) per 20 samples	1) Target analytes must be less than reporting limit. 2) If results are reported to MDL, target analytes in MB should be non-detect	1) Qualify results and / or re-extract associated samples. ***Exceptions:*** 1) If sample ND, report sample without qualification 2) If sample result <20x MB detects, and sample cannot be re-extracted or reanalyzed, report sample with appropriate qualifier indicating blank contamination. 3) If sample result >20x MB detects, report sample.
Laboratory Control Sample (LCS)	Method specified compounds: Base / Neutrals 1,2,4-Trichlorobenzene Acenapthene 2,4-Dinitrotoluene Pyrene N-Nitroso-di-n-propylamine 1,4-Dichlorobenzene *OR (alternative)* 70 compound LCS Mix	One (1) per batch of up to 20 samples. LCSD is performed for water samples where no volume provided for MS / MSD. Acids Pentachlorophenol Phenol 2-Chlorophenol 4-Chloro-3-methylphenol 4-Nitrophenol	Laboratory derived limits Method Specified List: All compounds must pass control criteria, with no exceptions. Full Target List: Marginal exceedances allowed according to NELAC 2003 Chap 5 D.1.1.2.1.e	1) Reanalyze the LCSto verify failure. 2) If LCS passes, review samples for potential injection problems 3) If problem persists, check spike solution 4) Re-extract 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. 2) Additional client specific requirements for the analysis of contract samples requires that hexacyclopentadiene, N-Nitrosodimethylamine, pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4-chloro3-methylphoneol, 2-nitrolaniline, 3-nitroaniline, and 4-chloroaniline recover ≥ 10%
Matrix Spike (MS)	Method specified compounds: Base / Neutrals 1,2,4-Trichlorobenzene Acenapthene 2,4-Dinitrotoluene Pyrene N-Nitroso-di-n-propylamine 1,4-Dichlorobenzene *OR (alternative)* 70 Compounds LCS Mix	One pair per batch of up to 20 samples Acids Pentachlorophenol Phenol 2-Chlorophenol 4-Chloro-3-methylphenol 4-Nitrophenol	Laboratory derived limits	1) If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences on parent sample only
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup	One (1) for every 5% of all environmental samples	Laboratory Derived Limits	1) Report results with an appropriate footnote.

12.3 **Sample Quality Control**

Table 12.2 – Sample Quality Control criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	1,4 Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	**Retention Time:** RT must be ± 30 seconds from last calibration check on all samples	**Retention Time Failure:** 1) If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting. 2) Affected samples should be reanalyzed in the absence of an obvious instrument or matrix related interference.
Surrogate Standards	Nitrobenzene-d5 2-Fluorobiphenyl Terphenyl-d14 Phenol-d6 2-Fluorophenol 2,4,6-Tribromophenol	Added to all samples, spikes, control samples and method blanks prior to extraction	Laboratory derived limits	1) Reanalyze extract to confirm failure. 2) Assess impact of sample matrix 3) In the absence of obvious matrix interference (high background, extremely dark extract), re-extract sample. ***Exceptions:*** 1) Surr rec above criteria and target compounds < RL, result may be reported with appropriate footnote. 2) Surr rec out of control due to obvious sample matrix interference (i.e. co-elution), report results with appropriate footnote. 3) One acid and one base surrogate compound is allowed to be out of control. If two or more acid or base surrogate compounds are out of control, the samples must be re-extracted and reanalyzed. If there is insufficient sample available for re-extraction, the data must be appropriately qualified.

13 METHOD PERFORMANCE

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted annually per S-ALL-Q-004, Method Detection Limit Studies for each matrix 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.

13.2.1 Analysis of four (4) replicates of reagent water spiked with 250 uL of the 8270 LCS Spiking Solution and 10 uL of nNPDA at a concentration of 50 ug/L or equivalent to the LCS. The recovery is to be within the current water LCS QC limits for the known concentrations and 30% RSD for all replicates.

13.2.2 Analysis of four (4) replicates of Ottawa sand spiked with 250 uL of 8270C LCS Spiking Solution and 10 uL of nNPDA at a concentration of 1670 ug/kg or equivalent to the LCS. The recovery is to be within the current LCS QC acceptance limits for the known concentration and 30% RSD for all replicates.

13.3 Method Modifications

Method modifications for EPA method 8270C are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- 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.
- Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.

14 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in the most current revision of S-GB-S-006, *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 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

15.2 USEPA, SW-846, Method 8000B, "Determinative Chromatographic Separations", December 1996.

15.3 USEPA, EPA 625, "Base/Neutrals and Acids".

16 TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS, APPENDICES, ETC.

Attachment I: Client Specific Requirements.

17 REVISIONS

Document Number	Reason for Change	Date
SOT-ALL-O-001-Rev.00	Restructured document format. Added tables and other content to create an "SOP Template" in which local edits are allowed. Modified signature page to include department manager and SOP template number. Updated SOP template number. Made the tuning criteria red so edits can be made to reflect local tuning criteria used. Updated calibration sections with new calibration policies described in the PASI Quality Manual revision 10.0.	28Nov2006
S-ALL-GB-O-001-Rev.00	First Issue using Corporate Template	04Jun2008
S-GB-O-049-Rev.00	Renamed document to follow Document numbering SOP: Renumbered document. Incorporate 1,2,4,5-Tetrachlorobenzene in all sections in document. Included client specific criteria and information. Added Attachment I	22Sep2009
S-GB-O-049-Rev.01	Updated Table 10.2 CCC Criteria to 15% RSD acceptance criteria for Non-ccc analytes when CCC analytes are not present. Updated SOP references	11Feb2010
S-GB-O-049-Rev.02	Updated numbering throughout document. Updated references throughout document. Section 14: Updated SOP reference for S-GB-S-006 Waste Handling and Management	06Jul2010
S-GB-O-049-Rev.03	Updated references in sections 10.2.1.2 to 11.6.4, 10.4 to Table 10.2, 10.5.1 to 9.2.4.6, and Section 11.2 to Section 11.2	09Jul2010

18 ATTACHMENT I:

Throughout document, reference to Client Specific requirements refers to samples analyzed following the BP Technical Requirements Version 9.0.

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STANDARD OPERATING PROCEDURE

DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS (SELECTIVE ION MONITORING)

REFERENCE METHODS: EPA Method SW-846 Method 8270C-SIM

LOCAL SOP NUMBER:	S-GB-O-050-REV.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-050-REV.00
SOP TEMPLATE NUMBER:	SOT-ALL-O-008-Rev.00

LOCAL APPROVAL

Nils Melberg, Laboratory General Manager — Date: 07/07/11

Kate Grams, Laboratory Quality Manager — Date: 7/7/11

Chris Haase, Department Manager — Date: 7/7/11

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

This Standard Operating Procedure (SOP) is used to determine the concentration of semi-volatile organic compounds in water and solid samples based on SW-846 Method 8270C while using Selective Ion Monitoring (SIM). Samples for analysis are prepared by SW846 Method 3510C and SW846 Method 3546 following Pace SOPs S-GB-O-053 *Separatory Funnel Extraction by SW846 3510C*, and S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbon,s and Base/Neutral/Acid, and Total Petroleum Hydrocarbons s in Solid Matrices by SW846 3546*.

2. SUMMARY OF METHOD

2.1 Sample extracts are prepared for analysis by an appropriate sample preparation method. The semi-volatile organic compounds are introduced into the gas chromatograph (GC) by injecting an aliquot of the sample extract. The GC conditions are programmed to separate the analytes. The GC effluent is directly introduced to a mass spectrometer (MS) for both identification and quantification of analytes. Analytes are identified by comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected major (quantitation) ion relative to an internal standard using a multi-point calibration curve.

3. SCOPE AND APPLICATION

3.1 This procedure is principally used to determine concentrations of polynuclear aromatic hydrocarbons (PAHs) but can also be used to determine concentrations of other neutral, acidic, and basic semi-volatile organic compounds in extracts prepared from many types of water samples, soil samples and wastes. Analytes must be soluble in dichloromethane and amenable to capillary gas chromatography. A list of applicable compounds is shown herein. Pace Reporting Levels (PRLs) are also shown for water and soil samples. PRLs are subject to change based on current analytical system performance and actual sample matrices.

3.2 This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interference with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data

3.3 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of semi-volatile configured GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.4 This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

4. INTERFERENCES

4.1 Interferences may be introduced into sample extracts by contaminants in solvents, reagents, glassware, and any other material that comes in contact with the sample or extract during extract preparation. These interferences must be closely monitored by analyzing Method Blank samples and taking corrective action as required.

4.2 Matrix interferences may result from materials co-extracted from some samples.

4.3 Significant phthalate contamination may result at any time if consistent quality control is not practiced. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

4.4 Contamination by carryover can occur when high concentration extracts are analyzed prior to low concentration extracts. The contamination may also cause degradation of labile analytes. Whenever carryover is suspected, the effected extracts should be re-analyzed. If significant degradation of the GC/MS systems is suspected, system performances samples should be analyzed and corrective action taken as needed.

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.

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.

5.3 Equipment

5.3.1 Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones.

5.3.2 The GC pneumatic system uses gas under high pressure. These high pressures introduce the risk of injury due to flying objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

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.

6.1 Run Sequence Log – A logbook that lists all injections and analyses performed on a particular piece of equipment regardless of the use of the data collected from each analysis.

6.2 Tune Period – The period after the DFTPP instrument tune check within which analyses may be performed.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	One (1) 1-Liter Amber Glass	None	4 ± 2°C	7 days
Soil/Solid (non-aqueous)	One (1) 8-oz Glass Jar	None	4 ± 2°C	14 days
Extracts	2-5mL Glass vials, same as used for standard storage	None	≤-10°C	40 days

8. EQUIPMENT AND SUPPLIES

8.1 Instrumentation

Analytical Instrument/Peripherals	EPIC Pro Name	Serial Number
HP 6890 GC	40MSS2	US00024411
HP 5973 Mass Selective Detector	40MSS2	US81221570
HP 7683 Auto-injector	40MSS2	US95110867
HP 7683 Tray	40MSS2	US11111030
Edwards E2M2 Rough Pump	40MSS2	54939
HP Chem Station Data System	40MSS2	_
HP 6890 GC	40MSS3	US00032758
HP 5973 Mass Selective Detector	40MSS3	US90460025
HP 7683 Auto-injector	40MSS3	US95110858
HP 7683 Tray	40MSS3	US94706602
Edwards HP# G1099-80023 Rough Pump	40MSS3	996318298
HP Chem Station Data System	40MSS3	
HP 6890 GC	40MSS4	US00024414
HP 5973 Mass Selective Detector	40MSS4	US81221586
HP 7683 Auto-injector	40MSS4	US81000351
HP G2614A Tray	40MSS4	CN24822508
Gerstel MACH Unit	40MSS4	1077
Edwards HP# G1099-80023 Rough Pump	40MSS4	
HP Chem Station Data System	40MSS4	

8.2 Chromatography Supplies

Item	Vendor	Model / ID	Catalog #	Description
Analytical Column	Restek	XTI-5 w/ Integraguard	12223-124	30 m, 0.25 mm ID, 0.25 df
Gerstel MACH Column	J&W	DB-5MS	100-2000	30 m, 0.25 mm ID, 0.50 df
Fluorocarbon O-rings	Restek		20377	
Vespel/Graphite Ferrules	Restek		20229	1/16” x 0.4 mm ID
Gooseneck Splitless Liner	Restek		20800	4 mm x 6.5 x 78.5 for Aligent GCs
Guard Column	Restek		20231	0.5 MID
Ferrules	Fisher Scientific		5-5	

8.3 Glassware

Glassware	Description	Vendor / Item # / Description
Volumetric Flasks	10mL, 25mL, 50mL	Class A
Glass Storage Vials	5mL, 10mL, 12mL, with Teflon-lined screw caps	MG Scientific / T102-3-INV, T102-1-INV V138-19, B510-1
Glass Autosampler Vials	2.0 mL with Teflon-lined crimp caps	MG Scientific / V300-3 / V300-20N

8.4 General Supplies

Glassware	Description	Vendor/ Item # / Description
Gas tight syringes	10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL, as needed, Hamilton or equivalent.	Fisher Scientific / Various
Pipettes	Borosilicate Glass 9" Pipette	MG Scientific / D200-9

9. REAGENTS AND STANDARDS

9.1 Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Dichloromethane		Pesticide Grade or equivalent

9.2 Analytical Standards

9.2.1 Definitions

Standards are required for mass spectrometer tuning, initial calibration, calibration verification standards, second source verification, internal standards, surrogates, and for preparing LCS, MS, and MSD samples. Table 9.1 describes the standards used.

Table 9.1 Standard Definitions and vendors

Standard	Description	Comments
Tune Standard	Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, pentachlorophenol, and benzidine solution in dichloromethane used to verify ion response ratios and system inertness prior to analysis. [For PAH only analysis, breakdown and tailing factors do not need to be evaluated or controlled]	Must inject no more than 50ng on column. The DFTPP must meet ion ratio criteria as per Section 12.2.1.
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the accuracy of the calibration curve.	
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Naphthalene-d_8 Acenaphthene-d_{10} Phenanthrene-d_{10} Chrysene-d_{12} Perylene-d_{12}
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to extraction.	2-Fluorobiphenyl Terphenyl-d_{14}
Spiking Standard	This solution contains all target analytes and is prepared from the same standards as the calibration standards.	

9.2.2 Storage Conditions

Table 9.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	▪ Concentrated reference solution purchased directly from approved vendor	▪ Manufacturer's recommended expiration date for unopened ampulated standards. ▪ All other stock standards must be replaced 1 year after ampule is opened or on expiration date, whichever is sooner.	▪ 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	▪ 1 year from preparation or the expiration date listed for the stock source, whichever is sooner. ▪ Working solutions must be checked frequently and replaced if degradation or evaporation is suspected.	▪ Store in amber vials with Teflon lined screw caps ▪ Manufacturer's recommended storage conditions for stock source solution. ▪ If stock source conditions conflict, store according to method requirements.

9.2.3 Standard Sources

Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat materials are present in Table 9.3. Recipes for preparing dilutions and working standards, and concentrations for all compounds are presented under Section 9.2.5. All intermediate standards are prepared using Fisher Optima grade (or better) dichloromethane and stored frozen in glass vials with Teflon lined screw caps or Mininert valves or as recommended by the standard manufacturer.

9.2.4 Stock Standards

Table 9.3 Stock Standards

Standard	Conc.	Purity	Manufacturer	Vendor	Catalog #
DFTPP Tuning Standard	1000 ug/mL	99%	Supelco	Supelco	47548-4
Internal Standard	4000 ug/mL	99%	Restek	Restek	31006
Surrogate Standard	5000 ug/mL	99%	Restek	Restek	31082
Calibration Standard	500 ug/mL	99%	Accustandard	Accustandard	M-610-FL-R-5X
Benzo(e)pyrene Calibration Standard	1000 ug/mL	99%	Absolute Standard	Absolute Standard	71016
Initial Calibration Verification Standard	2000 ug/mL	99%	Supelco	Supelco	47543-U
Initial Calibration Verification Standard for Benzo(e)pyrene	50ug/mL	99%	Accustandard	Accustandard	H-112S

9.2.5 Preparation Procedures

9.2.5.1. Intermediate / Working Standard Preparation

Intermediate or working standards are prepared in dichloromethane. Standards made for direct analysis on the GC/MS are made in dichloromethane. Standards made for addition into samples as part of the preparation are made into dichloromethane. Depending on the volume of each solution needed, the standards are brought to volume in volumetric flasks or prepared in smaller, glass vials and brought to volume by additions of solvent with micro syringes.

Volumetric Flask Standard Preparation – Fill appropriate volumetric flask 2/3 full with dichloromethane. Introduce appropriate amount of standard into dichloromethane in flask with a micro-syringe. Dilute to volume with dichloromethane.

9.2.5.2. Calibration Standard Preparation

Calibration standards are made into dichloromethane for the purpose of direct analysis by the analytical instrumentation. The standards must be made in a volumetric fashion. Several alternatives exist but the method employed by Pace – Green Bay utilizes volumetric flasks according to the following procedure. The individual standards can be made according to the details provided Section 9.2.5.3.

9.2.5.3. Preparation of Analytical Standard Solutions

Standards are prepared from commercially available multi-compound stock solutions. The sources of materials, recipes for preparing dilutions and working standards, and concentrations for all compounds are presented below. All standards are prepared using dichloromethane and stored in clear vials with PTFE lined caps.

Storage and Stability of Analytical Standards - All standards must be stored in the dark at less than –10 °C or at a temperature recommended by the manufacturer. They must be replaced every 12 months, or sooner if the standards show signs of degradation. As each standard, from the vendor is opened, record all pertinent information in the stock standard logbook. Record all standard preparations in the working standard logbook.

9.2.5.3.1 DFTPP tuning standard

A Dichloromethane solution containing 50ng/uL of decafluorotriphenylphosphine (DFTPP) is prepared. A stock solution; Supelco cat.# 47548-4; containing DFTPP at 1000ug/mL, is diluted taking 0.500mL and diluting to 10mL of dichloromethane.

9.2.5.3.2 Internal Standard solutions

Stock Solution - Obtained from Restek cat.#31006 containing 1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12 and Perylene-d12 all at 4000ug/mL in dichloromethane.

Water Intermediate Standard - This intermediate standard is prepared by diluting 50uL upto 10mLs of dichloromethane, producing a solution that is 20ug/mL for each component. 10uL of this standard is added to 1mL of every standard, sample, spike and blank analyzed by this method. Producing a on column concentration of 200ug/L.

Soil Intermediate Standard - This intermediate standard is prepared by diluting 500uL upto 1.0mL of dichloromethane, producing a solution that is 2000 ppm for each component. 10uL of this standard is added to 1mL of every standard, sample, spike and blank analyzed by this method. Producing a on column concentration of 20 ppm.

9.2.5.3.3 Surrogate Standard solution

Stock Solution obtained from Restek, cat. #31082 containing 1,2-Dichlorobenzene-d4, Nitrobenzene-d5, 2-Fluorobiphenyl and p-Terphenyl-d14 all at 5000ug/mL in dichloromethane. An intermediate standard is prepared by diluting 300uL up to 10mLs of dichloromethane, producing a standard that is 150ug/mL of each component.

Water Samples

This intermediate is further diluted to produce the daily working standard by diluting 66.7uL of the 150ug/mL intermediate to 50mLs of Dichloromethane. 1.0mL of this daily working standard is spiked into all QC and Samples, producing an on column value of 200ug/L.

Soil Samples

100 uL of the 150 ug/mL intermediate is spiked into all QC and samples, producing an on column value of 15 ug/mL.

9.2.5.3.4 **Initial Calibration Intermediate Standard**

Water Samples

Stock solution is obtained from Accustandard, product # M-610-FL-R-5X, containing the 18 PAH compounds, all at 500ug/mL. The intermediate is prepared by diluting 20uL of the 500ug/L PAH stock along with 67uL of the 150ug/L surrogate intermediate (9.2.5.3.3) up to 10.0mLs dichloromethane, producing a intermediate calibration standard that is 1000ug/L of all components

Soil Samples

Stock solution is obtained from Accustandard, product # M-610-FL-R-5X, containing the 18 PAH compounds, all at 500ug/mL. The intermediate is prepared by diluting 500 uL of the 500 ug/mL PAH Stock Standard along with 50 uL of the 5000 ug/mL B/N Surrogate Stock Solution to 10.0 mL with methylene chloride producing the high standard of 25 ug/mL. Per Client Request Benzo(e)pyrene may be included as a separate calibration curve. Stock Solution is obtained from Absolute Standards, catalog # 71016, containing Benzo(e)pyrene at 1000ug/mL. The intermediate is prepared by diluting 250uL of 1000ug/mL Benzo(e)pyrene Stock Standard up to 10.0mLs dichloromethane, producing an intermediate calibration standard that is 25ug/mL. The standards are then further diluted to obtain the concentrations used for calibration.

9.2.5.3.5 **Initial Calibration Working Standards**

9.2.5.3.5.1 Water Samples

The following working calibration standards are prepared by using the Initial Calibration Intermediate Standard for Water Samples listed above (9.2.5.3.4):

Standard Concentration (ng on column)	Amount of Intermediate Standard Added (uL)	Amount of Intermediate Internal Standard Added (uL)	Final Volume (uL)
25	25	10	1010
50	50	10	1010
100	100	10	1010
200	200	10	1010
300	300	10	1010
500	500	10	1010
1000	1000	10	1010
CCV - 200	400	10	1010

9.2.5.3.5.2 Soil Samples

The following working calibration standards are prepared by using the Initial Calibration Intermediate Standard for Soil Samples listed above (9.2.5.3.4):

Standard Concentration (ng on column)	Amount of Intermediate Standard Added (uL)	Amount of Intermediate Internal Standard Added (ul)	Final Volume (uL)
0.25	10	10	1010
0.50	20	10	1010
1.0	40	10	1010
4.0	160	10	1010
10.0	400	10	1010
20.0	800	10	1010
25.0	1000	10	1010
CCV – 10.0	400	10	1010

9.2.5.3.6 Initial Calibration Verification Standard

9.2.5.3.6.1 Water Samples

Stock Solution is obtained from Supelco, cat# 47543-U, containing the 18 PAH compounds all at 2000ug/mL. A working solution is prepared by diluting 5uL of the 2000ug/mL PAH stock, along with 67uL of the 150ug/mL B/N surrogate intermediate(9.2.5.3.3) up to 50mLs with dichloromethane. This produces an ICV with a concentration of 200ug/L.

9.2.5.3.6.2 Soil Samples

Stock solution is obtained from Supelco, cat # 47543-U, containing the 18 PAH compounds, all at 2000 ug/mL. An intermediate is prepared by diluting 100 uL of the 2000 ug/mL PAH Stock along with 40 uL of 5000 ug/mL B/N Surrogate Stock Solution to 2.0 mL with dichloromethane. To produce the working ICV solution, the above intermediate ICV solution is further diluted by taking 100 uL of the intermediate ICV and diluting to 1.0 mL with dichloromethane to produce a 10 ug/mL working ICV standard. A separate Benzo(e)pyrene ICV is required. Stock Solution is obtained from Accustandard, catalog # H-112S, containing Benzo(e)pyrene at 50ug/mL. The working ICV solution is prepared by diluting 200uL of 50ug/mL Benzo(e)pyrene Stock Standard up to 1.0mLs dichloromethane to produce a working initial calibration verification standard that is 10ug/L.

9.2.5.3.7 LCS/MS Spiking Standard

9.2.5.3.7.1 Water Samples

Stock solution is obtained from Accustandard, product # M-610-FL-R-5X, containing the 18 PAH compounds, all at 500ug/mL. Intermediate standard is prepared by diluting 10uL of the 500ug/mL upto 25.0mLs with Dichloromethane. 1.0 mL of this standard is spiked into all Lab Control and Matrix Spikes, producing a on-column concentration of 200ug/L.

9.2.5.3.7.2 Soil Samples

Stock solution is obtained from Accustandard, product # M-610-FL-R-5X, containing the 18 PAH compounds, all at 500ug/mL. 20 uL of this solution is spiked into all LCS and MS samples, producing an on-column concentration of 10 ug/mL.

10. CALIBRATION

10.1 Tune Verification

The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. For analysis by Selective Ion Monitoring, the response ratio criteria for DFTPP may be immaterial unless a custom spectral library were established under SIM conditions. Therefore, unless a program specific requirement mandates ion ratio criteria be followed, the laboratory must analyze a DFTPP standard but only for the purpose of verifying the alignment of the mass spectral axis. If the quality program requires otherwise and the ion ratios do not meet the criteria for the method (see section 11.2.1), follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be verified after the tuning procedures.

Refer to section **11.2 Procedures** for details on the analysis and evaluation of this standard.

10.2 Initial Calibration (ICAL)

10.2.1 Analysis of Standards

An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to Tables 10.1 and 10.2 for compound concentrations.

Table 10.1 – Calibration Standard Compound Concentrations for Water Analysis

Analyte	PQL water (ug/L)	Std 1 ug/L	Std 2 ug/L	Std 3 ug/L	Std 4 ug/L	Std 5 ug/L	Std 6 ug/L	Std 7 ug/L
Acenaphthene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Acenaphthylene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Anthracene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benz(a)anthracene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benzo(a)pyrene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benzo(b)fluoranthene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benzo(g,h,i)perylene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Benzo(k)fluoranthene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Chrysene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Dibenz(a,h)anthracene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Fluoranthene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Fluorene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Indeno(1,2,3-cd)pyrene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
2-Methylnaphthalene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Naphthalene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Phenanthrene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
Pyrene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0
1-Methylnaphthalene	0.050	0.025	0.050	0.10	0.20	0.30	0.50	1.0

Table 10.2 – Calibration Standard Compound Concentrations for Soil Analysis

Analyte	PQL soil (ug/kg)	Std 1 (ug/kg)	Std 2 (ug/kg)	Std 3 (ug/kg)	Std 4 (ug/kg)	Std 5 (ug/kg)	Std 6 (ug/kg)	Std 7 (ug/kg)
Acenaphthene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Acenaphthylene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Anthracene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benz(a)anthracene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(a)pyrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(b)fluoranthene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(e)pyrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(g,h,i)perylene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Benzo(k)fluoranthene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Chrysene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Dibenz(a,h)anthracene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Fluoranthene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Fluorene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Indeno(1,2,3-cd)pyrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
2-Methylnaphthalene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Naphthalene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Phenanthrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
Pyrene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3
1-Methylnaphthalene	16.7	8.3	16.7	33.3	133.3	333.3	666.7	833.3

10.2.2 Calibration Response Factors

10.2.2.1 Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

10.2.2.2 To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:
A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L).
C_x = Concentration of the compound being measured (µg/L).

10.2.2.3 Most, if not all modern chromatography data systems are capable of calculating this factor and using it to quantify analyte concentrations. The 8270C method has minimum requirements that these response factors must meet in order to be considered valid. The method uses a subset of the target analyte list to evaluate the performance of the system. These compounds are referred to as the System Performance Check Compounds or the SPCCs. The SPCCs serve as an indicator of instrument sensitivity and, by meeting a minimum value, ensure that the laboratory has adequate sensitivity to analyze and reliably report data for environmental samples. For the SIM method, all target compounds are considered to be SPCC compounds and the average RF for each SPCC compound must be as follows:

Naphthalene	≥0.700
2-Methylnaphthalene	≥0.400
Acenaphthylene	≥0.900
Acenaphthene	≥0.900
Fluorene	≥0.900
Phenanthrene	≥0.700
Anthracene	≥0.700
Fluoranthene	≥0.600
Pyrene	≥0.600
Benzo(a)anthracene	≥0.800
Chrysene	≥0.700
Benzo(b)fluoranthene	≥0.700
Benzo(k)fluoranthene	≥0.700
Benzo(a)pyrene	≥0.700
Benzo(e)pyrene	≥0.700
Indeno(1,2,3-cd)pyrene	≥0.500
Dibenzo(a,h)anthracene	≥0.400
Benzo(g,h,i)perylene	≥0.500

Two of the PAH's response factors are allowed to not meet these requirements.

10.2.3 Calibration Curve Fit

The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the "goodness of fit".

10.2.3.1 Average Response Factor (ARF): The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the ARF (see Table 10.3).

The % RSD is calculated as follows: %RSD = SD*100 / ARF

Where: SD = Standard deviation of the averaged RFs for a given compound

The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The ***Calibration Check Compounds (CCCs)*** are a subset of the target analyte list that must meet specific criteria (see Table 10.3) for the calibration to be acceptable. For the CCCs, the %RSD for each is compared to the method criteria (≤30% for CCCs). If that of any CCC exceeds the criteria, the system needs to be inspected for potential sources of errors and recalibrated. **For the SIM method, all target compounds are considered CCC compounds.**

10.2.3.2 Linear Regression: The linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of y=ax+b where "a" is the slope of the line and "b" is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y axis at the origin. However, because the linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. A calculation of the correlation coefficient "r" is used to determine the acceptability of a linear regressed curve. A linear curve maybe used if the RSD is >15%, but <30% (see Table 10.3).

10.2.3.3 Non-linear Regression: The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

A calculation of the coefficient of determination (COD) is used to determine the acceptability of a non-linear regressed curve (see Table 10.3). Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious problem is discovered and documented. The point must be dropped in its entirety or reanalyzed. Re-analysis should be within the same 12 hour time window and must occur within 8 hours of the original analysis. If not possible, the entire calibration curve must be re-analyzed. Within a new 12 hour window.

10.3 Calibration Verification

10.3.1 Second Source Verification (SCV)

In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the "fit" of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

Therefore, to assess the accuracy relative to the purity of the standards, a single 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. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{SCV} - \text{TrueValue}_{SCV}] / \text{TrueValue}_{SCV} * 100$$

See Section 9.2.5.3.6 for details on the preparation of this standard. See Table 10.3 for control criteria

10.3.2 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 at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished 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 change calculated. The percent change must meet the method specified criteria for the analysis to proceed for an additional 12 hours.

The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

% Difference: $[RF_{CCV} - AvgRF_{CAL}] / AvgRF_{CALl} \times 100$

% Drift: $[Result_{CCV} - TrueValue_{CCV}] / TrueValue_{CCV} \times 100$

Table 10.3 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor Linear Regression Non-linear Regression	%RSD ≤ 15% r ≥ 0.99 COD ≥ 0.99	If not met, try linear regression fit if RSD is >15% and <30%. If not met, try non-linear regression fit If not met, remake standards or perform instrument maintenance and recalibrate
System Performance Check Compounds (SPCCs)	All Target compounds	Naphthalene ≥0.700 2-Methylnaphthalene ≥0.400 Acenaphthylene ≥0.900 Acenaphthene ≥0.900 Fluorene ≥0.900 Phenanthrene ≥0.700 Anthracene ≥0.700 Fluoranthene ≥0.600 Pyrene ≥0.600 Benzo(a)anthracene ≥0.800 Chrysene ≥0.700 Benzo(b)fluoranthene ≥0.700 Benzo(k)fluoranthene ≥0.700 Benzo(a)pyrene ≥0.700 Benzo(e)pyrene ≥0.700 Indeno(1,2,3-cd)pyrene ≥0.500 Dibenzo(a,h)anthracene ≥0.400 Benzo(g,h,i)perylene ≥0.500 Two of the PAH's response factors are allowed to not meet these requirements.	Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, poor purging efficiency, and active sites in the column or chromatographic system. Additional client specific requirements for the analysis of contract samples requires that all PAH's and surrogate compounds also be considered SPCCs and must meet the minimum RRF criterion of 0.05.
Calibration Check Compounds (CCCs)	All Target compounds	%RSD < 30%	%RSD for the calibration check compounds (CCC's) must be **≤30**% regardless of curve fit used.
Second Source Verification Standard	Immediately after each initial calibration	% Diff ±30%	Acceptance criteria are ±30% for all analytes. See S-ALL-Q-025 Additional client specific requirements for the analysis of contract samples requires that all compounds must be within ±20%
Continuing Calibration Verification	Prior to the analysis of any samples and every 12 hours thereafter		If the requirements for continuing calibration are not met, these corrective actions must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back.
	SPCCs	Must meet response criteria listed above	
	Internal Standard RT Internal Standard Response	RT ± 30 sec -50% to + 200%	Use midpoint calibration standard as reference Use midpoint calibration standard as reference
	CCCs	RF ± 20% Diff. Result ± 20% Drift	Use for Avg RF calibration curvesUse for linear and non-linear calibration curves

10.4 Calibration Corrective Actions

10.4.1 Calibration Linearity Problems

10.4.1.1Check instrumentation/equipment condition. Document instrument maintenance in the logbook.

10.4.1.2Perform another initial calibration.

10.4.1.3No data can be reported.

10.4.1.4Generate on Non-Conformance Memo.

10.4.2 Secondary Verification Problems

10.4.2.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.

10.4.2.2 Perform another initial calibration.

10.4.2.3 No data can be reported.

10.4.2.4Generate on Non-Conformance Memo

10.4.3 Continuing Verification Problems

10.4.3.1 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. (It is recommended that instrument maintenance be performed prior to the second CCV injection. Document all instrument maintenance in the appropriate logbook.) If the second CCV is acceptable, the analytical sequence is continued. If both CCVs fail, the analytical sequence is terminated and corrective action is initiated.

10.4.3.2 Complete recalibration of instrument.

10.4.3.3 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 injection log with a thorough explanation for the deviation from policy.

> *Exceptions:* If calibration verification is above the upper control limit, samples non-detected for those analytes may be reported without reanalysis.

10.4.4 General Comment: When constructing a linear initial calibration curve, the analyst can drop curve points as follows:

The lowest curve point can be dropped as long as there is a standard that can meet the necessary reporting limits of the associated samples (or the reporting limits would have to be raised accordingly).

The highest curve point, or points, can be dropped but this decreases the upper calibration range, thereby limiting the analyst to reporting data within this new calibration range (this may cause more dilutions).

Mid point injections in the curve can be removed if it can be proven that it was a bad injection, instrument failure, etc. The same curve point is required to be reanalyzed and incorporated in the curve prior to sample analysis. A supervisor or the Quality Manager is required to approve any such instance

11. PROCEDURES

11.1 GC/MS System Preparation

11.1.1 Operating Parameters

Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration.

Table 11.1 –Instruments and Operating Parameters

Instrument IDs	Component	Settings and Consumables	
40MSS2	Gas Chromatograph	**Column:** Restek XTI-5 w/ Integraguard 30 m, 0.25 mm ID, 0.25df **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite	Mass Range: 50-500 AMU Mode: SIM Pressure / Flow: 2.0 mL / min Initial Temperature: 100°C Initial Time: 1.5 min Final Temperature: 60°C / min to 155°C 20°C / min to 320°C 3 min hold Final Time: 13.61 min Injector Temperature: 295°C Transfer Line Temperature: 310°C Source Temperature: 310°C Injector : EPC in Pulsed Splitless Mode, 100 psi until 1.0 min
	Mass Spectrometer	Tune File: Named to date of tune	
	Autosampler	1.0 uL Injection 3 Solvent rinses from each solvent vial	
Instrument IDs	**Component**	**Settings and Consumables**	
40MSS3	Gas Chromatograph	**Column:** Restek XTI-5 w/ Integraguard 30 m, 0.25 mm ID, 0.25df **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite	Mass Range: 50-500 AMU Mode: SIM Pressure / Flow: 2.0 mL / min Initial Temperature: 100°C Initial Time: 1.5 min Final Temperature: 60°C / min to 155°C 20°C / min to 320°C 3 min hold Final Time: 13.61 min Injector Temperature: 295°C Transfer Line Temperature: 310°C Source Temperature: 310°C Injector : EPC in Pulsed Splitless Mode, 100 psi until 1.0 min Transfer Line Temperature: 310°C Source Temperature: 310°C Injector : EPC in Pulsed Splitless Mode, 70 psi until 1.0 min
	Mass Spectrometer	Tune File: Named to date of tune	
	Autosampler	1.0 uL Injection 3 Solvent rinses from each solvent vial	
Instrument IDs	**Component**	**Settings and Consumables**	
40MSS4	Gas Chromatograph	**Column:** Gerstel MACH Column DB-5MS 30 m, 0.25 mm ID, 0.50 df **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite	Mass Range: 50-500 AMU Mode: SIM Pressure / Flow: 2.0 mL / min Initial Temperature: 50°C Initial Time: 30 sec Final Temperature: 50°C / min to 270°C 5°C / min to 280°C 50°C / min to 340°C 129 sec hold Final Time: 10.25 min Injector Temperature: 270°C Transfer Line Temperature: 310°C Source Temperature: 310°C Injector : EPC in Pulsed Splitless Mode, 70 psi until 1.0 min
	Mass Spectrometer	Tune File: Named to date of tune	
	Autosampler	1.0 uL Injection 3 Solvent rinses from each solvent vial	

11.2 Tune Verification

At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified. This is done by analyzing a standard containing DFTPP (refer to table 9.3). The tune verification standard can be combined with the CCV standard provided that the amount of DFTPP introduced into the system meets the criteria in Table 11.2. For semi-volatile analysis, the system must also be verified for inertness **unless PAHs are the only class of analytes to be analyzed**. This is done simultaneously by the inclusion of DDT, benzidine and pentachlorophenol. DDT is used to verify breakdown conditions; benzidine and pentachlorophenol are used to check for tailing due to system activity.

11.2.1 Mass Axis Alignment / Ion Ratio Verification

After the analysis of this standard, the mass spectrum of DFTPP must be evaluated against the following criteria.

Table 11.2

Mass (m/z)	Mass Axis Criteria	Ion Abundance criteria
51	Present	30.0-80.0% of m/z 198
68	-	<2.0% of m/z 69
69	Present	Present
70	-	<2.0% of m/z 69
127	Present	25.0-75.0% of m/z 198
197	-	<1.0% of m/z 198
198	Present	Base peak, 100% relative abundance
199	Present	5.0-9.0% of m/z 198
275	Present	10.0-30.0% of m/z 198
365	-	>0.75% of m/z 198
441	-	Present, but less than m/z 443
442	Present	40.0-110.0% of m/z 198
443	Present	15.0-24.0% of m/z 442

To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for DFTPP. If the software has a program or macro for automatically selecting the spectra and evaluating the response ratios, use this option. Otherwise, the spectra must be obtained in one of the following manners, in the listed order.

1. **Take one scan for DFTPP; or**
2. **Using an average of three scans, centered on the apex of the peak; or,**
3. **Using an average of all scans across the width of the peak, taken at half height; or,**
4. **Using an average of all scans taken across the width of the peak from baseline to baseline.**

A background scan taken immediately before but not including the peak must be subtracted.

Once obtained, evaluate the ion ratios against the criteria listed above. If the ions and ratios meet the criteria, then analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date / time for the DFTPP tune verification. After that, the tune must be verified again to establish a new analytical window. The same Ion Abundance Criteria used for the DFTPP tune coupled with the initial calibration must be used for all subsequent analyses associated with that initial calibration.

11.2.2 Tailing Factor Verification

Benzidine and Pentachlorophenol should be present at their normal responses, and peak tailing should not be to an excess.

11.2.3 Breakdown Verification

The GC/MS system must be sufficiently inert such that DDT will not breakdown excessively while in the injection port. The inertness is assessed by calculating the percent breakdown of DDT into the products DDD and DDE. The calculation is performed as follows:

$$\text{\% DDT Breakdown} = \frac{\text{(Sum of responses for DDD and DDE)}}{\text{(Sum of responses for DDT, DDD and DDE)}} \times 100$$

The % breakdown must not exceed 20% unless all required compounds are within 20% on the CCV. If the breakdown of DDT exceeds this amount, samples may be analyzed if all requested analytes are within +/- 20% in the CCV (documented on an out-of-control form), otherwise corrective action must be taken prior to analysis of samples. The breakdown must be verified by the analysis of another breakdown standard after corrective action is taken. Follow the following steps to return the system to an acceptable operating condition.

If the tune standard does not meet the criteria, refer to the following corrective actions to address the problem:

- Retune the mass spectrometer following the equipment manufacturers' instructions. The tune status must be verified after the tuning procedures.
- If this fails, change filament and retune.
- If this fails, take down the mass spectrometer and clean the instrument.

11.3 Calibration Verification

After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. If it has already been determined that the instrument needs to be recalibrated, follow the procedures listed in section 10.2.1 (Analysis of Standards). Otherwise, analyze a Continuing Calibration Verification Standard to determine the current calibration status.

If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed section 10.4.3 (Continuing Verification Problems). If the tune verification has been combined with the CCV, the 12 hour analysis window begins from the analysis date / time of the CCV.

Note: In situations where the instrument will run unattended (i.e. overnight), the analyst may load sequential (back-to-back) CCVs in anticipation of that the first in the series may fail due to carry over from a previous sample. If so, both CCVs must be evaluated according to the protocol set forth in the Quality Assurance Manual within Section 6 – Equipment and Measurement Traceability.

11.4 Sample Preparation

11.4.1 Samples

11.4.1.1 Water Samples

Aqueous samples are prepared according to EPA Method 3510C. These procedures are contained in a separate standard operating procedure. Refer to S-GB-O-053 *Separatory Funnel Extraction by 3510C* for details on the preparation of aqueous samples.

Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10uL of the internal standard solution (9.2.5.3.2).

11.4.1.2 Soil Samples

Solid samples are prepared according to EPA Method 3546. These procedures are contained in a separate standard operating procedure. Refer to S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons, Base/Neutral/Acids, and Total Petroleum Hydrocarbons in Solid Matrices by 3546* for details on the preparation of soil or solid samples.

Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10uL of the internal standard solution (9.2.5.3.2).

11.4.1.3 Dilutions

Dilutions on sample extracts must be prepared in a volumetric fashion. Sample aliquots should be taken in volumetric syringes and brought to volume by the addition of solvent via an appropriate syringe. In the event a dilution is made to bring a target analyte into calibration range, the analyst should make a dilution such that the target analyte is roughly the equivalent of the mid calibration point whenever possible.

If dilutions are made on extracts that already contain internal standards, a proportional aliquot of internal standard solution must be added to the diluted extract based on the volume of diluent used.

11.5 Sample Analysis

11.5.1 GC/MS System Preparation

11.5.1.1 Operating Parameters – Set up the instrument parameters shown in Table 11.1.

11.5.1.2 System Tuning and GC Performance Checks – Analyze the Tuning Solution and tune the mass spectrometer to meet the criteria shown in Section 11.2.1. Verify acceptable GC system performance as described in Section 11.2.1. Print out a tune report.

11.5.2 Batch Sequence – Generate a sequence to run a batch of samples.

11.5.2.1 Initial Calibration – The typical batch for initial calibration should include:

Tune Standard
Calibration Level 1
Calibration Level 2
Calibration Level 3
Calibration Level 4
Calibration Level 5
Calibration and System Performance Solution

11.5.2.2 Sample Analysis – The typical batch for sample analysis should include the following. Preparation of LCS, MS, MSD, and Duplicate sample extracts is described in the appropriate sample preparation SOP.

Tune Standard
Calibration and System Performance Solution
Instrument Blank
Method Blank
Laboratory Control Sample
Laboratory Control Sample Duplicate
20 samples
Matrix Spike
Matrix Spike Duplicate

11.5.3 **Autosampler** – Load the autosampler with standards and samples for the batch created above.

11.5.4 **Analyze Samples** – Analyze all standards, quality control samples, and environmental samples.

11.6 Data Reduction

11.6.1 Qualitative Analysis

Retention Time Comparison: The relative retention time (RRT) of the sample component must be within ±0.06 RRT units of the component in the calibration verification standard. Extracted Ion Current Plots (EICPs) may be used to provide a more reliable assignment of RT in the presence of co-eluting components.

Mass Spectrum Comparison: The characteristic ions from the reference mass spectrum are defined as 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.

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.
- 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. When daily check standards meet criteria for resolution, nothing will be done. When daily check standard does not meet the method requirement for resolution, samples running under that check standard will be flagged stating the method requirement for resolution was not met for affected compounds.
- Under SIM conditions, only those ions collected will be present in the spectrum. Therefore, the best benchmark for spectral comparison should be the spectra obtained from the opening CCV. Ion intensity ratios should agree within 30% of those obtained in the standard.
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

Table 11.3 Ions

Compound	Primary Ion	Secondary Ion
Naphthalene	128	
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
Acenaphthylene	152	
Acenaphthene	154	152
Fluorene	166	165
Phenanthrene	178	
Anthracene	178	
Fluoranthene	202	
Pyrene	202	
Benzo(a)anthracene	228	226
Chrysene	228	226
Benzo(b)fluoranthene	252	
Benzo(k)fluoranthene	252	
Benzo(a)pyrene	252	
Indeno(1,2,3-cd)pyrene	276	
Dibenzo(a,h)anthracene	278	
Benzo(g,h,i)perylene	276	
Benzo(e)pyrene	225	
Nitrobenzene-d(5)-SS	82	
2-Fluorobiphenyl-SS	172	
Terphenyl-d(14)-SS	244	
Naphthalene-d(8)-IS	136	
Acenaphthene-d(10)-IS	164	
Phenanthrene-d(10)-IS	188	
Chrysene-d(12)-IS	240	
Perylene-d(12)-IS	264	

11.6.2 Quantitative Analysis – Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. See Table 11.4 for Internal Standard Assignments.

Table 11.4 Internal Standard Assignments

Compound	Internal Standard
Naphthalene	Naphthalene-d(8)-IS
2-Methylnaphthalene	Naphthalene-d(8)-IS
1-Methylnaphthalene	Naphthalene-d(8)-IS
Acenaphthylene	Acenaphthene-d(10)-IS
Acenaphthene	Acenaphthene-d(10)-IS
Fluorene	Acenaphthene-d(10)-IS
Phenanthrene	Phenanthrene-d(10)-IS
Anthracene	Phenanthrene-d(10)-IS
Fluoranthene	Phenanthrene-d(10)-IS
Pyrene	Chrysene-d(12)-IS
Benzo(a)anthracene	Chrysene-d(12)-IS
Chrysene	Chrysene-d(12)-IS
Benzo(e)pyrene	Perylene-d(12)-IS
Benzo(b)fluoranthene	Perylene-d(12)-IS
Benzo(k)fluoranthene	Perylene-d(12)-IS
Benzo(a)pyrene	Perylene-d(12)-IS
Indeno(1,2,3-cd)pyrene	Perylene-d(12)-IS
Dibenzo(a,h)anthracene	Perylene-d(12)-IS
Benzo(g,h,i)perylene	Perylene-d(12)-IS
2-Fluorobiphenyl-SS	Acenaphthene-d(10)-IS
Terphenyl-d(14)-SS	Chrysene-d(12)-IS

Raw Data Results: The GC/MS data system will calculate the concentration of each analyte in the sample extract. If supplied with the preparation parameters, the system may be able to calculate the results back to the original matrix. The calculation for the concentration of the target analyte in the original matrix is listed below and is based on the calibration table in units of ppm (ug/mL).

If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents near the middle of the linear range of the curve.

11.6.2.1 Results Calculation – Aqueous Sample:

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(I_s)}{(A_{is})(RF)(V_o)}$$

Where:

A_x= Area of characteristic ion for compound being measured.
I_s = Amount of internal standard injected (ng).
A_{is} = Area of characteristic ion for the internal standard.
RF = Average Relative Response factor for compound being measured.
V_o = Volume of water purged (ml), taking into consideration any dilutions made.

11.6.2.2 Soil/ Solid calculations:

$$Low\ \text{Conc.}\,(\text{ug/kg}) = \frac{(A_x)(I_s)}{(A_{is})(RF)(W_s)}$$

Where:

A_x, I_s, A_{is}, RF = Same as in water and water-miscible waste above.
V_t = Volume of total extract (ml).
V_i = Volume of extract added (ml) for purging.
S_v = Volume of diluted extract.
W_s= Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

12 QUALITY CONTROL

Instrument Quality Control: Refer to Table 10.3 for initial and continuing calibration criteria and corrective actions.

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One (1) per 20 samples	1) Target analytes must be less than reporting limit. 2) If results are reported to MDL, target analytes in MB should be non-detect	1) Qualify results and / or re-extract associated samples. ***Exceptions:*** 1) If sample ND, report sample without qualification 2) If sample result >20x MB result report sample without any qualifier. 3) If sample result <20x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Full Target List compounds	One (1) per batch of up to 20 samples. LCSD is performed for water samples where no volume provided for Ms/MSD.	Laboratory derived limits Full Target List: Marginal exceedances allowed according to NELAC 2003 Chap 5 D.1.1.2.1.e	1) If LCS passes, no further action necessary. 2) Check spike solution .or appropriately qualify data. 3) Re-extract samples where possible or appropriately qualify data. ***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)	Full Target List compounds	One (1) per batch of up to 20 samples, must include one TCLP MS for any analyzed in sequence	Laboratory derived limits	1) If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences. Qualify the parent sample only.
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup	One (1) for every 5% of all environmental samples	Laboratory Derived Limits	1) Report results with an appropriate footnote.

12.2 Sample Quality Control

Table 12.2 – Sample Quality Control criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	Naphthalene-d_8 Acenaphthene-d_{10} Phenanthrene-d_{10} Chrysene-d_{12} Perylene-d_{12}	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	**Retention Time:** RT must be ± 30 seconds from last calibration check on all samples	**Retention Time Failure:** 1) If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting. 2) Affected samples should be reanalyzed in the absence of an obvious instrument or matrix related interference.
Surrogate Standards	2-Fluorobiphenyl Terphenyl-d_{14}	Added to all samples, spikes, control samples and method blanks prior to extraction.	Laboratory derived limits	Assess impact of sample matrix In the absence of obvious matrix interference (high background, extremely dark extract), re-extract sample. ***Exceptions:*** Surr rec above criteria and target compounds < RL, result may be reported with appropriate footnote. Surr rec out of control due to obvious sample matrix interference (i.e. co-elution), report results with appropriate footnote. If any surrogate compounds is out of control, the samples must be re-extracted and reanalyzed. If there is insufficient sample available for re-extraction, the data must be appropriately qualified.

13 METHOD PERFORMANCE

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted annually per S--GB-Q-020, *Determination of the LOD and LOQ* for each matrix 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 Method Modifications

13.4 Method modifications for EPA method 8270C are as follows:

13.4.1 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.

13.4.2 All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.

13.4.3 Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.

14 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in S-GB-S-006, *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 8270C, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

15.2 USEPA, SW-846, Method 8000B, "Determinative Chromatographic Separations", December 1996.

16 TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS, APPENDICES, ETC.

Not applicable to this SOP.

17 REVISIONS

Document Number	Reason for Change	Date
SOT-ALL-O-008-rev.00	Restructured document format. Added tables and other content to create an "SOP Template" in which local edits are allowed. Modified signature page to include department manager and SOP template number. Updated SOP template number. Made the tuning criteria red so edits can be made to reflect local tuning criteria used. Updated calibration sections with new calibration policies described in the PASI Quality Manual revision 10.0.	28Nov2006
S-ALL-O-008-Rev.00	First issue in Template format.	15May2008
S-GB-O-050-Rev.00	Updated Signature Page Renamed document to follow Document numbering SOP Removed Nitrobenzene-d5 from text. Added Benzo(e)pyrene to document. Updated Instrument Operating Parameter Updated Table References throughout document. Updated SOP references throughout document.	02July2010
S-GB-O-050-Rev.01	Updated SOP reference	07Jul2011

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STANDARD OPERATING PROCEDURE

SEPARATORY FUNNEL EXTRACTION

REFERENCE METHODS: EPA Method 3510C

LOCAL SOP NUMBER:	S-GB-O-053-Rev.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-053-Rev.00
SOP TEMPLATE NUMBER:	SOT-ALL-O-003-Rev.00

LOCAL APPROVAL

Nils Melberg, Laboratory General Manager — Date: 07/07/11

Kate Grams, Laboratory Quality Manager — Date: 7/7/11

Chris Haase, Department Manager — Date: 7/7/11

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 for extracting non-volatile and semi-volatile organic compounds from aqueous samples in a separatory funnel while meeting the requirements specified in EPA method 3510C.

2. Summary of Method

2.1. A measured volume of sample, usually about 1 liter, is serially extracted with solvent in a separatory funnel. Some extractions also require the monitoring and adjusting of the pH of the sample. The extract is separated from the sample and is concentrated, followed by cleanup or analysis.

3. Scope and Application

3.1. This procedure is for extracting water insoluble or slightly water soluble organic compounds from aqueous samples using methylene chloride as the extraction solvent.

3.2. This procedure is applicable for the extraction of semi-volatile compounds (BNAs), polynuclear aromatic hydrocarbons (PAHs), pesticides, PCBs and total petroleum hydrocarbons (TPH).

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of separatory funnel equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.4. Analytical SOPs using this extraction procedure include:

S-GB-O-049	Determination of Semivolatile Organics by GC/MS
S-GB-O-050	Determination of Semivolatile Organics by GC/MS (Selective Ion Monitoring)
S-GB-O-026	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography
S-GB-O-027	Analysis of Organochlorine Pesticides by Gas Chromatography
S-GB-O-023	Total Petroleum Hydrocarbons

4. Interferences

4.1. Solvents, reagents and glassware can all contribute to compound artifacts or raised baselines; both conditions that can affect chromatography. Analyzing method blanks is therefore crucial in determining the presence of contaminants.

4.2. Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.

4.3. Extracts that exhibit interferences can be run through a cleanup procedure (see EPA method 3600). Before using a cleanup method, the analyst should run a series of calibration standards through the procedure to ensure that the elution order of compounds remains the same and that no new interferent has been introduced by the cleanup method. Cleanup SOPs that can be used for this extraction procedure include:

S-GB-O-032	Gel Permeation Chromatography
S-GB-O-034	Sulfuric Acid Cleanup
S-GB-O-035	Mercury Cleanup for the Removal of Sulfur from PCB Samples
S-GB-O-036	Florisil Cleanup for PCBs
S-GB-O-037	Florisil Cartridge Cleanup
S-GB-O-038	Silica Gel Cleanup for Organic Analysis
S-GB-O-039	Copper Cleanup for the Removal of Sulfur from PCB Samples

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.

Methylene chloride creates excessive pressure very rapidly when shaken in an enclosed apparatus, as directed in this method. The shaker funnels used in conjunction with the extraction vessel are designed to automatically vent excess pressure created by methylene chloride. As a result, the extraction mixer should be operated in a hood to avoid exposure of the technician to solvent vapors.

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. All distillations should be conducted under a fume hood.

6. Definitions

Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid (preferably 1L widemouth).	None	4 ± 2°C	Samples must be extracted within 7 days of collection and must be analyzed within 40 days of extraction.
TCLP and SPLP	One (1) 1-Liter Amber Glass	None	4 ± 2°C	TCLP and SPLP Leachates must be solvent extracted within 7 days of the completion of the tumbling process.

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
Lab-Line Automated Separatory Funnel Extractor	Barnsteadt	Model #1600	
Concentrator Water Bath	Fisher	S-EVAP	
Turbo Vaps II	Zymark	Zw 8002	

8.2. General Supplies

Item	Vendor	Model / ID	Catalog #	Description
Self-venting Separatory Funnels	Fisher		NC9802352	Glass, able to hold 2L; with PTFE stopcocks and Teflon lids
Analytical Balance	Ohaus	Model # AR 5120		Capable of reading to 0.01g
Glass beakers	Fisher			
Glass Autosampler Vials	MG Scientific	V300-3 / V300-20N		2.0 mL with Teflon-lined crimp
Micro-syringes	Hamiliton			10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL,
Glass funnels	Fisher			
Whatman #41 Filters	Fisher	1441-185		185 mm
Glass Stirring Rods	Fisher			
Kuderna-Danish flasks	HGF Scientific	192006-03		500mL with ground glass joints
Kuderna Danish concentrator tubes	HGF Scientific	192010-12		10mL, graduated, with ground glass fitting
Snyder columns	HGF Scientific	192002-DP1/ 192002-M2B		2-ball and 3-ball varieties
Keck clips to hold KD glassware together	Fisher	NO19	05-880D	Keck Clip #19
Boiling Chips	Fisher	09-191-20		450 g
pH test strips	CTL		921-10	Wide Range 0-14
Dispensing Pipettes	Fisher			Adjustable over range of 50 .0 to 100.0 mL, one for each solvent
Wash bottles, PTFE	Fisher			One for each solvent
Powder funnels	Fisher			
Glass wool 8 micron	Fisher		551940	
Disposable Pasteur pipettes	MG	P200-1 5 3/4” P200-2 9”		

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	De-ionized water	ASTM Type II water
Sodium Sulfate	Anhydrous, granular, baked at 400°C for 4 hours before use.	MG Scientific / catalog # 8024-24
Methylene Chloride	Extraction solvent	MG Scientific / catalog # 9266-8P
Acetone	Extraction solvent	MG Scientific / catalog # 010-4
Hexane	Exchange solvent (pesticides and PCBs)	MG Scientific / catalog # 9262-8P
Sulfuric Acid, Conc.	Concentrated	Fisher / catalog # 9681-33
Sulfuric Acid Solution 1:1	Add 400 mL conc. Sulfuric Acid to 400 mL Reagent Water	
Sodium Hydroxide pellets		Fisher / catalog # 5318-3
Sodium Hydroxide Solution (10N)	Dissolve 400g sodium hydroxide pellets into 1L of reagent water	

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogates are added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains all target analytes.	Same solution can be used for the LCS and MS/MSD

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Surrogate Standards

Standard	Concentration	Manufacturer	Catalog #	Storage
Restek B/N Surrogate Mix for PAHs	1000 ug/mL	Restek	31002	Refrigerator 4 +/- 2°C
Restek B/N Surrogate Mix for BNAs	5000 ug/mL	Restek	31082	Refrigerator 4 +/- 2°C
Restek Acid Surrogate Mix for BNAs	7500 ug/mL	Restek	31083	Refrigerator 4 +/- 2°C
Equity Pesticide Surrogate Spike Mix	200µg/mL each TMX and DCB in Acetone	Supelco or Restek	5-05935 or 32457	Refrigerator 4 +/- 2°C
O-Terphenyl Surrogate	10000 ug/mL	Restek	31097	Refrigerator 4 +/- 2°C

LCS and MS Standards

Standard	Concentration	Manufacturer	Catalog #	Storage
PAH Standard	500 ug/mL	Accustandard	M-160-FL-R-5X	Refrigerator 4 +/- 2°C
BNA Standard - 70 Component Custom LCS Mix	200 ug/mL	Supelco	861389-U	Refrigerator 4 +/- 2°C
BNA Standard - n-nitrosodiphenylamine	5000 ug/mL	Supelco	46702-U	Refrigerator 4 +/- 2°C
Pesticide Standard Mix A	5 – 50µg/mL in Hexane:Toluene (98:2)	Supelco or equivalent	4-8796	Refrigerator 4 +/- 2°C
Pesticide Standard Mix B	5 – 50µg/mL in Hexane:Toluene (99:1)	Supelco or equivalent	4-8196	Refrigerator 4 +/- 2°C
Aroclor 1260	1000µg/mL in isooctane	Supelco or equivalent	4-4809	Room Temperature
Aroclor 1016*	1000µg/mL in isooctane	Supelco or equivalent	4-8097	Room Temperature °C
Toxaphene Standard	1000µg/mL in Hexane	Restek Corporation or equivalent	32005	Refrigerator 4 +/- 2°C
Diesel Fuel #2	50000µg/mL	Restek	32158	Room Temperature

*South Carolina State Requirement – Both 1016 and 1260 must be spiked in all LCS, MS and MSD

9.2.3. Preparation Procedures

PAHs by SW-846 8270C-SIM

- **Working Spiking Standard Preparation**

Table 9.4 Preparation of PAH LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
PAH Working Spike Solution	PAH Standard	200µL	500mL of methylene chloride	0.2µg/mL	1000µL

- **Working Surrogate Standard Preparation**

Table 9.5 Preparation of PAH Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Working PAH Surrogate Stock Solution	Restek B/N Mix, Cat. # 31082	100µL	500mL of methylene chloride	0.2µg/mL	1000 µL

BNAs by SW-846 8270C

- **Working Spiking Standard Preparation**

Table 9.6 Preparation of BNA LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
BNA Working Spike Solution	BNA Standard - 70 Component Custom LCS Mix	250µL	250µL	50µg/mL	250µL
	BNA Standard - n-nitrosodiphenylamine	10µL	10µL	50µg/mL	10µL

- **Working Surrogate Standard Preparation**

Table 9.7 Preparation of BNA Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
BNA Surrogate Spike Solution	Restek Acid Surrogate Mix for BNAs	5000µL	500mL of Methylene chloride	75µg/mL Acids	1.0 mL
	Restek B/N Surrogate Mix for BNAs	5000µL		50µg/mL Base/Neutrals	

Pesticide by SW-846 8081

- **Working Spiking Standard Preparation**

Table 9.8 Preparation of Pesticide LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide Matrix Spike Solution	Pesticide Standard Mix A and Mix B	4000µL of each	50mL of Acetone	0.4 – 4.0µg/mL	1000µL
Toxaphene Matrix Spike Solution	Toxaphene Standard	5000µL	100mL of Acetone	50µg/mL	800µL

- **Working Surrogate Standard Preparation**

Table 9.9 Preparation of Pesticide Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide/PCB Surrogate Solution	Equity Pesticide Surrogate Spike Mix	5000µL	500mL of Acetone	2.0µg/mL	500µL

PCB by SW-846 8082

- **Working Spiking Standard Preparation**

Table 9.10 Preparation of PCB LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
PCB Matrix Spike Solution	Aroclor 1260	1000µL	200mL of Acetone	5.0µg/mL	1000µL
PCB Matrix Spike Solution	Aroclor 1016*	1000µL	200mL of Acetone	5.0µg/mL	1000µL

*South Carolina State Requirement – Both 1016 and 1260 must be spiked in all LCS, MS and MSD samples.

- **Working Surrogate Standard Preparation**

Table 9.11 Preparation of Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide/PCB Surrogate Solution	Equity Pesticide Surrogate Spike Mix	5000µL	500mL of Acetone	2.0µg/mL	500µL

TPH by SW-846 8015B

- **Working Spiking Standard Preparation**

Table 9.12 Preparation of TPH LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Working Diesel Spike Solution	Restek Diesel Fuel #2	2mL	100mL of Methylene Chloride	1000µg/mL	500µL

- **Working Surrogate Standard Preparation**

Table 9.13 Preparation of Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
TPH Surrogate Solution	O-Terphenyl Surrogate	2500µL	250mL of Methylene Chloride	100µg/mL	500µL

10. Calibration

10.1. Not applicable to this SOP.

11. Procedures

11.1. See the latest revision of Pace SOP S-GB-O-015, *Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics* for the specifics on glassware cleaning.

11.2. Inspect all required glassware to ensure it is clean and dry. Set up each extraction mixer with as many as four, 2L shaker funnels, including caps and stopcocks; assemble 500mL KD apparatus; prepare water funnels with glass wool and sodium sulfate; and pre-rinse all glassware with methylene chloride.

11.3. Check the pH of any sample aliquots that will be analyzed for Methods 8270, 8081, 8082, or 8015 by removing a few drops with a disposable pipette for application to pH strips. Record result.

11.4. Mark the sample level on the container, pour the entire contents into separatory funnel and adjust pH accordingly. Add the appropriate working spike and working surrogate solutions as outlined in 11.6. Rinse the container with 60 mL of methylene chloride and pour into separatory funnel. Narrow-mouth bottles contain 1060 mL of sample. Wide-mouth bottles contain 1000 mL of sample. Containers that were not received full have the sample level meniscus marked as stated above, and after pouring of the sample into the separatory funnel and rinsing with methylene chloride, the container is filled to the mark with water. This water is then poured into a 1000 mL graduated cylinder and the sample volume recorded in the extraction log. If the sample container contains sediment on the bottom of the container, care is taken not to disturb and pour this material into the separatory funnel.

11.4.1. If high concentrations are anticipated, a smaller sample aliquot may be diluted to 1000 mL with nano pure water prior to extraction.

11.4.2. For TCLP and SPLP Leachates 200 mL of sample is measured with a Class A Graduated Cylinder and the contents poured into a separatory funnel. An additional 800 mL of Nanopure of Millipore water is also added to the separatory funnel. The volumes are recorded in the SW846 3510C Extraction Log. Add working spike and working surrogate solutions as outlined in 11.6. Rinse the graduated cylinder with 60 mL of methylene chloride and pour into the separatory funnel.

11.5. For each batch of 20 samples, or less, prepare two additional sample aliquots. One will serve as a MS and the second as a MSD. As an alternative if no sample in the batch has sufficient volume available for both a MS and MSD, prepare a second aliquot of a different sample for a Duplicate instead of the MSD. Also prepare 2 aliquots of reagent water to serve as the Method Blank and LCS.

11.5.1. For TCLP and SPLP samples a MS must be prepared for each sample matrix type. (For example a sand is determined to be a different matrix type than a sludge, and a separate matrix spike must be prepared for each one.) This information is recorded on the TCLP/SPLP extraction paperwork, and is provided the organic preparation personnel prior to extraction. The organic preparation personnel also records this information on the SW846 3510C Extraction Log.

11.6. Add Working Spike and Working Surrogate Solutions as follows:

11.6.1. PAH Analysis – Add 1.0 mL of 0.2 ug/mL Working Spike Solution (9.4) to the LCS, LCSD, MS, and MSD. Add 1.0 mL of the 0.2 ug/mL Working Surrogate Solution (9.5) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.

11.6.2. BNA Analysis – Add 250 uL of Supelco 70 Component Custom LCS Spike Mix, 200 ug/mL (9.6) and 10 uL of Supelco n-Nitrosodiphenylamine, 5000 ug/mL (9.6) to the LCS, LCSD, MS, and MSD. Add 1.0 mL of the Working Surrogate Solution, 75 ug/mL Acids, 50 ug/mL Base/Neutral (9.7) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.

11.6.3. Pesticide Analysis – Add 1.0 mL of 0.4-4.0 ug/mL Working Spike Solution (9.8) to the LCS, LCSD, MS, and MSD. Add 500 uL of the 2.0 ug/mL Working Surrogate Solution (9.9) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.

11.6.4. PCB Analysis – Add 1.0 mL of 5.0 ug/mL Working Spike Solution (9.10) to the LCS, LCSD, MS, and MSD. Add 500 uL of the 2.0 ug/mL Working Surrogate Solution (9.11) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.

11.6.5. Toxaphene Analysis – Add 800 uL of 50 ug/mL Working Spike Solution (9.8) to the LCS, LCSD, MS, and MSD. Add 500 uL of the 2.0 ug/mL Working Surrogate Solution (9.9) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.

11.6.6. TPH analysis – Add 500 µL of 1000 µg/mL Working Spike Solution (9.12) to the LCS, LCSD, MS, and MSD. Add 500 µL of 100 µg/mL Working Surrogate Solution (9.13) to each sample aliquot, LCS, LCSD, MB, MS, and MSD.

11.7. Adjust sample aliquots that will be analyzed for Method 8270 to pH <2 with 1:1 sulfuric acid solution. Adjust sample aliquots that will be analyzed for Methods 8081A or 8082 to pH >11 with 10N sodium hydroxide solution.

11.8. Add 60 mL of dichloromethane to each sample aliquot. If the entire content of a sample bottle is extracted, rinse the container with the dichloromethane before adding to the separatory funnel. Then pour tap water into the container to the mark, and measure the tap water volume with the graduated cylinder.

11.9. Seal the separatory funnel and set the Lab-Line extraction mixer to shake for 3.0 minutes at a speed of 30 cycles per minutes. After shaking allow the sample and extract to settle for at least 10 minutes.

11.10. If an emulsion is observed at the water-solvent interface with a volume more than 1/3 of the solvent layer, mechanical techniques should be used to complete separation. Mechanical techniques include stirring, filtration through s plug of glass wool, or centrifuging. If 80% of the solvent cannot be recovered, an alternative extraction method may be required.

11.11. Drain and dry the extract through powder funnels containing glass wool and anhydrous sodium sulfate. As the extract filters through the funnel, collect the extracts for BNA, PCB, Pesticide, and TPH analysis in a KD apparatus; and collect the extracts for PAH analysis in a Turbovap tube. Rinse the sodium sulfate with additional methylene chloride.

11.12. Add an additional aliquot of dichloromethane to the separatory funnel and repeat the operations in Sections 11.9 to 11.11 twice, for a total of three extractions.

11.13. For samples to be analyzed by Method 8270C and the target analyte list includes basic compounds, adjust the aliquot to pH $\geq$ 11 with 10 N sodium hydroxide solution and repeat the operations in Sections 11.9 to 11.12, combining all extracts in the same concentrator tube.

11.14. Concentration

11.14.1. Kuderna-Danish Method

11.14.1.1. Add 1-3 boiling chips to each flask and attach a Synder column

11.14.1.2. Place the apparatus on the water bath with the receiver tube partially immersed in the water. The temperature of the water bathe is 70-75°C. The balls in the Synder column should actively chatter, but not flood with solvent through the concentration process.

11.14.1.3. Remove the K-D from the water bath when the extract volume is 3-5 mL. After the unit has cooled, remove the Synder column. Rinse the flask with dichloromethane before removing the Receiver tube.

11.14.2. Turbo Vap® Method (Dry-Vap)

11.14.2.1. Place the concentrator receiver into the evaporator unit. Cover with a piece of aluminum foil held on with a rubber band to avoid cross contamination. A disposable pipette is used to create a hole in the aluminum foil to allow the solvent to escape.

11.14.2.2. Operate the unit according to the manufacturer's instructions until the extract volume is 1-5 mL. Remove the receiver and allow to cool. Rinse down the sides of the flask.

11.15. Solvent – Exchange – Extracts to be analyzed by Methods 8081A and 8082 must be exchanged to hexane.

11.16. Finalizing

11.16.1. Further concentrate extracts and the concentrator rinses to approximately ½ of the target final volume by a gentle stream of dry nitrogen using a blow down manifold or N-EVAP. BNA final volume is then archived by putting on hot plate water bath. Samples are brought up to 1 mL final volume in a syringe. PAH Extracts concentrated by the Turbo Vap® method are bought to final volume in the Turbo Vap tube. Extracts can also be concentrated by adding a fresh boiling chip, fitting the K-D receiver tube with a micro Synder column, and returning the tubes to the heated water bath.

11.16.2. Quantitatively transfer the target final extract volume of 1.0 mL to a 2.0-mL vial for PAH and BNA analyses. Quantitatively transfer the target final extract volume of 1 mL to a 1 mL vial for TPH analyses. Quantitatively transfer the target final extract volume of 10 mL to a 10-mL vial for Pesticides, PCB, and Toxaphen analyses. Final extract volumes may be adjusted to meet client requirements.

11.16.3. Dilute the final extract to volume with the final solvent. The TPH extract is ready for analysis without diluting to volume.

11.16.4. If the final extract will not be analyzed immediately, it must be stored at less than -10°C or at 4°C, as required by the SOP for the determinative procedure.

11.16.5. If the extract cannot be concentrated to the volumes specified above, it should be diluted or subjected to an approved extract cleanup procedure.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples or 12-hour window (whichever is most frequent)	Target analytes must be less than reporting limits	Re-extract and re-analyze associated samples if blank result is greater than RL
Laboratory Control Sample (LCS)	All target analytes	One per batch of up to 20 samples	See analytical SOPs	Re-extract and re-analyze a new LCS if original LCS is outside acceptance limits. **Exceptions:** 1) If LCS recovery is > QC limits and target analytes are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)/Matrix Spike Duplicate	All target analytes	One set per batch of up to 20 samples	See analytical SOPs	No corrective actions necessary. If LCS recovery is in range, the system is considered valid and the out-of-control MS/MSDs are footnoted appropriately by the analyst.
Surrogates	All applicable surrogate compounds	Added to each sample, blank and QC sample	See analytical SOPs	Surrogates above limits but no hits- report samples with footnote. Surrogate limits above limits but with hits- re-extract if possible or report as biased high. Surrogates below limits: re-extract if possible or report as biased low.

12.2. LCS equation

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3. MS/MSD equation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

RPD calculations:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13. Method Performance

13.1. **Method Detection Limit (MDL) Study**: An MDL study must be conducted every 12 months per S-GB-Q-020, *Determination of the LOD and LOQ* for each matrix 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*.

14. Pollution Prevention and Waste Management

14.1. Procedures for handling waste generated during this analysis are addressed in ALL-S-002, Waste Handling.

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. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3510C "Separatory Funnel Extraction".

15.2. Pace Analytical Quality Manual; latest revision.

15.3. NELAC Standard; Quality Systems section; latest revision.

15.4. S-GB-I-025 *TCLP Toxicity Characteristic Leaching Procedure*

15.5. S-GB-O-049 *Determination of Semi-Volatile Organics by GC/MS*

15.6. S-GB-O-050 *Determination of Semi-Volatile Organics by GC/MS (Selective Ion Monitoring)*

15.7. S-GB-O-026 *Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography*

15.8. S-GB-O-027 *Analysis of Organochlorine Pesticides by Gas Chromatography*

15.9. S-GB-O-028 *Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes*

15.10. S-GB-O-032 *Gel Permeation Chromatography*

15.11. S-GB-O-034 *Sulfuric Acid Cleanup*

15.12. S-GB-O-015 *Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics*

15.13 S-GB-O-023 *Total Petroleum Hydrocarbons*

16. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

16.1. Not applicable to this SOP.

17. Revisions

Document Number	Reason for Change	Date
SOT-ALL-O-003-rev.00	1. Restructured document format to new corporate template. 2. Updated formatting and Responsibilities and Distribution section	16Nov2006
S-ALL-GB-O-003-Rev.00	First issue as Corporate Template	10Jun2008
S-GB-O-053-Rev.00	Updated cover page to Periodic Review Renamed SOP to be consistent with SOP: S-ALL-Q-003 *Document Numbering* Section 7.0: Included TCLP/SPLP Preservation/Hold time Section 11.4: Added spiking/surrogate solutions must be added to sample prior to adding Methylene Chloride Section 11.4.1 and 11.4.2 Included TCLP/SPLP Extraction Procedure information. Updated table in section 8.1, 8.2, 9.1, 9.3,and 9.4 Added TPH standards and procedures	04Jan2010
S-GB-O-053-Rev.01	Updated SOP reference S-ALL-GB-O-001 to S-GB-O-049. Updated SOP reference S-ALL-GB-O-008 to S-GB-O-050 Section 13.1: Updated SOP to S-GB-Q-020: Determination of the LOD and LOQ.	07Jul2011

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STANDARD OPERATING PROCEDURE

ULTRASONIC EXTRACTION

REFERENCE METHODS: EPA Method 3550

LOCAL SOP NUMBER:	S-GB-O-054-Rev.00
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	ALL-O-005-Rev.0, S-ALL-GB-O-005-Rev.00, S-GB-O-029-Rev.1
SOP TEMPLATE NUMBER:	SOT-ALL-O-005-Rev.00

LOCAL APPROVAL

Nils Melberg, Laboratory General Manager — Date: 3/18/10

Kate Grams, Laboratory Quality Manager — Date: 3/18/10

Glen Coder, Department Manager — Date: 3/18/10

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 The purpose of this SOP is to provide a laboratory specific procedure for extracting non-volatile and semi-volatile organic compounds from solid samples, such as soils, sludges, and other wastes using an ultrasonic extractor (or sonicator) while meeting the requirements specified in EPA method 3550. Extracts extracted by this method are analyzed by SW-846 8081A/8081B and SW-846 8082/8082A following the latest revisions of Pace's SOPs S-GB-O-026 Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography, and S-GB-O-027 Analysis of Organochlorine Pesticides by Gas Chromatography.

2. SUMMARY OF METHOD

2.1 A 30g aliquot of sample is weighed and mixed with anhydrous sodium sulfate to form a free-flowing powder mixture. This mixture is solvent-extracted three times using ultrasonic extraction. The extract is separated from the sample and is concentrated, followed by cleanup or analysis. A smaller aliquot of sample may be used if high amounts of contaminants are expected or if problems with the matrix prohibit the use of 30g.

3. SCOPE AND APPLICATION

3.1 This procedure is for extracting water insoluble or slightly water soluble organic compounds from soils or other solid waste materials.

3.2 This procedure is applicable for the extraction of pesticides and PCBs.

3.3 This method was incorporated using methylene chloride as the extraction solvent. Certain clients may require other solvent systems.

3.4 This method has not been validated by the EPA for the extraction of organophosphorus pesticides due to breakdown of these compounds during extraction.

3.5 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ultrasonic extraction equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. INTERFERENCES

4.1 Solvents, reagents and glassware can all contribute to compound artifacts or raised baselines; both conditions that can affect chromatography. Analyzing method blanks is therefore crucial in determining the presence of contaminants.

4.2 Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.

4.3 Extracts that exhibit interferences can be run through a cleanup procedure (see EPA method 3600). Before using a cleanup method, the analyst should run a series of calibration standards through the procedure to ensure that the elution order of compounds remains the same and that no new interferent has been introduced by the cleanup method.

5. SAFETY

5.1 Standards and Reagents

5.1.1 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

5.2.1 Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

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.

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
Solid	Glass container with Teflon-lined lid (preferably 8oz jar).	None	4 ± 2°C	Samples must be extracted within 14 days of collection and must be analyzed within 40 days of extraction.

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	Serial Number	Description / Comments
Ultrasonic Extractor	Misonix Inc	Model # S4000	5090306	Equipped with dual ¾ inch horns
Ultrasonic Extractor	Misonix Inc.	Model # S3000	R1941	Equipped with dual ¾ inch horns

8.2 General Supplies

Item	Vendor	Model / ID	Description
Analytical Balance	Ohaus	Model # AR 5120	Capable of reading to 0.01g
Glass beakers	Fisher	-	-
Glass Autosampler Vials	MG Scientific	V300-3 / V300-20N	2.0 mL with Teflon-lined crimp caps
Micro-syringes	Hamiliton (or equivalent)	-	10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL, as needed.
Glass funnels	Fisher	-	-
Whatman #41 Filters	Fisher	1441-185	185 mm
Spatulas	Fisher	-	-
Kuderna-Danish flasks	HGF Scientific	192006-03	500mL with ground glass joints
Kuderna Danish concentrator tubes	HGF Scientific	192010-12	10mL, graduated, with ground glass fitting
Snyder columns	HGF Scientific	192002-DP1/ 192002-M2B	2-ball and 3-ball varieties
Keck clips to hold KD glassware together	HGF Scientific	192002-DP4	Keck Clip #19
Water Bath Concentrators	-	-	8 place
Boiling Chips	Fisher	09-191-20	450 g

9. REAGENTS AND STANDARDS

9.1 Reagents

Table 9.1: Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	De-ionized water	ASTM Type II water
Sodium Sulfate	Anhydrous, granular, baked at 400°C for 4 hours before use.	MG Scientific / catalog # 8024-24
Methylene Chloride	Extraction solvent	MG Scientific / catalog # 9266-8P
Acetone	Extraction solvent	MG Scientific / catalog # 010-4
Hexane	Exchange solvent (pesticides and PCBs)	MG Scientific / catalog # 9262-8P
Ottawa sand	Mesh size 20-30, baked at 400°C for 4 hours before use.	Fisher / catalog # S23-50

9.2 Analytical Standards

9.2.1 **Definitions**

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogates are added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains all target analytes.	Same solution can be used for the LCS and MS/MSD

Table 9.3 Surrogate Standards

Standard	Concentration	Manufacturer	Catalog #	Storage
Equity Pesticide Surrogate Spike Mix	200µg/mL each TMX and DCB in Acetone	Supelco or equivalent	5-05935	Refrigerator 4 +/- 2°C

Table 9.4 LCS and MS Standards

Standard	Concentration	Manufacturer	Catalog #	Storage
Pesticide Standard Mix A	5 – 50µg/mL in Hexane:Toluene (98:2)	Supelco or equivalent	4-8796	Refrigerator 4 +/- 2°C
Pesticide Standard Mix B	5 – 50µg/mL in Hexane:Toluene (99:1)	Supelco or equivalent	4-8196	Refrigerator 4 +/- 2°C
Aroclor 1260	1000µg/mL in isooctane	Supelco or equivalent	4-4809	Refrigerator 4 +/- 2°C
Aroclor 1016*	1000µg/mL in isooctane	Supelco or equivalent	4-8097	Refrigerator 4 +/- 2°C
Toxaphene Standard	1000µg/mL in Hexane	Restek Corporation or equivalent	32005	Refrigerator 4 +/- 2°C

***South Carolina State Requirement – Both 1016 and 1260 must be spiked in all LCS, MS and MSD**

9.2.2 **Preparation Procedures**

Pesticide by SW-846 8081

9.3 Working Spiking Standard Preparation

Table 9.5 Preparation of LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide Matrix Spike Solution	Pesticide Standard Mix A and Mix B	4000µL of each	50mL of Acetone	0.4 – 4.0µg/mL	1000µL
Toxaphene Matrix Spike Solution	Toxaphene Standard	5000µL	100mL of Acetone	50µg/mL	800µL

9.4 Working Surrogate Standard Preparation

Table 9.6 Preparation of Surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide/PCB Surrogate Solution	Equity Pesticide Surrogate Spike Mix	5000µL	500mL of Acetone	2.0µg/mL	500µL

PCB by SW-846 8082

9.5 Working Spiking Standard Preparation

Table 9.7 Preparation of LCS and MS Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
PCB Matrix Spike Solution	Aroclor 1260	1000µL	200mL of Acetone	5.0µg/mL	1000µL
PCB Matrix Spike Solution	Aroclor 1016*	1000µL	200mL of Acetone	5.0µg/mL	1000µL

***South Carolina State Requirement – Both 1016 and 1260 must be spiked in all LCS, MS/D samples.**

9.6 Working Surrogate Standard Preparation

Table 9.8 Preparation of surrogate Standard Solutions

Spike	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Amount Spiked into a Sample Aliquot
Pesticide/PCB Surrogate Solution	Equity Pesticide Surrogate Spike Mix	5000µL	500mL of Acetone	2.0µg/mL	500µL

10. CALIBRATION

10.1 The ultrasonic extractors used by the laboratory are self-tuning.

11. PROCEDURES

11.1 The ultrasonic extractor must be in proper working condition to achieve maximum extraction efficiency.

11.1.1 The extractor must have a minimum of 300 watts of power and be equipped with appropriate size horns.

11.2 Sonicator Setup

11.2.1 Make sure the generator is turned off.

11.2.2 Make sure the threads and touching surfaces of the converter and the horn are cleaned, polished, and oil free.

11.2.3 Gently screw the horn into the converter until the horn and converter meet. Do not touch the horn to the bench top or any other object.

11.2.4 Using the horn-tightening wrench, apply pressure to secure the horn snuggly.

11.2.5 After the horn is attached vertically secure the converter into the ring stand in the sound box.

11.3 Sonicator Maintenance

11.3.1 The horns must be properly maintained per manufacturer's instructions including cleaning prior to use and checking periodically for signs of wear.

11.3.2 Proper care of horns and tips is essential for good performance and long service life. Cleanliness of threads and mating faces is critical to successful operation.

11.3.3 Methylene Chloride is recommended for cleaning studs and threaded holes.

11.3.4 All threaded joints must mate smoothly by hand. Never attempt to force a threaded joint with wrenches before the mating faces come snuggly into contact without resistance. A bad joint is indicated by severe heating at the joint, high or off-scale meter readings or inability to tune.

11.3.5 Studs are hardened alloy steel; should a stud break off, consult the factory for repair or replacement.

11.4 The horn tip should always be positioned just below the surface of the extraction solvent but above the actual sample and the three extractions should be performed in the pulse mode of the device.

11.5 The samples must be properly prepared by mixing with sodium sulfate to form a free-flowing sample prior to the addition of the extraction solvent. Proper mixing of a sample is evidenced by very active mixing when the ultrasonic pulse is activated.

11.6 Please see latest revision of Pace SOP S-GB-O-015 *Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics* for specifics on glass-washing. Inspect all required glassware to ensure it is clean and dry. Pre-rinse glassware and discard the rinses as waste.

11.7 Homogenize each sample with a stainless steel spatula within the sample container without decanting the free standing liquid. Note on the extraction log if free-standing liquid was present in the sample.

11.8 Weigh a 30.0 g aliquot into a beaker. Add approximately 60 g of prepared anhydrous sodium sulfate to each sample aliquot and mix thoroughly to for a free-flowing mixture.

11.9 Weigh out two 30 g aliquot of prepared Ottawa Sand into beakers to serve as the method blank and LCS.

11.10 For each batch of 20 samples, or less, prepare two additional sample aliquots from the same sample. One will serve as the MS and the second as a MSD.

11.11 See Tables 9.6 and 9.8 for the amount of Working Surrogate Solution to add to each sample, method blank, LCS and MS/MSD. See Tables 9.5 and 9.7 for the appropriate Working Spike Solution, and amount of Working Spike Solution to add to each sample, method blank, LCS, and MS/MSD.

11.12 Add 60 mL of methylene chloride:actetone (80:20) mixture for each sonication of the sample aliquot. The sample must be completely covered by solvent. Client-specific requirements for toxaphene analysis call for the use of 60 mL of the solvent mixture hexane:acetone (1:1).

11.13 Place the bottom surface of the disrupter horn approximately ½" below the surface of the solvent, but above the solids.

11.14 Sonicate the sample-solvent mixture for three minutes at full power in the pulse mode (50% duty cycle).

11.15 Decant the extract and filter it through a glass soil funnel equipped with Whatman #41 filter paper and approximately 10 g prepared anhydrous sodium sulfate. As the extract

filters gravimetrically, collect it in a KD apparatus and rinse the sodium sulfate with additional methylene chloride. If the filtration takes more than 15 minutes, the technician may apply vacuum suction to assist in the process.

11.16 Repeat the extraction as described in Sections 11.12 – 11.15 for a total of three extractions, decanting the extract after each sonication.

11.17 Kuderna-Danish Concentration Method

11.17.1 Add 1-2 boiling chips to each flask and attach a Synder column.

11.17.2 Place the apparatus on the water bath with the receiver tube partially immersed in the water. The water bath temperature should be 98-100°C. After heating to temperature the balls in the Synder column should activity chatter, but not flood with solvent.

11.17.3 Remove the K-D from the water bath when the extract volume is 1-5 mL.

11.17.4 Extracts to be analyzed by SW-846 8081A/8081B and SW-846 8082/8082A must be exchanged to hexane. Add approximately 50 mL of hexane to the concentrated methylene chloride:acetone extract, and concentrate a second time as described in Section 11.17. Client request for Pesticides by SW-846 8081A may require gel permeation cleanup by the latest revision of Pace's SOP GB-O-032 *Gel Permeation Chromatography* before solvent exchange to hexane.

11.17.4.1 Remove the K-D from the water bath when the extract volume is 1-5 mL. After the unit has cooled, remove the Synder column. Rinse the flask with hexane before removing the receiver tube.

11.17.4.2 Extracts are brought to a final volume of 10 mL with hexane and transferred to a 15 mL vial with Teflon-lined cap.

11.17.4.3 Extracts for Toxaphene by SW-846 8081A/8081B and PCBs by SW-846 8082/8082A are ready for sulfuric acid cleanup following the latest revision of Pace's SOP S-GB-O-034 *Sulfuric Acid Cleanup.*

11.18 All extracts are transferred to 15 mL vials with Teflon-lined caps and stored at 4 +/- 2°C.

12. QUALITY CONTROL

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Prepared Ottawa Sand	One per 20 samples or 12-hour window (whichever is most frequent)	Target analytes must be less than reporting limits	Re-extract and re-analyze associated samples if blank result is greater than RL
Laboratory Control Sample (LCS)	Prepared Ottawa Sand with All target analytes	One per batch of up to 20 samples	See analytical SOPs	Re-extract and re-analyze a new LCS if original LCS is outside acceptance limits. **Exceptions:** 1) If LCS recovery is > QC limits and target analytes are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)/Matrix Spike Duplicate	All target analytes	One set per batch of up to 20 samples	See analytical SOPs	No corrective actions necessary. If LCS recovery is in range, the system is considered valid and the out-of-control MS/MSDs are footnoted appropriately by the analyst.
Surrogates	All applicable surrogate compounds	Added to each sample, blank and QC sample	See analytical SOPs	Surrogates above limits but no hits- report samples with footnote. Surrogate limits above limits but with hits- re-extract if possible or report as biased high. Surrogates below limits: re-extract if possible or report as biased low.

12.1 **LCS equation**

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.2 **MS/MSD equation**

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

RPD calculations

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13. METHOD PERFORMANCE

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months per S-ALL-Q-004, *Method Detection Limit Studies* most current revision, for each matrix 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* most current revision.

13.3 Method Modifications:

Method modifications for EPA method 3550 are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- 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.
- Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- Pace – Green Bay currently incorporates free standing water into the sample during the homogenization process.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in S-GB-O-006, *Waste Handling and Management* most current revision.

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 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3550 "Ultrasonic Extraction".

15.2 Pace Analytical Quality Manual; latest revision.

15.3 NELAC Standard; Quality Systems section; latest revision.

15.4 S-GB-O-026 *Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography*

15.5 S-GB-O-047 *Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by SW846 8082A*

15.6 S-GB-O-027 *Analysis of Organochlorine Pesticides by Gas Chromatography*

15.7 S-GB-O-028 *Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes*

15.8 GB-O-032 *Gel Permeation Chromatography*

15.9 S-GB-O-034 *Sulfuric Acid Cleanup*

15.10 S-GB-O-015 *Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics*

16. TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS, APPENDICES, ETC.

16.1 Not applicable to this SOP.

17. REVISIONS

Document Number	Reason for Change	Date
SOT-ALL-O-005-rev.00	Restructured document format to new corporate template.	16Nov2006
S-ALL-GB-O-005-Rev.00	First issue as Corporate Template	16Apr2008
S-GB-O-054-Rev.00	Updated cover page to Periodic Review. Renumbered document to follow corporate SOP: S-ALL-Q-003 Document Numbering. Removed Section 7 – Responsibilities and Distribution Renumbered document Section 9: Renumbered Spike/Surrogate Tables. Section 11.3: Updated sonicator maintenance to current practice. Removed notation of "retuning" , new Sonicators are self-tuning. Section 11.7, 13.3: Added language that outlines homogenization of free standing liquid in the sample. Updated references in 11.16 and 11.17 Updated 8.1 Instrumentation table. Included SW846 8081B and SW846 8082A throughout document.	17Mar2009

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STANDARD OPERATING PROCEDURE

Determination of Volatile Organics by GC/MS

Reference Methods: EPA Method SW-846 8260B with 5030B and 5035; and EPA Method 624

LOCAL SOP NUMBER:	S-GB-O-056-Rev.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-O-056-Rev.02
SOP TEMPLATE NUMBER:	TMP-ALL-O-002-Rev.01

LOCAL APPROVAL

Nils Melberg, Laboratory General Manager — Date: 06/29/11

Kate Grams, Laboratory Quality Manager — Date: 5/23/11

Jeff Bushner, Department Manager — Date: 05/24/2011

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Green Bay to determine the concentration of Volatile Organic Compounds (VOCs) in environmental samples. The laboratory utilizes purge-and-trap GC/MS and bases these documented procedures on those listed SW-846 Methods 5030B, 5035 and 8260B; and EPA 624.

2. Summary of Method

Volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot or extract by purging with helium. The purged analytes are collected in a trap. At the completion of the purge time, the trap is rapidly heated and back flushed with helium to drive out the trapped analytes. The analytes are transferred into the inlet of a capillary gas chromatography column. The carrier gas flow through the column is controlled and the temperature is increased according to a set program to achieve optimum separation of purged analytes. The mass spectrometer is operated in a repetitive scan mode. Analytes are identified by the GC/MS retention times and by a comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected primary ion relative to an internal standard against a calibration curve.

3. Scope and Application

3.1. This method is applicable to most organic compounds that have boiling points below 200 ºC and are insoluble or slightly soluble in water. Volatile water-soluble compounds may also be determined although quantitation limits are typically higher due to their hydrophilic properties (e.g. ketones, oxygenates). Please see: Table 10.1 Calibration standard compound concentrations, for a listing off analytes analyzed by SW-846 Methods 5030B, 5035 and 8260B and EPA 624.

3.2. This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interference with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.4. This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

4. Interferences

4.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the absorbent trap. The use of polytetrafluoroethylene (PTFE, Teflon) as thread sealants, tubing, or in flow controllers is highly recommended since other materials can be sources of contamination which may concentrate in the trap during the purging.

4.2. A common source of interfering contamination is carryover. This may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive action to this condition is rinsing the purging apparatus and sample syringes with two or more portions of organic free water between samples. Analyze one or more blanks to check for cross contamination prior to sample analysis.

4.3. Since methylene chloride and acetone are common laboratory solvents, special precautions must be taken. The volatiles analysis and sample storage area should be located as far as possible from areas where these solvents are used or stored. Where possible, the volatiles analysis and sample storage area should be served by a separate HVAC system and maintained under positive pressure to prevent intrusion of contaminants. Laboratory clothing previously exposed to methylene chloride fumes during extraction procedures can contribute to sample contamination.

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.

5.3. Equipment

Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones.

The purge and trap concentrator and autosampler use gas under pressure to purge samples and, in some cases, drive the robotic assemblies. These high pressures introduce the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

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.

6.1. **Run Sequence Log** – A logbook that lists all injections and analyses performed on a particular piece of equipment regardless of the use of the data collected from each analysis.

6.2. **Toxicity Characteristic Leaching Procedure (TCLP)** – An extraction procedure used to determine if a sample is acceptable for upland disposal. The extraction procedure is meant to simulate the leaching of contaminants under the environmental conditions typically found in a landfill.

6.3. **Tune Period** – The period after the BFB instrument tune check within which analyses may be performed.

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	Two (2) VOA vials	Acidified w/ 1:1 HCl (1-2 drops) to pH<2, no headspace *Note:* 2-CLEVE, Styrene, and Vinyl Chloride requires an unpreserved sample.	4 ± 2°C	Unpreserved: 7 days pH Preserved: 14 days
Low Level Aliquot Soil/Solid (non-aqueous)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) 5-g aliquots in vials with magnetic stir bar, 5.0 mL reagent water and 1.0 g sodium bisulfate as needed. *OR (alternative):* Two (2) EnCore, TerraCore or similar sampling tubes.	No preservation *OR* sodium bisulfate **Note:** If sample effervesces on contact with the preservative, the sodium bisulfate should be eliminated for that sample.	With sodium bisulfate: 4 ± 2°C Without preservation (including EnCore, TerraCore or similar): 4 ± 2°C for up to 48 hours before storing between -7°C and -20°C, inclusive, until analysis.	Unpreserved or not stored frozen: 48 hours Preserved with sodium bisulfate or stored frozen: 14 days
High Level Aliquot Soil/Solid (non-aqueous)	One (1) 10-g aliquot in vial with 10.0 mL purge and trap grade MeOH. *OR (alternative)* One (1) 10-g aliquot in empty vial	Methanol - if sample was collected in empty vial it must be transferred into 10 mL of purge & trap grade MeOH within 48 hours of collection	With methanol: 4 ± 2°C	Unpreserved: 48 hours Preserved with methanol: 14 days
TCLP Leachates	Tedlar bag or THREE (3) VOA vials.	Filled and capped to eliminate any headspace. Vials with bubbles larger than 5 mm should be discarded.	4 ± 2°C	14 days from end of leaching procedure

Table 7.2 – Trip Blank Requirements

Aqueous	**Low Level Aliquot Soil/Solid**	**High Level Aliquot Soil/Solid**
One (1) 40mL VOA vial w/ reagent DI water	One (1) 40mL VOA vial w/ 5mL sodium bisulfate (or reagent DI water) and magnetic stir bar	One (1) 40mL VOA vial w/ 5mL purge and trap grade MeOH

8. Equipment and Supplies

Table 8.1 Equipment

Analytical Instrument/Peripherals	EPIC Pro Name	Serial Number
HP 5890 Series II GC	40MSV1	3235A46437
HP 5972 MSD	40MSV1	3306A00154
Archon Autosampler	40MSV1	11948-696A
Tekmar 3000 Purge and Trap Concentrator	40MSV1	94104001
HP 6890 GC	40MSV2	US00032794
HP 5973 MSD	40MSV2	US90460014
Archon Autosampler	40MSV2	11768-695A
Tekmar 3000 Purge and Trap Concentrator	40MSV2	9535005
Agilent 6850 GC	40MSV3	CN10719006
Agilent 5975 MSD	40MSV3	US71216198
Dynatech PTA-30 Autosampler	40MSV3	11153-593E
Tekmar 3000 Purge and Trap Concentrator	40MSV3	94104006
HP 5890 Series II GC	40MSV4	3336A53831
HP 5972 MSD	40MSV4	3341A01395
Dynatech PTA-30 Autosampler	40MSV4	11647-1194
Tekmar 3000 Purge and Trap Concentrator	40MSV4	98197002
HP 6890 GC	40MSV5	US00025885
HP 5973 MSD	40MSV5	US82322154
Archon Autosampler	40MSV5	11894-396A
Tekmar 3000 Purge and Trap Concentrator	40MSV5	94067011
HP 5890 Series II GC	40MSV6	3336A59437
HP 5972 MSD	40MSV6	3341A01384
Archon Autosampler	40MSV6	11851-195A
Tekmar 3000 Purge and Trap Concentrator	40MSV6	95039012
HP 6890 GC	40MSV7	US00040707
HP 5973 MSD	40MSV7	US03950358
Archon Autosampler	40MSV7	11931-596A
Tekmar 3000 Purge and Trap Concentrator	40MSV7	93217001
Agilent Technologies 6850 Network GC System	40MSV8	CN1065-1003
Agilent Technologies 5975B MSD	40MSV8	US65115041
EST 8100 Autosampler	40MSV8	15048
Teledyne Tekmar 14-9800-100 Stratum Purge and Trap System	40MSV8	US07152010
HP 6890 GC	40MSV9	US00002310
HP 5973 MSD	40MSV9	US63810274
Archon Autosampler	40MSV9	11365-196A
Tekmar 3000 Purge and Trap Concentrator	40MSV9	98205001
Agilent 5975C GCMS	40MSVA	US10042016
Agilent 7890A GC	40MSVA	CN10031128
EST 8100 Autosampler	40MSVA	MS1003W022
Tekmar Stratum Purge and Trap Concentrator	40MSVA	US10055004

Table 8.2 Supplies

Supplies	Manufacturer	Vendor	Catalog #
10ul Gastight 1701	Hamilton	Fisher Scientific	14-815-1
25ul Gastight 1702	Hamilton	Fisher Scientific	14-815-29
50ul Gastight 1705	Hamilton	Fisher Scientific	14-824-30
100ul Gastight 1710	Hamilton	Fisher Scientific	13-684-100
250ul Gastight 1725	Hamilton	Fisher Scientific	13-684-102
500ul Gastight 1750	Hamilton	Fisher Scientific	13-684-106
1ml Gastight 1001	Hamilton	Fisher Scientific	14-824-25
5ml Gastight 1005	Hamilton	Fisher Scientific	13-684-96
50ml Gastight 1050	Hamilton	Fisher Scientific	14-815-195
DB-624 Capillary column, 20mX0.18 mm i.d.X1.0 um	J&W Scientific	VWR Scientific	121-1324
K-Trap, Vocarb3000, Tekmar3000	Supelco	Supelco	24920-U
Fritless 5-ml Sparge Tube	Supelco	Supelco	22780
IceBlue Septa, 11mm	Restek	Restek	22392
Single Gooseneck Injection port liners (4mm)	Restek	Restek	20799
Gold-plated inlet seals	Restek	Restek	21306
Viton O-rings	Restek	Restek	20377
0.4mm Vespel/Graphite ferrules	Restek	Restek	20211
GCMS Filaments	Agilent Technologies	Agilent Technologies	05972-60053
Stir Bar	Fisher Brand	Fisher Brand	14-511-60A
40 ml VOA vials	QEC	QEC	3112-40ml
10 ml volumetric	Kimax Brand	Fisher Scientific	10-212AA
25 ml volumetric	Kimax Brand	Fisher Scientific	10-212BB
50 ml volumetric	Kimax Brand	Fisher Scientific	10-212A
100 ml volumetric	Kimax Brand	Fisher Scientific	10-212B
200 ml volumetric	Kimax Brand	Fisher Scientific	10-212C
500 ml volumetric	Kimax Brand	Fisher Scientific	10-218D
Pasteur Pipettes	Fisher Scientific	Fisher Scientific	13-678-20A
Pipette bulb	Fisher Scientific	Fisher Scientific	14-065B
0.1-2.5 ml Repipettor	Brinkmann	Fisher Scientific	13-688-130
1-5 ml Repipettor	Brinkmann	Fisher Scientific	13-688-131
2-10 ml Repipettor	Brinkmann	Fisher Scientific	13-688-133
5-25 ml Repipettor	Brinkmann	Fisher Scientific	13-688-134
1.8 ml amber vials & caps	Restek	Restek	24637
10 ml graduate cylinder	Kimex Brand	Fisher Scientific	08-554B “to deliver”
40 ml VOA vials HCl preserved	QEC	QEC	3112-40HCL
2 oz. jars with Teflon lids	QEC	QEC	2114-0002
Spatulas	Fisher Scientific	Fisher Scientific	14-511-60A

9. Reagents and Standards

9.1. Reagents

Table 9.1 Reagents

Reagent	Conc.	Purity	Manufacturer	Vendor	Catalog #
Methanol	100%	Purge and Trap grade	Burdick & Jackson	VWR Scientific	232-1
Sodium Bisulfate	Granular	Certified grade	Fisher Scientific	Fisher Scientific	S-240-3
Helium gas		99.999%	Michigan Airgas	Michigan Airgas	
OFW		ASTM Type II	Barnstead-Nanopure	VWR Scientific	D8961
Ottawa Sand, 20-30 mesh		ASTM C190	Fisher Scientific	Fisher Scientific	S23-3

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for mass spectrometer tuning, initial calibration, calibration verification standards, second source verification, internal standards, surrogates, and for preparing LCS, MS, and MSD samples. Table 9.2 describes the standards used. Table 9.3 lists the stock standards used. Table 9.4 lists the compounds in each stock standard.

Table 9.2 Standard Definitions

Standard	Description	Comments
Tune Standard	4-Bromofluorobenzene (BFB) solution used to verify ion response ratios prior to analysis	Must purge between 5 and 50ng
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the accuracy of the calibration curve.	For volatiles analysis, this may be used as the LCS if analyzed once every 20 samples.
Internal Standard	A solution added all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Pentafluorobenzene 1,4-Difluorobenzene Chlorobenzene-d5 1,4-Dichlorobenzene-d4
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to analysis.	Dibromofluoromethane Toluene-d8 4-Bromofluorobenzene
Spiking Standard	This solution contains all target analytes and should not be prepared from the same standards as the calibration standards.	For volatiles analysis, this can be used as the second source verification standard.

9.2.2. Stock Standards

Table 9.3 Stock Standards

Standard	Conc.	Purity	Manufacturer	Vendor	Catalog #
4-BFB Tuning Standard	5000 ug/mL	99%	Restek	Restek	30003-520
502.2 Calibration Mix#1	2000 ug/mL	99%	Restek	Restek	30042
502.2 Calibration Mix#1 – ICV	2000 ug/mL	99%	Accustandard	Accustandard	M-502B-10X
502.2 Cal 2000 Megamix	2000 ug/mL	99%	Restek	Restek	30431
502.2 Cal 2000 Megamix – ICV	2000 ug/mL	99%	Accustandard	Accustandard	M-502A-R-10X
Vinyl Acetate	Neat	99+%	Chem Service	Chem Service	F718
Vinyl Acetate	Neat	99+%	Aldrich Chemical	Aldrich Chemical	V150-3
VOA Calibration Mix #1	5000 ug/mL	99%	Restek	Restek	30006
VOA Calibration Mix #1 – ICV	5000 ug/mL	99%	Accustandard	Accustandard	CLP-022K-25X
Pace GB Custom Mix	2000 ug/mL	99+%	Restek	Restek	56230
Pace GB Custom Mix – ICV	2000 ug/mL	99%	Accustandard	Accustandard	S-13659
Pace GB Custom Mix – Cyclohexanes	10000 ug/mL	99+%	Restek	Restek	58314
Pace GB Custom Mix – Cyclohexanes – ICV	10000 ug/mL	99%	Accustandard	Accustandard	S-7134-R1-5X
Pace GB Custom Mix – NELAP	Various	99+%	Restek	Restek	553772
Pace GB Custom Mix – NELAP – ICV	Various	99%	Accustandard	Accustandard	S-13661
Pace GB Custom Mix – Acrolein	20000 ug/mL	99+%	Chem Service	Chem Service	F2
Pace GB Custom Mix – Acrolein – ICV	20000 ug/mL	99%	Accustandard	Accustandard	QM-8015B/5031-03-2X
Pace GB Custom Mix – 2-Chloroethylvinyl Ether	2000 ug/mL	99+%	Restek	Restek	30265
Pace GB Custom Mix – 2-Chloroethylvinyl Ether – ICV	2000 ug/mL	99%	Accustandard	Accustandard	M-601C-10X
Pace GB Custom Revised Volatiles Standard Mix	Various	98+%	Restek	Restek	560395
Pace GB Custom Appendix 9 Standard Mix – ICV	Various	98.9%	Accustandard	Accustandard	S-13660-R1
8260 Internal Standard	2500 ug/mL	99%	Restek	Restek	30173
8260 Surrogate Standard	2500 ug/mL	99%	Restek	Restek	30174
Cal 2000 Mix A/B	2000 ug/mL	99%	Restek	Restek	30438
VOA Cal Mix # 5	2000 ug/mL	99%	Restek	Restek	30010
502.2 Mix #1	2000 ug/mL	99%	Restek	Restek	30042
Michigan GRO Mix	2000 ug/mL	99%	Restek	Restek	30468

9.2.3. Standard Mixes

Table 9.4 Standard Mixes

Vendor	Standard Name	Catalog#	Compound list	Concentration (ug/mL)
Restek	**502.2 Cal Mix 1**	**30042**	Bromomethane	2000
			Chloroethane	2000
			Chloromethane	2000
			Dichlorodifluoromethane	2000
			Trichlorofluoromethane	2000
			Vinyl Chloride	2000
Accustandard	**502.2 Cal Mix 1 – ICV**	**M-502B-10X**	Bromomethane	2000
			Chloroethane	2000
			Chloromethane	2000
			Dichlorodifluoromethane	2000
			Trichlorofluoromethane	2000
			Vinyl Chloride	2000
Restek	**502.2 MegaMix**	**30431**	1,1,1,2-Tetrachloroethane	2000
			1,1,1-Trichloroethane	2000
			1,1,2,2-Tetrachloroethane	2000
			1,1,2-Trichloroethane	2000
			1,1-Dichloroethane	2000
			1,1-Dichloropropene	2000
			1,1-Dichlroethene	2000
			1,2,3-Trichlorobenzne	2000
			1,2,3-Trichloropropane	2000
			1,2,3-Trimethylbenzene	2000
			1,2,4-Trimethylbenzene	2000
			1,2-Dibromo-3-chloropropane	2000
			1,2-Dibromoethane	2000
			1,2-Dichlorethane	2000
			1,2-Dichlorobenzene	2000
			1,2-Dichloropropane	2000
			1,3,5-Trimethylbenzene	2000
			1,3-Dichlorobenzene	2000
			1,3-Dichloropropane	2000
			1,4-Dichlorobenzene	2000
			2,2-Dichloropropane	2000
			2-Chlorotoluene	2000
			4-Chlorotoluene	2000
			4-Isopropyltoluene	2000
			Benzene	2000
			Bromobenzene	2000
			Bromochloromethane	2000
			Bromodichloromethane	2000

Vendor	Standard Name	Catalog#	Compound list	Concentration (ug/mL)
			Bromoform	2000
			Carbon Tetrachloride	2000
			Chlorobenzene	2000
			Chloroform	2000
			cis-1,2-Dichloroethene	2000
			cis-1,3-Dichloropropene	2000
			Dibromochloromethane	2000
			Dibromomethane	2000
			Ethylbenzene	2000
			Hexachloro-1,3-butadiene	2000
			Isopropylbenzene	2000
			Methylene Chloride	2000
			m-Xylene	2000
			Naphthalene	2000
			n-Butylbenzene	2000
			n-Propylbenzene	2000
			o-Xylene	2000
			p-Xylene	2000
			sec-Butylbenzene	2000
			Styrene	2000
			tert-Butylbenzene	2000
			Tetrachloroethene	2000
			Toluene	2000
			trans-1,2-Dichloroethene	2000
			trans-1,3-Dichloropropene	2000
			Trichloroethene	2000
Accustandard	**502.2 MegaMix – ICV**	**M-502A-R-10X**	1,1,1,2-Tetrachloroethane	2000
			1,1,1-Trichloroethane	2000
			1,1,2,2-Tetrachloroethane	2000
			1,1,2-Trichloroethane	2000
			1,1-Dichloroethane	2000
			1,1-Dichloropropene	2000
			1,1-Dichlroethene	2000
			1,2,3-Trichlorobenzne	2000
			1,2,3-Trichloropropane	2000
			1,2,3-Trimethylbenzene	2000
			1,2,4-Trimethylbenzene	2000
			1,2-Dibromo-3-chloropropa	2000
			1,2-Dibromoethane	2000
			1,2-Dichlorethane	2000
			1,2-Dichlorobenzene	2000
			1,2-Dichloropropane	2000
			1,3,5-Trimethylbenzene	2000
			1,3-Dichlorobenzene	2000
			1,3-Dichloropropane	2000

Vendor	Standard Name	Catalog#	Compound list	Concentration (ug/mL)
			1,4-Dichlorobenzene	2000
			2,2-Dichloropropane	2000
			2-Chlorotoluene	2000
			4-Chlorotoluene	2000
			4-Isopropyltoluene	2000
			Benzene	2000
			Bromobenzene	2000
			Bromochloromethane	2000
			Bromodichloromethane	2000
			Bromoform	2000
			Carbon Tetrachloride	2000
			Chlorobenzene	2000
			Chloroform	2000
			cis-1,2-Dichloroethene	2000
			cis-1,3-Dichloropropene	2000
			Dibromochloromethane	2000
			Dibromomethane	2000
			Ethylbenzene	2000
			Hexachloro-1,3-butadiene	2000
			Isopropylbenzene	2000
			Methylene Chloride	2000
			m-Xylene	2000
			Naphthalene	2000
			n-Butylbenzene	2000
			n-Propylbenzene	2000
			o-Xylene	2000
			p-Xylene	2000
			sec-Butylbenzene	2000
			Styrene	2000
			tert-Butylbenzene	2000
			Tetrachloroethene	2000
			Toluene	2000
			trans-1,2-Dichloroethene	2000
			trans-1,3-Dichloropropene	2000
			Trichloroethene	2000
Restek	**Vinyl Acetate**	**30216**	Vinyl Acetate	2000
Aldrich	**Vinyl Acetate – ICV**	**V150-3**	Vinyl Acetate	Neat
Restek	**VoaCalMix 1**	**30006**	2-Butanone	5000
			2-Hexanone	5000
			4-Methyl-2-pentanone	5000
			Acetone	5000

Vendor	Standard Name	Catalog#	Compound list	Concentration (ug/mL)
Accustandard	**VoaCalMix 1 – ICV**	**CLP-022K-25X**	2-Butanone	5000
			2-Hexanone	5000
			4-Methyl-2-pentanone	5000
			Acetone	5000
Restek	**Pace-GB Custom**	**56230**	1 1 2-TRICHLOROTRI-FLUOROETHANE	2000
	Mix		1 2 3-TRIMETHYLBENZENE	2000
			2 3-DICHLOROPROPENE	2000
			2-METHYLNAPHTHALENE	2000
			ACRYLONITRILE	2000
			ALLYL CHLORIDE	2000
			CARBON DISULFIDE	2000
			CIS-1 4-DICHLORO-2-BUTENE	2000
			DICHLOROFLUOROMETHANE	2000
			DIETHYL ETHER	2000
			DIISOPROPYL ETHER	2000
			ETHYL METHACRYLATE	2000
			HEXACHLOROETHANE	2000
			IODOMETHANE	2000
			METHYL METHACRYLATE	2000
			METHYL T-BUTYL ETHER	2000
			N-HEXANE	2000
			TETRAHYDROFURAN	2000
			TRANS-1 4-DICHLORO-2-BUTENE	2000
Accustandard	**Pace-GB Custom**	**S-13659**	1 1 2-TRICHLOROTRI-FLUOROETHANE	2000
	Mix – ICV		1 2 3-TRIMETHYLBENZENE	2000
			2 3-DICHLOROPROPENE	2000
			2-METHYLNAPHTHALENE	2000
			ACRYLONITRILE	2000
			ALLYL CHLORIDE	2000
			CARBON DISULFIDE	2000
			CIS-1 4-DICHLORO-2-BUTENE	2000
			DICHLOROFLUOROMETHANE	2000
			DIETHYL ETHER	2000
			DIISOPROPYL ETHER	2000
			ETHYL METHACRYLATE	2000
			HEXACHLOROETHANE	2000
			IODOMETHANE	2000
			METHYL METHACRYLATE	2000
			METHYL T-BUTYL ETHER	2000
			N-HEXANE	2000
			TETRAHYDROFURAN	2000
			TRANS-1 4-DICHLORO-2-BUTENE	2000

Vendor	Standard Name	Catalog#	Compound list	Concentration (ug/mL)
Restek	**Pace-GB Custom**	**58314**	CYCLOHEXANE	10000
	Mix – Cyclohexanes		METHYL ACETATE	10000
			METHYLCYCLOHEXANE	10000
Accustandard	**Pace-GB Custom**	**S-7134-R1-5X**	CYCLOHEXANE	10000
	Mix – Cyclohexanes – ICV		METHYL ACETATE	10000
			METHYLCYCLOHEXANE	10000
Restek	**Pace-GB Custom**	**553772**	1 4-DIOXANE	100000
	Mix – Nelap		1-PROPANOL	100000
			AMYL ACETATE	2000
			BUTYL ACETATE	2000
			CYCLOHEXANONE	20000
			ETHYL ACETATE	20000
			ISOAMYL ACETATE	2000
			ISOPROPANOL	20000
			N-BUTANOL	100000
			N-HEPTANE	2000
			N-PROPYL ACETATE	2000
			ISOPROPYL ACETATE	2000
Accustandard	**Pace-GB Custom**	**S-13661**	1 4-DIOXANE	100000
	Mix – Nelap – ICV		1-PROPANOL	100000
			AMYL ACETATE	2000
			BUTYL ACETATE	2000
			CYCLOHEXANONE	20000
			ETHYL ACETATE	20000
			ISOAMYL ACETATE	2000
			ISOPROPANOL	20000
			N-BUTANOL	100000
			N-HEPTANE	2000
			N-PROPYL ACETATE	2000
			ISOPROPYL ACETATE	2000
Chem Service	**Pace-GB Custom**	**F2**	ACROLEIN	20000
	Acrolein			
Accustandard	**Pace-GB Custom**	**QM-8015B/5031-03-2X**	ACROLEIN	20000
	Acrolein – ICV			
Restek	**Pace-GB Custom**	**30265**	2-CHLOROETHYLVINYL ETHER	2000
	2-chloroethylvinyl ether			

Vendor	Standard Name	Catalog#	Compound list	Concentration (ug/mL)
Accustandard	**Pace-GB Custom**	**M-601C-10X**	2-CHLOROETHYLVINYL ETHER	2000
	2-chloroethylvinyl ether – ICV			
Restek	**Pace-GB Custom**	**560395**	ACETONITRILE	5000
	Revised Volatiles		CHLOROPRENE	5000
	Standard Mix		ETHANOL	200000
			ETHYL TERT BUTYL ETHER	2000
			ISOBUTANOL	20000
			METHACRYLONITRILE	5000
			PROPIONITRILE	5000
			TERT AMYL METHYL ETHER	2000
			TERT BUTYL ALCOHOL	10000
Accustandard	**Pace-GB Custom**	**S-13660-R1**	ACETONITRILE	5000
	Appendix 9 Standard		CHLOROPRENE	5000
	Mix		ETHANOL	200000
			ETHYL TERT BUTYL ETHER	2000
			ISOBUTANOL	20000
			METHACRYLONITRILE	5000
			PROPIONITRILE	5000
			TERT AMYL METHYL ETHER	2000
			TERT BUTYL ALCOHOL	10000

9.2.4. Standard Storage Conditions

Table 9.5 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	1. Concentrated reference solution purchased directly from approved vendor	1. Manufacturer's recommended expiration date for unopened ampulated standards. 2. Gas standards must be replaced 6 months after ampule is opened. 3. All other stock standards must be replaced 6 months after ampule is opened or on expiration date, whichever is sooner.	1. Manufacturer's recommended storage conditions 2. When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	1. Reference solutions prepared by dilutions of the stock solution	1. 6 months from preparation or the expiration date listed for the stock source, whichever is sooner. 2. 6 months for gas working standards. 3. Working solutions must be checked frequently and replaced if degradation or evaporation is suspected.	1. Store in amber vials with Teflon lined screw caps 2. Manufacturer's recommended storage conditions for stock source solution. 3. If stock source conditions conflict, store standard at coldest condition of any source.

9.2.5. Standard Sources

Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat materials are listed in Table 9.3. The recipes for preparing dilutions and all working and intermediate standards, and concentrations for all compounds are presented in Tables 9.6 and 9.7. All intermediate standards are prepared using purge and trap grade methanol and stored frozen in glass vials with Teflon lined screw caps or Mininert valves or as recommended by the standard manufacturer.

9.2.6. Preparation Procedures

Table 9.6 Intermediate Standard Preparation

Standard	Acronym	Concentration	Direction found in Section:
Level 1 Calibration Standard	VSTD001	1.0ug/L all compounds except as follows: 2.5 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 5.0 ug/L tert butyl alcohol; 10 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 50 ug/L 1,4-dioxane, 1-propanol, n-butanol; 100 ug/L ethanol	Table 9.7
Level 2 Calibration Standard	VSTD005	5.0 ug/L all compounds except as follows: 12.5 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 25 ug/L tert butyl alcohol; 50 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 250 ug/L 1,4-dioxane, 1-propanol, n-butanol; 200 ug/L ethanol.	Table 9.7
Level 3 Calibration Standard	VSTD020	20 ug/L all compounds except as follows: 50 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 100 ug/L tert butyl alcohol; 200 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 1000 ug/L 1,4-dioxane, 1-propanol, n-butanol; 2000 ug/L ethanol.	Table 9.7
Level 4 Calibration Standard	VSTD050	50 ug/L all compounds except as follows: 125 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 250 ug/L tert butyl alcohol; 500 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 2500 ug/L 1,4-dioxane, 1-propanol, n-butanol; 5000 ug/L ethanol	Table 9.7
Level 5 Calibration Standard	VSTD100	100 ug/L all compounds except as follows: 250 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/L tert butyl alcohol; 1000 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/L 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/L ethanol.	Table 9.7
Level 6 Calibration Standard	VSTD200	200 ug/L all compounds except as follows: 500 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 1000 ug/L tert butyl alcohol; 2000 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 10000 ug/L 1,4-dioxane, 1-propanol, n-butanol; 20000 ug/L ethanol.	Table 9.7

Standard	Acronym	Concentration	Direction found in Section:
Level 7 Calibration Standard	VSTD0005	0.50ug/L all compounds except as follows: 1.25 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 2.5 ug/L tert butyl alcohol; 5.0 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 25 ug/L 1,4-dioxane, 1-propanol, n-butanol; 50 ug/L ethanol	Table 9.7
Level 8 Calibration Standard	VSTD0004	0.40ug/L all compounds except as follows: 1.0 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 2.0 ug/L tert butyl alcohol; 4.0 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 20 ug/L 1,4-dioxane, 1-propanol, n-butanol; 40 ug/L ethanol	Table 9.7
Level 9 Calibration Standard (Water Curve only)	VSTD300	300 ug/L all compounds except as follows: 750 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 1500 ug/L tert butyl alcohol; 3000 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 15000 ug/L 1,4-dioxane, 1-propanol, n-butanol; 30000 ug/L ethanol.	Table 9.7
Independent Calibration Verification Standard	ICV050	50 ug/L all compounds except as follows: 125 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 250 ug/L tert butyl alcohol; 500 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 2500 ug/L 1,4-dioxane, 1-propanol, n-butanol; 5000 ug/L ethanol.	Table 9.7
Calibration Verification Standard	VSTD050	50 ug/L all compounds except as follows: 125 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 250 ug/l tert butyl alcohol; 500 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 2500 ug/L 1,4-dioxane, 1-propanol, n-butanol; 5000 ug/L ethanol	Table 9.7
Surrogate Standard	SS	50 ug/L	Table 9.7
Internal Standard	IS	50 ug/L	Table 9.7
Method Blank	MB	< Reporting Limit	Table 9.7
BFB	Vtunxxx	50 ng injection	Table 9.7
Matrix Spike/Blank Spike Stock Solution - TCL 3.4 List for Water and Low Level Soil Samples	MS/BS TCL 3.4 List Stock Solution for Water and Low Level Soil Samples	100 ug/mL for all compounds	Table 9.7

Standard	Acronym	Concentration	Direction found in Section:
Matrix Spike/Blank Spike Stock Solution - TCL 3.4 List for Methanol Preserved Soil Samples	MS/BS TCL 3.4 List Stock Solution for Methanol Preserved Soil Samples	500 ug/mL for all compounds	Table 9.7
Matrix Spike/Blank Spike Stock Solution - Full List for Water and Low Level Soil Samples	MS/BS Stock Solution – Full List for Water and Low Level Soil Samples	100 ug/mL all compounds except as follows: 1000 ug/mL Acrolein	Table 9.7
Matrix Spike/Blank Spike Stock Solution - Full List for Methanol Preserved Soil Samples	MS/BS Stock Solution – Full List for Methanol Preserved Soil Samples	500 ug/mL all compounds except as follows: 5000 ug/mL Acrolein	Table 97
MS/MSD – BS/BSD Spike for Water Samples	MS/MSD BS/BSD	50 ug/L for most compounds, Various for others.	Table 9.7
MS/MSD – BS/BSD Spike for Low Level Soil Samples	MS/MSD BS/BSD	50 ug/kg for most compounds, Various for others.	Table 9.7
MS/MSD – BS/BSD Spike for Methanol Preserved Soil Samples	MS/MSD BS/BSD	2500 ug/kg for most compounds, Various for others.	Table 9.7
Extraction Blank	EBLK	< Reporting Limit	

Table 9.7 Preparation of Standards

Standard	Acronym	Concentration of Intermediate	Reagents Used	Final Volume
4-Bromofluorobenzene	BFB	50 ug/mL	100µL of 5000 ug/mL BFB into methanol	10 mL
Internal Standard	IS	250 ug/mL	5000 µL of 2500 ug/mL IS into methanol	50 mL
Surrogate Standard	SS	250 ug/mL	5000 µL of 2500 ug/mL SS into methanol	50 mL
Internal/Surrogate Std.	IS/SS	250 ug/mL	5000 µL of 2500 ug/mL IS/SS into MeOH	50 mL
100 ug/mL Calibration Std. 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol.	CAL Stock Standard	100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol	1250 µL of 2000 ug/mL 502.2 Calibration Mix #1 1250 µL of 2000 ug/mL 502.2 Cal 2000 Megamix 1250 µL of 2000 ug/mL Vinyl Acetate 500 µL of 5000 ug/mL VOA Calibration Mix #1 1250 µL of 2000 ug/mL Pace GB Custom Mix 250 µL of 10000 ug/mL Pace GB Custom Mix – Cyclohexanes 1250 µL of 20000 ug/mL Pace GB Custom Mix – Acrolein	25 mL

			1250 µL of 2000 ug/mL Pace GB Custom Mix – 2-Chloroethylvinyl ether 1250 µL of Various Concentration Pace GB Custom Mix – NELAP 1250 µL of Pace GB Custom Revised Volatiles Standard Mix. Diluted into methanol	
100 ug/mL Calibration Std. 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol.	ICV Stock Standard	100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol.	1250 µL of 2000 ug/mL 502.2 Calibration Mix #1 – ICV 1250 µL of 2000 ug/mL 502.2 Cal 2000 Megamix – ICV 500 µL of 5000 ug/mL VOA Calibration Mix #1 – ICV 1250 µL of 2000 ug/mL Pace GB Custom Mix – ICV 250 µL of 10000 ug/mL Pace GB Custom Mix – Cyclohexanes – ICV 1250 µL of 20000 ug/mL Pace GB Custom Mix – Acrolein – ICV 1250 µL of 2000 ug/mL Pace GB Custom Mix – 2-Chloroethylvinyl ether – ICV 263 µL of 9520 ug/mL Vinyl Acetate 1250 µL of Various Concentration Pace GB Custom Mix – NELAP – ICV 1250 µL of Pace GB Custom Appendix 9 Standard Mix – ICV Diluted into methanol	25 mL
Vinyl Acetate	VA	9520 ug/mL	0.238 mg of neat Vinyl Acetate Diluted into methanol	25 mL
Level 1 Calibration Standard	VSTD001	1.0ug/L all compounds except as follows: 2.5 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 5.0 ug/L tert butyl alcohol; 10 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 50 ug/L 1,4-dioxane, 1-propanol, n-butanol; 100 ug/L ethanol.	Dilute 0.5 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	50 mL
Level 2 Calibration Standard	VSTD005	5.0 ug/L all compounds except as follows: 12.5 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 25 ug/L tert butyl alcohol; 50 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 250 ug/L 1,4-dioxane, 1-propanol, n-butanol; 200	Dilute 2.5 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water. If for a methanol curve, use 49	50 mL

		ug/L ethanol.	mL reagent water and 1 mL methanol.	
Level 3 Calibration Standard	VSTD020	20 ug/L all compounds except as follows: 50 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 100 ug/L tert butyl alcohol; 200 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 1000 ug/L 1,4-dioxane, 1-propanol, n-butanol; 2000 ug/L ethanol	Dilute 10 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	50 mL
Level 4 Calibration Standard	VSTD050	50 ug/L all compounds except as follows: 125 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 250 ug/L tert butyl alcohol; 500 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 2500 ug/L 1,4-dioxane, 1-propanol, n-butanol; 5000 ug/L ethanol	Dilute 25 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	50 mL
Level 5 Calibration Standard	VSTD100	100 ug/L all compounds except as follows: 250 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/L tert butyl alcohol; 1000 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/L 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/L ethanol	Dilute 50 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	50 mL
Level 6 Calibration Standard	VSTD200	200 ug/L all compounds except as follows: 500 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 1000 ug/L tert butyl alcohol; 2000 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 10000 ug/L 1,4-dioxane, 1-propanol, n-butanol; 20000 ug/L ethanol	Dilute 100 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	50 mL
Level 7 Calibration Standard	VSTD0005	0.50ug/L all compounds except as follows: 1.25 ug/L acetonitrile, chloroprene, propionitrile,	Dilute 0.25 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile;	50 mL

		methacrylonitrile; 2.5 ug/L tert butyl alcohol; 5.0 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 25 ug/L 1,4-dioxane, 1-propanol, n-butanol; 50 ug/L ethanol	500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	
Level 8 Calibration Standard	VSTD0004	0.40ug/L all compounds except as follows: 1.0 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 2.0 ug/L tert butyl alcohol; 4.0 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 20 ug/L 1,4-dioxane, 1-propanol, n-butanol; 40 ug/L ethanol	Dilute 0.20 uL 100 ug/mL all compounds except as follows: : 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	50 mL
Level 9 Calibration Standard (Water Curve only)	VSTD300	300 ug/L all compounds except as follows: 750 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 1500 ug/L tert butyl alcohol; 3000 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 15000 ug/L 1,4-dioxane, 1-propanol, n-butanol; 30000 ug/L ethanol	Dilute 150 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL Stock Standard into 50 mL reagent water.	50 mL
Independent Calibration Verification Standard	ICV050	50 ug/L all compounds except as follows: 125 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 250 ug/L tert butyl alcohol; 500 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 2500 ug/L 1,4-dioxane, 1-propanol, n-butanol; 5000 ug/L ethanol.	Dilute 25 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol ICV Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	50 mL
Calibration Verification Standard	VSTD050	50 ug/L all compounds except as follows: 125 ug/L acetonitrile, chloroprene, propionitrile, methacrylonitrile; 250 ug/L tert butyl alcohol; 500 ug/l acrolein, cyclohexanone, ethyl acetate, isopropanol,	Dilute 25 uL 100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol CAL	50 mL

		isobutanol; 2500 ug/L 1,4-dioxane, 1-propanol, n-butanol; 5000 ug/L ethanol.	Stock Standard into 50 mL reagent water. If for a methanol curve, use 49 mL reagent water and 1 mL methanol.	
Matrix Spike/Blank Spike Stock Solution - TCL 3.4 List for Water and Low Level Soil Samples	MS/BS TCL 3.4 List Stock Solution for Water and Low Level Soil Samples	100 ug/mL for all compounds	1250 µL of 2000 ug/mL Cal 2000 Mix A/B 1250 µL of 2000 ug/mL VOA Cal Mix #5 1250 µL of 2000 ug/mL 502.2 Cal. Mix #1 Dilute into methanol.	25 mL
Matrix Spike/Blank Spike Stock Solution - TCL 3.4 List for Methanol Preserved Soil Samples	MS/BS TCL 3.4 List Stock Solution for Methanol Preserved Soil Samples	500 ug/mL for all compounds	6250 µL of 2000 ug/mL Cal 2000 Mix A/B 6250 µL of 2000 ug/mL VOA Cal Mix #5 6250 µL of 2000 ug/mL 502.2 Cal. Mix #1 Dilute into methanol.	25 mL
Matrix Spike/Blank Spike Stock Solution - Full List for Water and Low Level Soil Samples	MS/BS Stock Solution – Full List for Water and Low Level Soil Samples	100 ug/mL all compounds except as follows: 250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 500 ug/mL tert butyl alcohol; 1000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 5000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 10000 ug/mL ethanol.	1250 µL of 2000 ug/mL 502.2 Calibration Mix #1 – ICV 1250 µL of 2000 ug/mL 502.2 Cal 2000 Megamix – ICV 500 µL of 5000 ug/mL VOA Calibration Mix #1 – ICV 1250 µL of 2000 ug/mL Pace GB Custom Mix – ICV 250 µL of 10000 ug/mL Pace GB Custom Mix – Cyclohexanes – ICV 1250 µL of 20000 ug/mL Pace GB Custom Mix – Acrolein – ICV 1250 µL of 2000 ug/mL Pace GB Custom Mix – 2-Chloroethylvinyl ether – ICV 263 µL of 9520 ug/mL Vinyl Acetate 1250 µL of Various Concentration Pace GB Custom Mix – NELAP – ICV 1250 µL of Pace GB Custom Appendix 9 Standard Mix – ICV Diluted into methanol	25 mL
Matrix Spike/Blank Spike Stock Solution - Full List for Methanol Preserved Soil Samples	MS/BS Stock Solution – Full List for Methanol Preserved Soil Samples	500 ug/mL all compounds except as follows: 1250 ug/mL acetonitrile, chloroprene, propionitrile, methacrylonitrile; 2500 ug/mL tert butyl alcohol; 5000 ug/mL acrolein, cyclohexanone, ethyl acetate, isopropanol, isobutanol; 25000 ug/mL 1,4-dioxane, 1-propanol, n-butanol; 50000 ug/mL ethanol.	6250 µL of 2000 ug/mL 502.2 Calibration Mix #1 – ICV 6250 µL of 2000 ug/mL 502.2 Cal 2000 Megamix – ICV 2500 µL of 5000 ug/mL VOA Calibration Mix #1 – ICV 6250 µL of 2000 ug/mL Pace GB Custom Mix – ICV 1250 µL of 10000 ug/mL Pace GB Custom Mix – Cyclohexanes – ICV 6250 µL of 20000 ug/mL Pace GB Custom Mix – Acrolein – ICV 6250 µL of 2000 ug/mL Pace GB Custom Mix – 2-Chloroethylvinyl	25 mL

			ether – ICV 1315 µL of 9520 ug/mL Vinyl Acetate 6250 µL of Various Concentration Pace GB Custom Mix – NELAP – ICV 6250 µL of Pace GB Custom Appendix 9 Standard Mix – ICV Diluted into methanol	
100 ug/mL Matrix Spike/Blank Spike Solution for Michigan List	MS/BS Stock Solution for Michigan List	100 ug/mL for all compounds	1250 µL of 2000 ug/mL Michigan GRO Mix Dilute into methanol.	25 mL
MS/MSD – BS/BSD Spike for Water Samples	MS/MSD BS/BSD	50 ug/L for most compounds, Various for others.	Add 21 µL of the appropriate Matrix Spike/Blank Spike Solution to 42 mL of sample for MS/MSD, or 42 mL of reagent water for BS/BSD	42 mL
MS/MSD – BS/BSD Spike for Low Level Soil Samples	MS/MSD BS/BSD	50 ug/kg for most compounds, Various for others.	Add 2.5 µL of the appropriate Matrix Spike/Blank Spike Solution to 5.0 g of sample containing 5 mL of reagent water for MS/MSD, or 5.0 g of Ottawa sand containing 5 mL of reagent water for BS/BSD	5 mL
MS/MSD – BS/BSD Spike for Methanol Preserved Soil Samples	MS/MSD BS/BSD	2500 ug/kg for most compounds, Various for others.	Add 50 µL of the appropriate Matrix Spike/Blank Spike Solution to 10 g of sample containing 10 mL of methanol for MS/MSD, amount of Spike Solution is adjusted according to the initial methanol volume; or 10 g of Ottawa sand containing 10 mL of methanol for BS/BSD	10 mL

10. Calibration

10.1. Tune Verification

The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. If the current tune status does not meet the ion ratio criteria in the method (see section 11.2), follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be verified after the tuning procedures.

Refer to section **11.2 Procedures** for details on the analysis and evaluation of this standard.

10.2. Initial Calibration (ICAL)

10.2.1. Analysis of Standards

An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to table 10.1 for compound concentrations.

An analyte must be present and calibration curve in control in order to be reported on the target analyte list. Analytes identified by mass spectral match but not present and in control in the calibration table may be reported as Tentatively Identified Compounds (TICs). Guidelines for identification are listed in Section 11.6.3. Results for these TICs should be reported only on a present/absent basis. However,

quantitative results may be reported provided they are qualified as estimated values.

Table 10.1 – Calibration standard compound concentrations

Analyte	PQL water (ug/L)	PQL soil (ug/kg)	PQL 5035 soil (ug/Kg)	Std 1 Ug/L	Std 2 ug/L	Std 3 ug/L	Std 4 ug/L	Std 5 ug/L	Std 6 ug/L	Std 7 ug/L	Std 8 Ug/L	Std 9 ug/L
1,1-Dichloroethane	1	50	5	1	5	20	50	100	200			300
1,1-Dichloroethene	1	50	5	1	5	20	50	100	200			300
1,1-Dichloropropene	1	50	5	1	5	20	50	100	200			300
1,1,1-Trichloroethane	1	50	5	1	5	20	50	100	200			300
1,1,2-Trichloroethane	1	50	5	1	5	20	50	100	200			300
1,1,1,2-Tetrachloroethane	1	50	5	1	5	20	50	100	200			300
1,1,2,2-Tetrachloroethane	1	50	5	1	5	20	50	100	200			300
1,2,4-Trichlorobenzene	1	50	5	1	5	20	50	100	200			300
1,2-Dichlorobenzene	1	50	5	1	5	20	50	100	200			300
1,2-Dibromo-3-chloropropane	5	250	5		5	20	50	100	200			300
1,2-Dichloroethane	1	25	5	1	5	20	50	100	200	0.50	0.40	300
1,2-Dibromoethane	1	50	5	1	5	20	50	100	200	0.50	0.40	300
1,2-Dichloropropane	1	50	5	1	5	20	50	100	200			300
1,2-Dichloroethene (Total)	2	100	10	2	10	40	100	200	400			600
1,2,3-Trimethylbenzene	1	50	5	1	5	20	50	100	200			300
1,2,4-Trimethylbenzene	1	50	5	1	5	20	50	100	200			300
1,2,3-Trichlorobenzene	1	50	5	1	5	20	50	100	200			300
1,2,3-Trichloropropane	1	50	5	1	5	20	50	100	200			300
1,3-Dichlorobenzene	1	50	5	1	5	20	50	100	200			300
1,3-Dichloropropane	1	50	5	1	5	20	50	100	200			300
1,3,5-Trimethylbenzene	1	50	5	1	5	20	50	100	200			300
1,4-Dichlorobenzene	1	50	5	1	5	20	50	100	200			300
1,4-Dioxane (p-Dioxane)	250	12500	250		250	1000	2500	5000	10000			15000
2,2-Dichloropropane	1	50	5	1	5	20	50	100	200			300
2,3-Dichloropropene	1	50	5	1	5	20	50	100	200			300
2-Butanone (MEK)	5	250	5		5	20	50	100	200			300
2-Chlorotoluene	1	50	5	1	5	20	50	100	200			300
2-Chloroethylvinyl ether	1	50	5	1	5	20	50	100	200			300
2-Hexanone	5	250	5		5	20	50	100	200			300
2-Methylnaphthalene	5	250	5		5	20	50	100	200			300
2-Propanol	250	12500	50	10	50	200	500	1000	2000			3000
Allyl chloride	5	50	5		5	20	50	100	200			300
4-Chlorotoluene	1	50	5	1	5	20	50	100	200			300
TOTAL BTEX	1	300	30	1	5	20	50	100	200			300
Carbon disulfide	1	50	5	1	5	20	50	100	200			300
Ethanol	100	5000	500	100	500	2000	5000	10000	20000			30000
Acetone	5	250	5		5	20	50	100	200			300
Acrolein	10	2500	50	10	50	200	500	1000	2000			3000
Acetonitrile	20	1000	20	2.5	12.5	50	125	250	500			750
Acrylonitrile	5	250	5		5	20	50	100	200			3200
Bromochloromethane	1	50	5	1	5	20	50	100	200			300
Benzene	1	25	5	1	5	20	50	100	200	0.50	0.40	300

Bromobenzene	1	50	5	1	5	20	50	100	200			300
Bromodichloromethane	1	50	5	1	5	20	50	100	200			300
Bromomethane	1	50	5	1	5	20	50	100	200			300
Bromoform	1	50	5	1	5	20	50	100	200			300
cis-1,2-Dichloroethene	1	50	5	1	5	20	50	100	200			300
cis-1,3-Dichloropropene	1	50	5	1	5	20	50	100	200			300
cis-1,4-Dichloro-2-butene	5	250	5		5	20	50	100	200			300
Carbon tetrachloride	1	50	5	1	5	20	50	100	200			300
Cyclohexane	5	250	5		5	20	50	100	200			300
Chlorobenzene	1	50	5	1	5	20	50	100	200			300
Chloroprene	20	1000	20	2.5	12.5	50	125	250	500			750
Chloroethane	1	50	5	1	5	20	50	100	200			300
Chloroform	5	250	5		5	20	50	100	200			300
Chloromethane	1	50	5	1	5	20	50	100	200			300
Cyclohexanone	50	12500	50		50	200	500	1000	2000			3000
Dibromochloromethane	1	50	5	1	5	20	50	100	200			300
Dichlorofluoromethane	1	50	5	1	5	20	50	100	200			300
Diethyl ether (Ethyl ether)	1	50	5	1	5	20	50	100	200			300
Dichlorodifluoromethane	1	50	5	1	5	20	50	100	200			300
Diisopropyl ether	1	50	5	1	5	20	50	100	200			300
Dibromomethane	1	50	5	1	5	20	50	100	200			300
Ethyl acetate	50	2500	50		50	200	500	1000	2000			3000
Ethylbenzene	1	25	5	1	5	20	50	100	200	0.50	0.40	300
Ethyl methacrylate	5	250	5		5	20	50	100	200			300
Ethyl-tert-butyl ether	5	250	5		5	20	50	100	200			300
Hexachloro-1,3-butadiene	1	50	5	1	5	20	50	100	200			300
Hexachloroethane	5	50	5		5	20	50	100	200			300
Iodomethane	5	250	5		5	20	50	100	200			300
Isopropylbenzene (Cumene)	1	50	5	1	5	20	50	100	200			300
Isoamyl acetate	5	250	5		5	20	50	100	200			300
Isopropyl acetate	5	250	5		5	20	50	100	200			300
Isobutanol	50	2500	50		50	200	500	1000	2000			3000
Methyl acetate	5	250	5		5	20	50	100	200			300
Methylene Chloride	1	50	5	1	5	20	50	100	200			300
Methyl methacrylate	5	250	5		5	20	50	100	200			300
Methylcyclohexane	5	250	5		5	20	50	100	200			300
Methyl-tert-butyl ether	1	50	5	1	5	20	50	100	200			300
Methacrylonitrile	20	1000	20	2.5	12.5	50	125	250	500			750
4-Methyl-2-pentanone	5	50	5		5	20	50	100	200			300
m&p-Xylene	2	50	10	2	10	40	100	200	400	1.0	0.80	600
n-Amyl acetate	5	250	5		5	20	50	100	200	0.50	0.40	300
Naphthalene	5	50	5		5	20	50	100	200			300
n-Butyl acetate	5	50	5		5	20	50	100	200			300
n-Butanol	250	12500	250	50	250	1000	2500	5000	10000			15000
n-Butylbenzene	1	50	5	1	5	20	50	100	200			300
n-Heptane	5	250	5		5	20	50	100	200			300
n-Hexane	5	250	5		5	20	50	100	200			300
n-Propylbenzene	1	50	5	1	5	20	50	100	200			300

n-Propanol	250	2500	250	50	250	1000	2500	5000	10000			15000
o-Xylene	1	25	5	1	5	20	50	100	200	0.50	0.40	300
p-Isopropyltoluene	1	50	5	1	5	20	50	100	200			300
Propionitrile	20	1000	20		12.5	50	125	250	500			750
Propyl acetate	5	250	5		5	20	50	100	200			300
sec-Butlybenzene	1	50	5	1	5	20	50	100	200			300
Styrene	1	50	5	1	5	20	50	100	200			300
trans-1,2-Dichloroethene	1	50	5	1	5	20	50	100	200			300
trans-1,3-Dichloropropene	1	50	5	1	5	20	50	100	200			300
trans-1,4-Dichloro-2-butene	5	250	5		5	20	50	100	200			300
tert-Amyl-methyl ether	1	250	5	1	5	20	50	100	200			300
tert-Butyl Alcohol	25	1250	25		25	100	250	500	1000			1500
Tetrachloroethene	1	50	5	1	5	20	50	100	200			300
Tetrahydrofuran	5	250	5		5	20	50	100	200			300
1,1,2-Trichlorotrifluoroethane	5	50	5		5	20	50	100	200			300
Toluene	1	50	5	1	5	20	50	100	200			300
Trichloroethene	1	50	5	1	5	20	50	100	200			300
Trichlorofluoromethane	1	50	5	1	5	20	50	100	200			300
tert-Butylbenzene	1	50	5	1	5	20	50	100	200			300
Xylene (Total)	3	75	15	3	15	60	150	300	600	1.5	1.2	900
Vinyl acetate	5	250	5		5	20	50	100	200			300
Vinyl chloride	1	50	5	1	5	20	50	100	200	0.50	0.40	300

10.2.2. **Calibration Response Factors**

Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:
A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L).
C_x = Concentration of the compound being measured (µg/L).

Most, if not all modern chromatography data systems are capable of calculating this factor and using it to quantify analyte concentrations. The 8260B method has minimum requirements that these

response factors must meet in order to be considered valid. The method uses a subset of the target analyte list to evaluate the performance of the system. These compounds are referred to as the System Performance Check Compounds or the SPCCs. The SPCCs serve as an indicator of instrument sensitivity and, by meeting a minimum value, ensure that the laboratory has adequate sensitivity to analyze and reliably report data for environmental samples.

10.2.3. **Calibration Curve Fit**

The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the "goodness of fit".

Average Response Factor (ARF): The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the ARF (see Table 10.2).

The % RSD is calculated as follows: %RSD = SD*100 / ARF

Where: SD = Standard deviation of the averaged RFs for a given compound

The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The ***Calibration Check Compounds (CCCs)*** are a subset of the target analyte list that must meet specific criteria (see Table 10.2) for the calibration to be acceptable. For the CCCs, the %RSD for each is compared to the method criteria. If that of any CCC exceeds the criteria, the system needs to be inspected for potential sources of errors and recalibrated.

Linear Regression: The linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of y=ax+b where "a" is the slope of the line and "b" is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y-axis at the origin. However, because the linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. A calculation of the correlation coefficient "r" is used to determine the acceptability of a linear regressed curve (see Table 10.2)

Non-linear Regression: The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

Note: The State of South Carolina does not allow the use of Non-linear regression for compliance samples. Analytes that have poor purging efficiency or are problematic compounds may require the use of Non-linear regression curves. These may include: Bromomethane, 1-Propanol, Acrolein (2-propenal), n-Butanol, 2-Butanone, Carbon Disulfide, Hexachloroethane, and 1,2-Dibromo-3-chloropropane (DBCP)

Refer to section 10.2.3 for curve fit criteria. Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious problem is discovered and documented. The point must be dropped in its entirety and reanalyzed. Re-analysis should be within the same 12-hour time window and must occur within 8 hours of the original analysis.

10.3. Calibration Verification

10.3.1. Second Source Verification (SCV)

In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the "fit" of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

Therefore, to assess the accuracy relative to the purity of the standards, a single 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. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{SCV} - \text{TrueValue}_{SCV}] / \text{TrueValue}_{SCV} * 100$$

See Tables 9.6 and 9.7 for details on the preparation of this standard. See Table 10.2 for control criteria

10.3.2. 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 at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished 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 change calculated. The percent change must meet the method specified criteria for the analysis to proceed for an additional 12 hours.

The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\% \text{ Difference: } [RF_{CCV} - AvgRF_{CAL}] / AvgRF_{CALI} \times 100$$

$$\% \text{ Drift: } [\text{Result}_{CCV} - \text{TrueValue}_{CCV}] / \text{TrueValue}_{CCV} \times 100$$

Table 10.2 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor	%RSD ≤ 15%	If not met, try linear regression fit
	Linear Regression	r ≥ 0.99	If not met, try non-linear regression fit
	Non-linear Regression	COD ≥ 0.99	If not met, remake standards and recalibrate
System Performance Check Compounds (SPCCs)	Chloromethane 1,1-Dichloroethane Bromoform Chlorobenzene 1,1,2,2-Tetrachloroethane	Avg RF ≥ 0.10 Avg RF ≥ 0.10 Avg RF ≥ 0.10 Avg RF ≥ 0.30 Avg RF ≥ 0.30	Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, poor purging efficiency, and active sites in the column or chromatographic system.
Calibration Check Compounds (CCCs)	1,1-Dichloroethane Toluene Chloroform Ethylbenzene 1,2-Dichloropropane Vinyl Chloride	%RSD < 30%	%RSD for the calibration check compounds (CCC's) must be ≤30% regardless of curve fit used. If the CCCs are not included on a list of analytes for a project, and therefore not included in the calibration standards, then all compounds of interest must meet a ≤15% RSD criterion.
Second Source Verification Standard	Immediately after each initial calibration	% Diff ±30%	Acceptance criteria are ±30% for all analytes, with allowances for 5% of compounds @ ±40%. See ALL_Q_025_Rev.1
Continuing Calibration Verification	Prior to the analysis of any samples and every 12 hours thereafter		If the requirements for continuing calibration are not met, these corrective actions must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back.
	SPCCs	Must meet response criteria listed above	
	Internal Standard RT	RT ± 30 sec	Use midpoint calibration standard as reference
	Internal Standard Response	50 – 200%	Use midpoint calibration standard as reference
	CCCs	RF ± 20% Diff.	Use for Avg RF calibration curves
		Result ± 20% Drift	Use for linear and non-linear calibration curves Additional client specific requirements for the analysis of contract samples requires that BTEX, PAH, Oxygenates, and surrogate compounds also be considered CCCs and must meet the 20% CCV criterion.
	Non-CCC Targets	RF ± 50% Diff.* Result ±50% Drift	Some programs may require control over non-CCC target analytes. In the absence of specified criteria, use those listed *State of South Carolina requires non-CCC Compounds to meet ±30% Drift. Please Note: Analytes that have poor purging efficiency or are problematic compounds may require the use ±50% Drift. These may include: Bromomethane, 1-Propanol, Acrolein (2-propenal), n-Butanol, 2-Butanone, Carbon Disulfide, Hexachloroethane, and 1,2-Dibromo-3-chloropropane (DBCP)

10.4. Calibration Corrective Actions

10.4.1. Calibration Linearity Problems

10.4.1.1. Check instrumentation/equipment condition.

10.4.1.2. Enter maintenance in instrument maintenance logbook.

10.4.1.3. Perform another initial calibration.

10.4.1.4. No data can be reported.

10.4.1.5. Generate on Non-Conformance Memo.

10.4.2. Secondary Verification Problems

10.4.2.1. Check instrumentation/equipment condition.

10.4.2.2. Enter maintenance in instrument maintenance logbook.

10.4.2.3. Perform another initial calibration.

10.4.2.4. No data can be reported.

10.4.2.5. Generate on Non-Conformance Memo

10.4.3. Continuing Verification Problems

10.4.3.1. Reanalyze the original CCV standard to determine instrument consistency.

10.4.3.2. Prepare and analyze a new CCV standard to determine preparation consistency / standard integrity.

10.4.3.3. Document instrument maintenance

10.4.3.4. Reanalyze CCV standard to determine if maintenance was effective in restoring performance.

10.4.3.5. Complete recalibration of instrument.

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 injection log with a thorough explanation for the deviation from policy.

Exceptions: If calibration verification is above the upper control limit, samples non-detected for those analytes may be reported without reanalysis.

11. Procedures

11.1. Purge-Trap GC/MS System Preparation

11.1.1. Operating Parameters

Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration.

Table 11.1 –Instruments and Operating Parameters

Instrument ID	**Component**	**Settings and Consumables**	
40MSV1	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.5 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: 40°C Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	
Instrument ID	**Component**	**Settings and Consumables**	
40MSV2	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.5 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: NO Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	

Instrument ID	Component	Settings and Consumables	
40MSV3	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 1.0 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: 40°C Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	
Instrument ID	**Component**	**Settings and Consumables**	
40MSV4	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.5 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: NO Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	

Instrument ID	Component	Settings and Consumables	
40MSV5	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.5 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: NO Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	
Instrument ID	**Component**	**Settings and Consumables**	
40MSV6	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.8 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: NO Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	

Instrument ID	Component	Settings and Consumables	
40MSV7	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.5 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: 40°C Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	
Instrument ID	**Component**	**Settings and Consumables**	
40MSV8	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 1.0 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: NO Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: 90°C Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	

Instrument ID	**Component**	**Settings and Consumables**	
40MSV9	Gas Chromatograph	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.5 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	Tune File: Named to date of tune	
	Purge & Trap Concentrator	Prepurge: NO Preheat: NO Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	
Instrument ID	**Component**	**Settings and Consumables**	
40MSVA	**Gas Chromatograph**	**Column**: J&W Scientific DB-624 Capillary Column, 20m x 0.18 mm, i.d. 1.0 um **Inlet Liner:** Restek 4 mm Single Gooseneck Injection Port Liners **Inlet Seal:** Restek Gold Plated inlet seal **Column Ferrules:** Restek 04.mm Vespel/Graphite ferrules	Pressure / Flow: 0.5 mL / min Initial Temperature: 40°C Initial Time: 3 min Final Temperature: 8°C / min to 110°C 0 min hold 20°C / min to 220°C 1 min hold Final Time: 18.25 min Injector Temperature: 220°C Detector Temperature: 240°C
	Mass Spectrometer	**Tune File:** Named to date of tune.	
	Purge & Trap Concentrator	Prepurge: NO Preheat: NO Sample: 20°C Purge: 10 min Dry Purge: NO Desorb Preheat: 245°C Desorb: 250°C	Standby: - Bake: 270°C for 7 min BGB: OFF Valve: 150°C Line: 150°C Mount: N/A Transfer Line Temp: 150°C
	Autosampler	Syringe Flushes: 2 Sparge Tube Flushes: 2	

11.2. Tune Verification

At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified. This is done by analyzing a standard containing bromofluorobenzene (refer to table 9.2). The tune verification standard can be combined with the CCV standard provided that the amount of BFB introduced into the system meets the criteria in Section 11.2.

After the analysis of this standard, the mass spectrum of BFB must be evaluated against the following criteria.

Mass (m/z)	Ion Abundance criteria
50	15.00-40.00% of m/z 95
75	30.00-60.00% of m/z 95
95	Base peak, 100% relative abundance
96	5.00-9.00% of m/z 95
173	<2.00% of m/z 174
174	50.00-99.00% of m/z 95
175	5.00-9.00% of m/z 174
176	95.00-101.00% of m/z 174
177	5.00-9.00% of m/z 176

To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for bromofluorobenzene. If the software has a program or macro for automatically selecting the spectra and evaluating the response ratios, use this option. Otherwise, the spectra must be obtained in one the following manners, in the listed order.

1. Using an average of three scans, centered on the apex of the peak; or,

2. Using an average of all scans across the width of the peak, taken at half height; or,

3. Using an average of all scans taken across the width of the peak from baseline to baseline.

A background scan taken immediately before but not including the peak must be subtracted.

Once obtained, evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, then analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date / time for the BFB tune verification. After that, the tune must be verified again to establish a new analytical window. The same Ion Abundance Criteria used for the BFB tune coupled with the initial calibration must be used for all subsequent analyses associated with that initial calibration.

If the ratios do not meet the criteria, refer to the following corrective actions to address the problem:

1. Retune the mass spectrometer following the equipment manufacturers' instructions. The tune status must be verified after the tuning procedures.
2. If this fails, change filament and retune.
3. If this fails, take down the mass spectrometer and clean the instrument.

11.3. Calibration Verification

After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. If it has already been determined that the instrument needs to be recalibrated, follow the procedures listed in section 10.2 (Analysis of Standards). Otherwise, analyze a Continuing Calibration Verification Standard to determine the current calibration status.

If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed section 10.4.3 (Continuing Verification Problems). If the tune verification has been combined with the CCV, the 12 hour analysis window begins from the analysis date / time of the CCV.

Note: In situations where the instrument will run unattended (i.e. overnight), the analyst may load sequential CCVs in anticipation of that the first in the series may fail due to carry over from a previous sample. If so, the CCV must be evaluated according to the protocol set forth in the Quality Assurance Manual within Section 6 – Equipment and Measurement Traceability.

11.4. Sample Preparation

11.4.1. Samples

11.4.1.1. Sample Pre-screening

11.4.1.1.1. Samples are pre-screened using a rapid GC headspace technique. See SOP S-GB-O-001 *Sample Screening Volatile Organics Prior to Preparation* for the specifics on the pre-screening of samples.

11.4.1.2. Water Samples

After pre-screening, water samples typically do not require any sample preparation unless they require a dilution to bring high-level contaminants within calibration range or to minimize matrix interference. Dilutions are made following Section 11.4.1.4.1.

After analysis check the residue in the vial following analysis using pH paper. The pH should be <2. Document results in the run sequence log as <2 or >2. Footnote any sample not meeting the pH requirement. If dilutions are required, pH preservation can be verified at the time the dilution is made using the sample remaining in the original sample container.

Soil Samples

11.4.1.2.1. Low concentration soils

Samples received for low level analysis should be contained in pre-weighed VOA vials either with or without Organic Free Water (OFW) and/or sodium bisulfate preservative. NOTE: some samples may be received in coring devices (e.g. Encore™, etc.). These samples must be extruded into a VOA vial either with or without OFW and/or sodium bisulfate and a magnetic stir bar within 48 hours of sample collection. If samples are received that are greater than 10g the PM must be notified and samples will be rejected for analysis.

11.4.1.2.1.1. Weight determination: Prior to preparation or analysis of any soil received in a pre-weighed VOA vial, the sample weight must be determined and recorded. Accurately weigh the VOA vial to 0.01 g in the laboratory; record this amount in the sample preparation logbook. Subtract the tare weight recorded on the vial; this will be the weight of sample in the vial.

11.4.1.2.1.2. Samples received pre-weighed in the field must be in 40 mL VOA vials and contain a magnetic stir bar, acid preservative and a field tare weight. The analyst will compare the field tare weight to the weight of the sample before analysis. The weight of the sample should be recorded.

11.4.1.2.1.3. All samples must be extruded from the coring devices within 48 hrs. of collection. If the samples are to also be analyzed within the 48 hr criteria, no acid preservation is required. If analysis is to occur after 48 hrs. but within 14 days, the samples must be preserved with Sodium Bisulfate or if preserved with OFW, stored frozen. The ratio of Sodium Bisulfate to sample weight is 0.2g of preservative to 1g of sample.

11.4.1.2.2. High concentration soils

11.4.1.2.2.1. Methanol-Preserved Samples: Samples received in pre-weighed vials preserved with methanol must be accurately weighed in the laboratory to 0.01 g and the sample weight determined. Subtract the weight written on the VOA vial from the weight determined in the laboratory. This will be the weight of sample in the VOA vial. The volume of methanol in the sample container should be at a 1 to 1 ratio of soil to methanol. See Section 11.4.1.3.2.3.

11.4.1.2.2.2. Unpreserved Samples: Samples received in unpreserved pre-weighed vials must be accurately weighed in the laboratory to 0.01 g. NOTE: some samples may be collected and transported to the laboratory in bulk containers. An accurately weighed ≥5 gram sub-sample must be taken and added to a 40-ml VOA vial. The non-compliant sample collection technique must be recorded in the preparation logbook and a qualifier added to the sample result. The samples are then preserved with 10 ml methanol within 48 hours of sample collection. To determine the sample weight: subtract the weight written on the VOA vial from the weight determined in the laboratory prior to the addition of the 10 ml methanol preservative. This will be the weight of sample in the VOA vial. See Section 11.4.1.3.2.3.

11.4.1.2.2.3. The balance is to be leveled before calibration. Calibration verification of the analytical balance is done with S-class weights. These values are to be noted in the Balance calibration logbook. The frequency of balance calibration verification is once per day before the balance is used or when the balance is moved.

11.4.1.2.3. Sample Preparation Problems

Missing Tare Weights on VOA vials: Mark the vial to indicate pre-weight must be determined after sample analysis. Record the weight of the full vial with sample. Following sample analysis, transfer any remaining sample into another vial. Clean the original vial and remove all trace of the sample. Add all contents that were present in the vial that were not sample content (magnetic stir bar, preservative, organic free water, etc). Take care to add the exact amount of contents (weights and volumes) that were originally in the vial when sample was collected. Weigh the vial with all non-sample contents and record this weight as the estimated initial weight of the VOA vial. Subtract this estimated initial vial weight from the full vial weight with sample (recorded prior to sample analysis). Document all steps and weights and include a narrative on sample report indicating action taken to estimate initial vial weight for sample weight determination.

Note: Unless otherwise requested by the client or Project Manager, an average weight of VOA methanol preserved vials is used to determine the tare weight. This method is followed in lieu of transferring the sample to another vial and weighing the original vial

11.4.1.3. Dilutions

11.4.1.3.1. Water

Dilutions on aqueous samples must be prepared in a volumetric fashion. Sample aliquots may be measured in either a volumetric pipette or syringe and brought to volume in a volumetric flask.

11.4.1.3.1.1. All steps must be performed without delays until the diluted sample is in a 40 mL VOA Vial.

11.4.1.3.1.2. Dilutions are made in gastight 50mL syringes.

11.4.1.3.1.3. Calculate the approximate volume of organic-free reagent water added to the syringe and add slightly less than this quantity of organic-free reagent water to the syringe barrel.

11.4.1.3.1.4. Inject the proper aliquot of sample using the appropriate 10uL to 5mL syringes to create the desired dilution in the 50 mL syringe. Dilute the sample to the mark with organic-free reagent water. Invert, and shake three times. Repeat the above procedure for additional dilutions.

11.4.1.3.1.5. Fill a 40-mL VOA vial with the diluted sample from the 50 mL syringe prepared in Section 11.4.1.4.1.4.

11.4.1.3.1.6. Place the VOA vial on the autosampler. All dilutions should keep the response of a major constituent (previously saturated peaks) in the upper half of the linear range of the curve.

11.4.1.3.1.7. The autosampler will add the 1.0 uL of the internal standard and surrogate to the sample and transfer 5 mLs over to the 5 mL sparge tube on the Tekmar 3000 concentrator.

11.4.1.3.2. Soil

11.4.1.3.2.1. Low Level Soils

11.4.1.3.2.1.1. It will be necessary to adjust the sample weight for quantitation purposes. Any analyte hits outside of the calibration range, 200 ug/kg, will be extracted into Methanol and analyzed under High Concentration Sample criteria.

11.4.1.3.2.2. High Concentration Soils

11.4.1.3.2.2.1. Dilute all samples according to the results of the screening data. A standard analytical dilution is 1:50. Add 1.0 mL of the sample extract measured with a microsyringe of appropriate volume to 49 mL of reverse osmosis water in a 50 mL syringe.

11.4.1.3.2.2.2. To make dilutions other than a standard 1:50 dilution, fill a 50 mL syringe to a volume of 49 mL with reverse osmosis water. Using a 1.0 mL syringe, inject methanol into the 50 mL syringe to bring the total volume of sample and methanol to equal 1.0 mL. Using an appropriate volume syringe, inject the sufficient amount of sample to reach desired dilution.

11.4.1.3.2.2.3. The 50 mL syringe contents are place into a 40 mL VOC vial by slowly deploying the plunger and injecting on the side of the vial to eliminate cavitation and loss of analytes to volatilization or sparging. Enough of the contents are injected to created a meniscus at the top of the vial that when capped will produce a no headspace sample.

11.4.1.3.2.2.4. The vial is capped and checked for headspace. If vial is free of headspace, it is ready for analysis as per Section 11.5.3.

11.4.2. Batch QC

Refer to Table 12.1 for details on Batch QC requirements.

11.4.2.1. Method Blank

11.4.2.1.1. Water

11.4.2.1.1.1. Fill a 40ml VOA vial with OFW and place on autosampler rack. The autosampler will add 1.0 uL of the internal standard and surrogate to the sample and transfer 5mLs over to the 5mL sparge tube on the Tekmar 3000 concentrator.

11.4.2.1.2. Low Level Soil

11.4.2.1.2.1. A method blank is prepared with 5 mL of OFW into a 40 mL VOA vial containing a disposable magnetic stir bar. The vial is placed onto the autosampler and 1 uL of IS/SS mix @ 250 ug/mL is added. The blank is preheated to 40°C and purged. The method blank must be analyzed under the same criteria as the samples.

11.4.2.1.3. High Concentration Soil

11.4.2.1.3.1. The method blank (extraction blank) is made by adding 10 µL of the 2500 ppm surrogate standard in 10 mL methanol placed in a 2 oz. jar containing 10g of Ottawa sand. A 1.0 mL portion of this is diluted with 49 mL of OFW for a final concentration on the instrument of 50 ug/L.

11.4.2.2. Laboratory Control Sample

See Table 9.7 for procedure.

11.4.2.3. MS/MSD Samples

See Table 9.7 for procedure

11.5. Analyze Samples

11.5.1. Water Samples

11.5.1.1. Create run sequence log. Place 40-mL VOA vial containing sample (Section 11.4.1.1), or appropriately diluted 40 mL VOA vial containing sample (Section 11.4.1.4.1) onto the autosampler. The autosampler will add the 1.0 uL of the internal standard and surrogate to the sample and transfer 5 mLs over to the 5 mL sparge tube on the Tekmar 3000 concentrator.

11.5.2. Low-level Samples

11.5.2.1. Create run sequence log. Place the already weighed 40 mL VOA vial containing the sample, stir bar and 5 mL of OFW or Sodium Bisulfate solution (Section 11.4.1.3.1) onto the autosampler where another 5 mLs of OFW and 1 uL of IS/SS @ 250 ug/mL will be added by the autosampler. The sample is preheated to 40°C and purged. The stir bar is moving continuously during the purge cycle. This also helps in compound recovery by breaking down any clumps that may remain in the sample.

It will be necessary to adjust the sample weight for quantitation purposes. Any analyte hits outside of the calibration range, 200 ug/kg, will be extracted into Methanol and

analyzed under High Concentration Sample criteria.

11.5.3. High Concentration Samples

11.5.3.1. Create run sequence log. Place 40-mL VOA vial containing sample (Section 11.4.3.2), or appropriately diluted 40 mL VOA vial containing sample (Section 11.4.1.4.2) onto the autosampler. The autosampler will add the 1.0 uL of the internal standard to the sample and transfer 5 mLs over to the 5 mL sparge tube on the Tekmar 3000 concentrator

11.6. Data Reduction

11.6.1. Qualitative Analysis

Retention Time Comparison: The relative retention time (RRT) of the sample component must be within ±0.06 RRT units of the component in the calibration verification standard. Extracted Ion Current Plots (EICPs) may be used to provide a more reliable assignment of RT in the presence of co eluting components.

Mass Spectrum Comparison: The characteristic ions from the reference mass spectrum are defined as 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.

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times.
- Compare computer-matched compounds with reference spectra to accept or reject each identification.
- All ions present in the reference spectrum that are at least 10% of the base peak must be present in the sample background-subtracted spectrum.
- The relative intensities of these ions must agree within +/-30% between the standard and sample spectra.
- While this is a good guideline, acceptance or rejection will depend upon the judgment of the analyst.

11.6.2. Quantitative Analysis – Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

Raw Data Results: The GC/MS data system will calculate the concentration of each analyte as µg/l (or ng/ml). For water samples, no further calculations are necessary unless a dilution of the sample has been performed. If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

11.6.2.1. Results Calculation – Aqueous Sample:

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(I_s)}{(A_{is})(RF)(V_o)}$$

Where:

A_x= Area of characteristic ion for compound being measured.
I_s = Amount of internal standard injected (ng).
A_{is} = Area of characteristic ion for the internal standard.
RF = Average Relative Response factor for compound being measured.
V_o = Volume of water purged (ml), taking into consideration any dilutions made.

11.6.2.2. Soil/ Solid calculations:

$$High \text{ Conc. (ug/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)}$$

$$Low \text{ Conc. (ug/kg)} = \frac{(A_x)(I_s)}{(A_{is})(RF)(W_s)}$$

Where:

A_x, I_s, A_{is}, RF = Same as in water and water-miscible waste above.
V_t = Volume of total extract (ml).
V_i = Volume of extract added (ml) for purging.
S_v = Volume of diluted extract.
W_s= Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

11.6.3. **Tentatively Identified Compounds (TICs)** – For some samples, identification may be desired for non-target compounds. A mass spectral library search may be conducted to attempt assignment of tentative identifications. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12. Quality Control

12.1. **Instrument Quality Control:** Refer to Table 10.2 for initial and continuing calibration criteria and corrective actions.

12.2. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One (1) per 20 samples or 12 hour window (whichever is most frequent)	Target analytes must be less than reporting limit. If results are reported to MDL, target analytes in MB should be non-detect	1. Re-analyze associated samples. ***Exceptions:*** 1. If sample ND, report sample without qualification 2. If sample result >20x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination. 3. If sample result <20x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Method specified compounds: Benzene, Chlorobenzene, 1,1-Dichloroethene, Toluene, Trichloroethene *OR (alternative)* Full Target List compounds	One (1) per batch of up to 20 samples	Laboratory derived limits Method Specified List: All compounds must pass control criteria, with no exceptions. Full Target List: Marginal exceedances allowed according to NELAC 2003 Chap 5 D.1.1.2.1.e	1. Analyze a new LCS 2. If problem persists, check spike solution 3. Perform system maintenance prior to new LCS run ***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.
Laboratory Control Sample Duplicate (LCSD)	Method specified compounds: Benzene, Chlorobenzene, 1,1-Dichloroethene, Toluene, Trichloroethene *OR (alternative)* Full Target List compounds	One (1) per batch of up to 20 samples	Laboratory derived limits Method Specified List: All compounds must pass control criteria, with no exceptions. Full Target List: Marginal exceedances allowed according to NELAC 2003 Chap 5 D.1.1.2.1.e	4. Analyze a new LCSD 5. If problem persists, check spike solution 6. Perform system maintenance prior to new LCSD run ***Exceptions:*** 1) If LCSD 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)	Method specified compounds: Benzene, Chlorobenzene, 1,1-Dichloroethene, Toluene, Trichloroethene *OR (alternative)* Full Target List compounds	One (1) per batch of up to 20 samples, must include one TCLP MS for any analyzed in sequence	Laboratory derived limits	1. If LCS/LCSD and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup	One (1) for every 5% of all environmental samples	Laboratory Derived Limits	1. Report results with an appropriate footnote.

12.3. **Sample Quality Control**

Table 12.2 – Sample Quality Control criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	Pentafluorobenzene 1,4 Difluororobenzene 1,4-Dichlorobenzene-d4 Chlorobenzene-d5	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	**Retention Time:** RT must be ± 30 seconds from last calibration check on all samples	**Retention Time Failure:** 1. If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting. 2. Affected samples should be reanalyzed in the absence of an obvious instrument or matrix related interference.
Surrogate Standards	Dibromofluoromethane Toluene-d8 4-Bromofluorobenzene	Added to all samples, spikes, control samples and method blanks prior to analysis	Laboratory derived limits	1. Check system parameters 2. Identify and correct likely cause 3. Re-run samples ***Exceptions:*** 1. Surr rec above criteria and target compounds < RL, result may be reported with appropriate footnote. 2. Surr rec out of control due to obvious sample matrix interference (i.e. co-elution), report results with appropriate footnote.

13. Method Performance

13.1. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually per S-ALL-Q-004, *Method Detection Limit Studies* for each matrix 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. **Method Modifications**

Method modifications for EPA method 8260B are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- 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.
- Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.

Method modifications for EPA method 5035 is as follows:

- The laboratory uses a modification of SW-846 Method 5035 for medium-level volatiles in soil. The laboratory uses 10 grams of soil and 10 mL of methanol whereas the method indicates 5 grams of soil and 5 mL of methanol.
- *Note*: Samples reported to the State of South Carolina requires the use of 5 grams of soil and 5 mL of methanol.

14. Pollution Prevention and Waste Management

14.1. Procedures for handling waste generated during this analysis are addressed in S-GB-S-006, *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 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

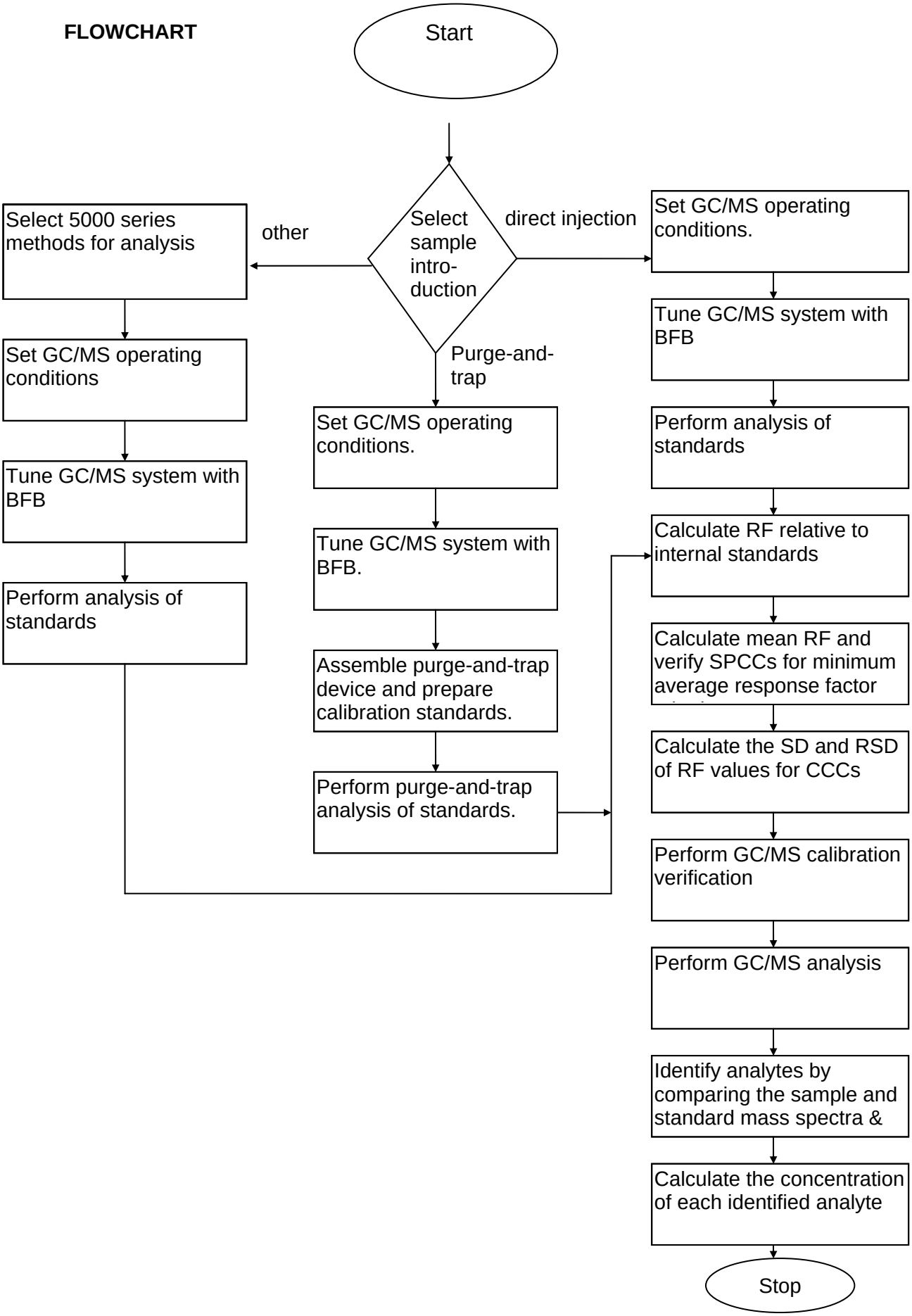
15.2. USEPA, SW-846, Method 5030B, "Purge and Trap for Aqueous Samples," December 1996.

15.3. USEPA, SW-846, Method 5035A, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Draft Revision 1 July 2002.

15.4. USEPA, SW-846, Method 8000B, "Determinative Chromatographic Separations", December 1996.

16. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

FLOWCHART



17. Revisions

Document Number	Reason for Change	Date
TMP_ALL_Q_001_Rev.01	Restructured document format. Added tables and other content to create an "SOP Template" in which local edits are allowed. Modified signature page to include department manager and SOP template number. Updated SOP template number. Made the tuning criteria red so edits can be made to reflect local tuning criteria used. Updated calibration sections with new calibration policies described in the PASI Quality Manual revision 10.0.	14July2006
S-GB-ALL-O-002-Rev.00	Added necessary information for the Green Bay Laboratory	19Mar2008
S-GB-ALL-O-002-Rev.01	Removed Section 7 Responsibilities and distribution. Added in section 11.4.1.3.1 Low Level Soils to determine corrective action for samples received with greater than 10 g of soil. Renumbered document. Up-dated section references in document. Table 8.1 – updated serial numbers. Tables 9.6, 9.7 and 10.1 – VSTD300 Standard added for the Water Curve.	12Dec2008
S-GB-ALL-O-002-Rev.02	Updated Signature page to Periodic Review. Included Listing of Analyte to Scope and Application. Included note to address State of SC disallowance of Non-linear regression.	21May2009
S-GB-O-056-Rev.00	Section 13.3 – Updated Method Modification to incorporate Method 5035. Table 10.2 – Included client specific acceptance criterion. Included Non-Target CCC acceptance criteria Added Attachment II: Client Specific Requirements. Updated Acrolein vendor information Deleted Sections 11.4.1.3.2.3.1-11.4.1.3.2.3.18 Added 40MSVA to instruments Table 8.1: Added 40MSVA serial numbers	22Apr2010
S-GB-O-056-Rev.01	Table 10.1, Page 28 of 51: Corrected trans-1,2-Dichloroethane to trans1,2-Dichloroethene Section 10.2.3: Updated reference to Section 10.2.3 Section 11.2: Updated reference from Table 9.2 to Section 11.2 Section 11.4.1.4.2.2.2: Removed reference for Table 1	07Jul2010
S-GB-O-056-Rev.02	Table 7.1: Added Styrene and Vinyl Chloride to analytes requiring an unpreserved sample. Table 10.2: Added samples analyzed from the State of South Carolina non-CCCs require ±30% Drift. Section 13.3: Added Note that method modification of 10g:10mL is not acceptable for samples reported to South Carolina. Table 8.1: Changed 40MSVA Tekmar 3000 to a Tekmar Stratum	13Sept2010
S-GB-O-056-Rev.03	Section 11: Removed check for effervescence. Section 11: Added note about average VOA methanol preserved vial weight.	10Mar2011

Attachment II:

Throughout document, reference to Client Specific requirements refers to samples analyzed following the BP Technical Requirements Version 9.0

Appendix D – Pace Analytical Services, Inc Information
(Enclosed on CD)

File Name	Description
Illinois 20050 exp 8-3-12.pdf	IEPA Certification
Quality Manual Rev 14.pdf	Quality Assurance Manual
S-ALL-GB-I-015-Rev.01 (pH).pdf	SOP – SW-846 Method 9040B and 9045C
S-GB-C-008-Rev.01 (% Moisture).pdf	SOP -- ASTM D 2974-87
S-GB-I-002-Rev.05 (Ignitability-SW846 1010A).pdf	SOP – SW-846 Method 1010A
S-GB-I-013-Rev.02 (Paint Filter_9095A).pdf	SOP – SW-846 Method 9095A
S-GB-I-037-Rev.03 (Walkley Black).pdf	SOP -- TOC
S-GB-I-059-Rev.02 (Apollo TOC by Tekmar Fusion).pdf	SOP -- EPA Method 9060A
S-GB-I-063-Rev.00 (TOC by Tekmar Fusion).pdf	SOP – TOC SW-846 Method 9060A Tekmar
S-GB-I-064-Rev.00 (SmartChem CN_9012A).pdf	SOP – SW-846 Method 9012A and EPA 335.4
S-GB-I-067-Rev.00 (TCLP, SPLP,ASTM).pdf	SOP – SW-846 Method 1311 and 1312
S-GB-I-068-Rev.00 (TSS, TDS, TVS, OM, FOC).pdf	SOP -- Method 2540, STM D2974 and EPA 160.4
S-GB-M-004-Rev.03 Mercury 7470A_7471A_B.pdf	SOP – SW-846 Method 7470A & 7471 A/B
S-GB-M-005-Rev.03 (6010C).pdf	SOP -- SW-846 Method 5030B and 5035
S-GB-O-005-Rev.03 (SW846-5035-5030B).pdf	SOP -- SW-846 Method 6010C with 3010A and 3050B
S-GB-O-008-Rev.04 (SW846-8015).pdf	SOP – SW-846 Method 8015C
S-GB-O-010-Rev.03 (SW846-5030B Aq Sample Prep).pdf	SOP – SW-846 Method 5030B
S-GB-O-023-Rev.04 (TPHSOP).pdf	SOP – SW-846 Method 8015B
S-GB-O-027-Rev.03 (Pesticides SOP).pdf	SOP – SW-846 Method 8081A
S-GB-O-041-Rev.03 (PCBs By 3541).pdf	SOP – SW-846 Method 3541
S-GB-O-045-Rev.03 (Microwave Extraction for PAH, BNA, and TPH So).pdf	SOP – SW-846 Method 3546
S-GB-O-047-Rev.02 (PCB 8082A SOP).pdf	SOP – SW-846 Method 8082A
S-GB-O-049-Rev.03 (8270 SVOA).pdf	SOP – SW-846 Method 8270

S-GB-O-050-Rev.00 (8270 SVOA SIM).pdf	SOP – SW-846 Method 8270 sim
S-GB-O-053-Rev.00 (Sep Funnel Extraction).pdf	SOP – EPA Method 3510
S-GB-O-054-Rev.00 (Ultrasonic Extraction).pdf	SOP – EPA Method 3550
S-GB-O-056-Rev.02 (VOA Analytical_Prep).pdf	SOP – SW-846 Method 8260B w/ 5030B & 5035

Appendix E

Appendix E – STAT Analysis Corporation Information
(Enclosed on CD)

File Name	Description
1. 4000 VOC - 8260B.pdf	SOP – SW-846 Method 8260B w/ 5030B & 5035
1a. Sop 4000add.pdf	VOC Addendum
2. 4020--SVOC.pdf	SOP – SW-846 Method 8270
2. Sop 4020add.pdf	SVOC Addendum
3.4500-pna.pdf	SOP – SW-846 Method 8270 SIM
4. 4050 PCBs.pdf	SOP – SW-846 Method 8081A-8082
4a. ADDENDUM 022908.pdf	PCB Addendum 1
4b. PCB4050ADDendum090810.pdf	PCB Addendum 2
5. 4510--metals.pdf	SOP – SW-846 Method 6020
5a. Addendum 1 - metals.pdf	Metals Addendum 1
5b. Addendum 2 - metals.pdf	Metals Addendum 2
6. SOP4600 Hexachrome.pdf	SOP -- Hex Chrom SW-846 Method 7196A
6a. sop4600add--Hexachrome.pdf	Hex Chrom Addendum
7. 4530 mercury.pdf	SOP – EPA Method 7470A & 7471A
8. 4710--CN.pdf	SOP -- SW-846 9012A
8a. cn - distillation--3610	SOP – EPA Method 9012A
9. 2125--TCLP.pdf	SOP -- EPA Method 1311 and 1312
9a. Sop2125add.PDF	Leaching Procedures Addendum
10. TPH 4090	SOP – EPA Method 8015B
11. sop4210 pH r01.pdf	SOP – SW-846 Method 9040B
12. sop4630toc.pdf	SOP -- EPA Method 415.1
13. 4480--moisture FOC.pdf	SOP – SW-846 Method 3550B
14. 2010 Paintfilter.pdf	SOP – EPA Method 9095A
15. 3615 RCNS dist.pdf	SOP -- EPA Method SW846 Chapter 7
16. SOP 5100 pcm.pdf	SOP -- Asbestos
16a. pcm add1.pdf	Asbestos Addendum 1
16b. pcm add2.pdf	Asbestos Addendum 2
17. 4550 LEAD BY FLAA RO5.pdf	Lead by AADA
18. 3005-metal aqua extr.pdf	SOP – SW-846 Method 3005A
19. 3110--metal solid extr.pdf	SOP – SW-846 Method 3050B
19a. Sop3110add.PDF	Metal Solid Extraction Addendum
20. 3500--Org.Extraction.pdf	SOP – SW-846 Method 3510C, 3520C, etc
20a. Org prep3500 Addendum.pdf	Organic Extraction Addendum
21. 4725 reactive sulfide.pdf	SOP - EPA Method 376.2 and 9034
22. 3620 phenolics dist.pdf	SOP -- EPA Method 9065
nvlap-23.pdf	NVLAP Certificate
QA 001 QA MANUAL Rev 09.pdf	QA Manual
STAT AnalysisIEPA CertList.pdf	IEPA Certification List

Appendix F

Appendix F – Field Data Sheets

Terracon Field Log
Terracon Field Boring Log
Terracon Well Construction Diagram
Pace Analytical Services Chain of Custody
Stat Analysis Corporation Chain of Custody
Laboratory Container Sample Label

DAILY FIELD LOG

PROJECT NAME _____ PROJECT NO. _____

PROJECT LOCATION _____

WEATHER _____

EQUIPMENT _____

FORM COMPLETED BY _____ DATE _____

WORK PERFORMED: _____

Terracon

LOG OF BORING NO. B-1

CLIENT

SITE

PROJECT

GRAPHIC LOG	DESCRIPTION	DEPTH, ft.	USCS SYMBOL	SAMPLES NUMBER	SAMPLES TYPE	SAMPLES RECOVERY, in.	SAMPLES SPT - N BLOWS / ft.	TESTS WATER CONTENT, %	TESTS DRY UNIT WT pcf	TESTS UNCONFINED STRENGTH, psf	PID, (ppm)
	0			1	DT						
		5		2	DT						
		10		3	DT						
		15		4	DT						
				5	DT						
	BOTTOM OF BORING	20									

The stratification lines represent the approximate boundary lines between soil and rock types: In-situ, the transition may be gradual.

WATER LEVEL OBSERVATIONS, ft		
WL	▽	▽
WL	▽	▽
WL		

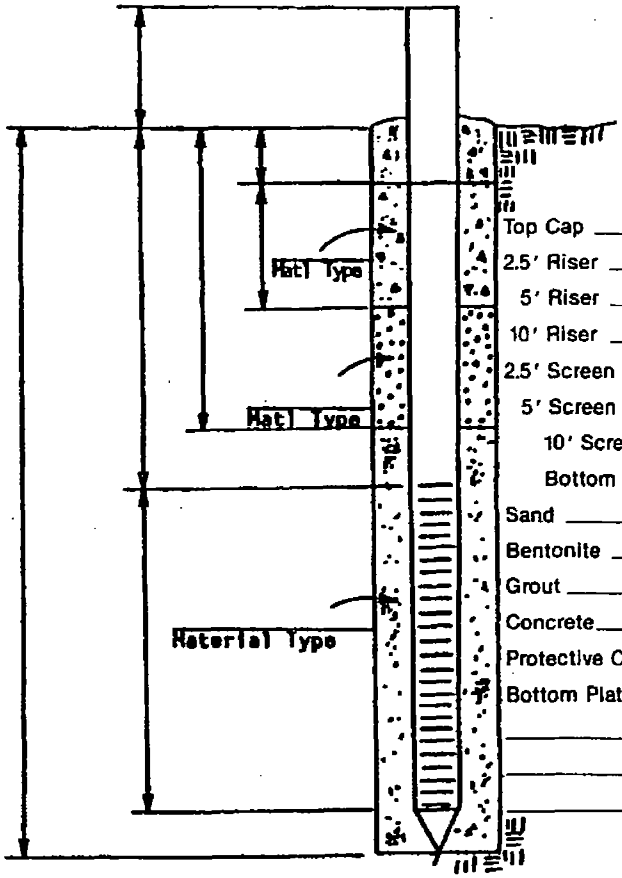
Terracon

BORING STARTED	
BORING COMPLETED	
RIG	FOREMAN
APPROVED	JOB #

BORE2 110S000.GPJ TERRACON.GDT 4/1/07

MONITORING WELL DETAILS

1. Fill in applicable dimensions and identify backfill materials
2. Sketch screen installation (if more than one section used)



Top Cap ______________________

2.5' Riser ______________________

5' Riser ______________________

10' Riser ______________________

2.5' Screen ______________________

5' Screen ______________________

10' Screen ______________________

Bottom Point ______________________

Sand ______________________

Bentonite ______________________

Grout ______________________

Concrete ______________________

Protective Casing ______________________

Bottom Plates ______________________

____

____

____

Protective steel cover? ☐ Yes (Lock Type)______________________)

Type ______________ (Key number______________________)

☐ No

Screen slot size or fabric type ______________________

Well Installation Accepted by ______________________ Driller ____________

BORING ELEVATIONS

Benchmark ______________________

____

Elevation ______________ ☐ Assumed ☐ Given

Boring/ Turning Point	Backsight	Height Instrument	Foresight	Elevation

Pace Analytical™
www.pacelabs.com

CHAIN-OF-CUSTODY / Analytical Request Document

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

Page: of

Section A Required Client Information:

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 ☐ GA ☐ IL ☐ IN ☐ MI ☐ NC ☐ OH ☐ SC ☐ WI ☐ OTHER___

Section D Required Client Information

SAMPLE ID

One Character per box.
(A-Z, 0-9 / ,-)
Sample IDs MUST BE UNIQUE

Valid Matrix Codes

MATRIX	CODE
DRINKING WATER	DW
WATER	WT
WASTE WATER	WW
PRODUCT	P
SOIL/SOLID	SL
OIL	OL
WIPE	WP
AIR	AR
OTHER	OT
TISSUE	TS

ITEM #	MATRIX CODE	SAMPLE TYPE G=GRAB C=COMP	COLLECTED COMPOSITE START DATE	COLLECTED COMPOSITE START TIME	COLLECTED COMPOSITE END/GRAB DATE	COLLECTED COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Requested Analysis:	Residual Chlorine (Y/N)	Pace Project No. Lab I.D.
1																			
2																			
3																			
4																			
5																			
6																			
7																			
8																			
9																			
10																			
11																			
12																			

Filtered (Y/N)

Additional Comments:

RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS			
							Y/N	Y/N	Y/N
							Y/N	Y/N	Y/N
							Y/N	Y/N	Y/N
							Y/N	Y/N	Y/N

SAMPLER NAME AND SIGNATURE		Temp in °C	Received on Ice	Custody Sealed Cooler	Samples Intact
PRINT Name of SAMPLER:					
SIGNATURE of SAMPLER:	DATE Signed (MM / DD / YY)				

STA Analysis Corporation
2242 W. Harrison Suite 200, Chicago, Illinois 60612 Phone: (312) 733-0551 Fax: (312) 733-2386
e-mail address: STATinfo@STATAnalysis.com AIHA, NVLAP and NELAP accredited

CHAIN OF CUSTODY RECORD

N°: _____ **Page :** _____ **of** _____

Company:

Project Number: Client Tracking No.:

Project Name:

Project Location:

Sampler(s):

Report To: _____ Phone: _____

_____ Fax: _____

QC Level: 1 ____ 2 ____ 3 ____ 4 ____ e-mail: _____

P.O. No.:

Quote No.:

Turn Around:

Results Needed:

/ / am/pm

Client Sample Number/Description:	Date Taken	Time Taken	Matrix	Comp.	Grab	Preserv.	No. of Containers	Remarks	Lab No.:

Relinquished by: (Signature) Date/Time:

Received by: (Signature) Date/Time:

Relinquished by: (Signature) Date/Time:

Received by: (Signature) Date/Time:

Relinquished by: (Signature) Date/Time:

Received by: (Signature) Date/Time:

Comments:

Preservation Code: A = None B = HNO_3 C = NaOH
D = H_2SO_4 E = HCl F = 5035/EnCore G = Other

Laboratory Work Order No.:

Received on Ice: Yes ☐ No ☐

Temperature: _____ °C

Pace Analytical Services, Inc.
1241 Bellevue Street, Ste. 9
Green Bay, WI 54302
(920) 469-2436

Client: _____

Client Sample ID: _____

Date Collected: _____ Time: _____

Collected by: _____

Analysis: _____

Preservatives: ☐None ☐HNO_3 ☐H_2SO_4 ☐NaOH ☐HCl

☐$Na_2S_2O_3$ ☐MeOH ☐Zn Acetate ☐Other: _____

Sample ID: 11050814-001B

ClientSampID: EE-EW
Received: 5/20/2011 2:50:00 PM
Collected: 5/20/2011 10:00:00 AM
Stored: R10
Bottle: 1 of 1
TESTS:
3550_PNA PMOIST
PNA_SOIL

Appendix G

Appendix G – Lab Certifications

STATE OF ILLINOIS

ENVIRONMENTAL PROTECTION AGENCY
NELAP - RECOGNIZED
ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

PACE ANALYTICAL SERVICES, INC. (BELLEUVE ST.)

1241 BELLEVUE STREET

GREEN BAY, WI 54302

NELAP ACCREDITED
ACCREDITATION NUMBER #200050

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: FL Department of Health, Bureau of Laboratories

Gary Germann
Manager
Environmental Laboratory Accreditation Program

Janet Cruse
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 002756
Expiration Date: 08/03/2012
Issued On: 07/26/2011

State of Illinois
Environmental Protection Agency

Certificate No.: 002756

Awards the Certificate of Approval

Pace Analytical Services, Inc. (Belleuve St.)
1241 Bellevue Street
Green Bay, WI 54302

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, Inorganic

1010

Ignitability

1311

TCLP (Organic and Inorganic)

1312

Synthetic Precipitation Leaching Procedure

6010C

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

6020A

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

7470A

Mercury

7471B

Mercury

9012A

Cyanide

9040B

Hydrogen Ion (pH)

9045C

Hydrogen Ion (pH)

State of Illinois
Environmental Protection Agency

Certificate No.: 002756

Awards the Certificate of Approval

Pace Analytical Services, Inc. (Belleuve St.)
1241 Bellevue Street
Green Bay, WI 54302

Hazardous and Solid Waste, Inorganic	*9056 (NPW Matrix Only)*	
Bromide	Chloride	Fluoride
Nitrate	Nitrite	Sulfate
9060		
Total Organic Carbon (TOC)		
9095A		
Paint Filter		
Hazardous and Solid Waste, Organic		
8015B		
Diesel range organics (DRO)	Gasoline range organics (GRO)	
8021B		
1,2,4-Trimethylbenzene	1,3,5-Trimethylbenzene	Benzene
Ethylbenzene	MTBE (Methyl-t-butyl ether)	m-Xylene
Naphthalene	o-Xylene	p-Xylene
Toluene	Total Xylenes	
8081A		
Toxaphene	trans-Nonachlor	
8081B		
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	Methoxychlor
Mirex		
8082A		
PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260		
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
1,3-Dichloropropane	1,4-Dichlorobenzene	1,4-Dioxane
1-Propanol	2,2-Dichloropropane	2-Butanone (Methyl ethyl ketone, MEK)
2-Chloroethyl vinyl ether	2-Chlorotoluene	2-Hexanone
2-Methyl-1-propanol (Isobutyl alcohol)	2-Propanol (Isopropyl alcohol)	4-Chlorotoluene
4-Methyl-2-pentanone (Methyl isobutyl ketone, I	Acetone	Acetonitrile
Acrolein (Propenal)	Acrylonitrile	Allyl chloride
Benzene	Bromobenzene	Bromochloromethane
Bromodichloromethane	Bromoform	Bromomethane

State of Illinois
Environmental Protection Agency

Certificate No.: 002756

Awards the Certificate of Approval

Pace Analytical Services, Inc. (Belleuve St.)
1241 Bellevue Street
Green Bay, WI 54302

Hazardous and Solid Waste, Organic	*8260B*	Carbon disulfide
Carbon tetrachloride	Chlorobenzene	Chlorodibromomethane (Dibromochlorometha
Chloroethane	Chloroform	Chloromethane
Chloroprene	cis-1,2-Dichloroethene	cis-1,3-Dichloropropene
cis-1,4-Dichloro-2-butene	Dibromomethane	Dichlorodifluoromethane
Dichloromethane (Methylene chloride)	Diethyl ether	Ethanol
Ethyl acetate	Ethyl methacrylate	Ethylbenzene
Hexachlorobutadiene	Hexachloroethane	Isopropylbenzene
Methacrylonitrile	Methyl ethyl ketone	Methyl iodide (Iodmethane)
Methyl methacrylate	Methyl-t-butyl ether	m-Xylene
Naphthalene	n-Butanol	n-Butylbenzene
n-Propylbenzene	o-Xylene	p-Isopropyltoluene
Propionitrile (Ethyl cyanide)	p-Xylene	sec-Butylbenzene
Styrene	t-Butyl alcohol	tert-Butylbenzene
Tetrachloroethene	Toluene	trans-1,2-Dichloroethene
trans-1,3-Dichloropropene	trans-1,4-Dichloro-2-butene	Trichloroethene
Trichlorofluoromethane	Vinyl acetate	Vinyl chloride
Xylenes (total)		
8270C		
1,2-Diphenylhydrazine	1,3-Dichlorobenzene	1,4-Dichlorobenzene
2,3,4,6-Tetrachlorophenol	2,4,5-Trichlorophenol	2,4,6-Trichlorophenol
2,4-Dichlorophenol	2,4-Dimethylphenol	2,4-Dinitrophenol
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene
2-Chlorophenol	2-Methylnaphthalene	2-Methylphenol (o-Cresol)
2-Nitroaniline	2-Nitrophenol	3,3'-Dichlorobenzidine
3-Methylphenol (m-Cresol)	3-Nitroaniline	4,6-Dinitro-2-methylphenol
4-Bromophenyl phenyl ether	4-Chloro-3-methylphenol	4-Chloroaniline
4-Chlorophenyl phenyl ether	4-Methylphenol (p-Cresol)	4-Nitroaniline
4-Nitrophenol	Acenaphthene	Acenaphthylene
Acetophenone	Aniline	Anthracene
Benzidine	Benzo(a)anthracene	Benzo(a)pyrene
Benzo(b)fluoranthene	Benzo(g,h,i)perlyene	Benzo(k)fluoranthene
Benzoic acid	Benzyl alcohol	Bis(2-chloroethoxy) methane
Bis(2-chloroethyl) ether	Bis(2-chloroisopropyl) ether	Bis(2-ethylhexyl) phthalate
Butyl benzyl phthalate	Carbazole	Chrysene
Dibenz(a,h)anthracene	Dibenzofuran	Diethyl phthalate
Dimethyl phthalate	Di-n-butyl phthalate	Di-n-octyl phthalate
Fluoranthene	Fluorene	Hexachlorobenzene
Hexachlorobutadiene	Hexachlorocyclopentadiene	Hexachloroethane
Indeno(1,2,3-cd) pyrene	Isophorone	m-Cresol (3-Methylphenol)
Naphthalene	Nitrobenzene	N-Nitrosodimethylamine
N-Nitrosodi-n-propylamine	N-Nitrosodiphenylamine	o-Cresol (2-Methylphenol)
p-Cresol (4-Methylphenol)	Pentachlorophenol	Phenanthrene
Phenol	Pyrene	Pyridine
Wastewater, Inorganic		
SM2130B,18Ed		

State of Illinois
Environmental Protection Agency

Certificate No.: 002756

Awards the Certificate of Approval

Pace Analytical Services, Inc. (Belleuve St.)
1241 Bellevue Street
Green Bay, WI 54302

Wastewater, Inorganic	*SM2130B,18Ed*	Turbidity
SM2310B4a,20Ed		
Acidity		
SM2340B,18Ed		
Hardness		
SM2540B,20Ed		
Residue (Total)		
SM2540C,20Ed		
Residue (TDS)		
SM2540D,20Ed		
Residue (TSS)		
SM3500Cr-B,20Ed		
Chromium VI		
SM4500H-B,20Ed		
Hydrogen Ion (pH)		
SM4500S-F,19Ed		
Sulfide		
SM5210B,18Ed		
Biochemical Oxygen Demand (BOD)	Carbonaceous Biochemical Oxygen Demand (C	
SM5310C,20Ed		
Total Organic Carbon (TOC)		
USEPA120.1		
Specific Conductance		
USEPA150.1		
Hydrogen Ion (pH)		
USEPA160.4		
Residue (Volatile)		
USEPA1631E		
Mercury		
USEPA300.0R2.1		
Bromide	Chloride	Fluoride
Nitrate	Nitrate-Nitrite (sum)	Nitrite
Sulfate		
USEPA310.2		
Alkalinity		
USEPA335.4R1.0		
Cyanide		
USEPA350.1R2.0		
Ammonia		
USEPA351.2R2.0		
Total Kjeldahl Nitrogen		
USEPA365.4		

State of Illinois
Environmental Protection Agency

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Pace Analytical Services, Inc. (Belleuve St.)
1241 Bellevue Street
Green Bay, WI 54302

Wastewater, Inorganic	*USEPA365.4*	Phosphorus
USEPA410.4R2.0		
Chemical Oxygen Demand (COD)		
Wastewater, Organic		
USEPA602		
Benzene	Ethylbenzene	Toluene
USEPA624		
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1-Dichloroethane	1,1-Dichloroethene	1,2-Dichlorobenzene
1,2-Dichloroethane	1,2-Dichloropropane	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2-Chloroethylvinyl ether	Acrolein (Propenal)
Acrylonitrile	Benzene	Bromodichloromethane
Bromoform	Bromomethane	Carbon tetrachloride
Chlorobenzene	Chloroethane	Chloroform
Chloromethane	cis-1,3-Dichloropropene	Dibromochloromethane
Dichloromethane (Methylene chloride)	Ethylbenzene	Tetrachloroethene
Toluene	trans-1,2-Dichloroethene	trans-1,3-Dichloropropene
Trichloroethene	Trichlorofluoromethane	Vinyl chloride
Xylenes (total)		
USEPA625		
1,2,4-Trichlorobenzene	1,2-Dichlorobenzene	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2,2-Oxybis(1-chloropropane)	2,4,6-Trichlorophenol
2,4-Dichlorophenol	2,4-Dimethylphenol	2,4-Dinitrophenol
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene
2-Chlorophenol	2-Methyl-4,6-dinitrophenol	2-Nitrophenol
3,3'-Dichlorobenzidine	4-Bromophenyl phenyl ether	4-Chloro-3-methylphenol
4-Chlorophenyl phenyl ether	4-Nitrophenol	Acenaphthene
Acenaphthylene	Anthracene	Benzidine
Benzo(a)anthracene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene	Benzyl butyl phthalate
Bis(2-chloroethoxy) methane	Bis(2-chloroethyl) ether	Bis(2-ethylhexyl) phthalate
Chrysene	Dibenz(a,h)anthracene	Diethyl phthalate
Dimethyl phthalate	Di-n-butyl phthalate	Di-n-octyl phthalate
Fluoranthene	Fluorene	Hexachlorobenzene
Hexachlorobutadiene	Hexachlorocyclopentadiene	Hexachloroethane
Indeno(1,2,3-cd) pyrene	Isophorone	Naphthalene
Nitrobenzene	N-Nitrosodimethylamine	N-Nitrosodi-n-propylamine
N-Nitrosodiphenylamine	Pentachlorophenol	Phenanthrene
Phenol	Pyrene	

STATE OF ILLINOIS

ENVIRONMENTAL PROTECTION AGENCY
NELAP - RECOGNIZED

ENVIRONMENTAL LABORATORY ACCREDITATION

is hereby granted to

STAT ANALYSIS CORPORATION

2242 WEST HARRISON STREET

CHICAGO, IL 60612

NELAP ACCREDITED

ACCREDITATION NUMBER #100445

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.

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Gary Germann
Manager
Environmental Laboratory Accreditation Program

Janet Cruse
Accreditation Officer
Environmental Laboratory Accreditation Program

Certificate No.: 002618
Expiration Date: 09/30/2011
Issued On: 09/30/2010

State of Illinois
Environmental Protection Agency

Certificate No.: 002618

Awards the Certificate of Approval

STAT Analysis Corporation
2242 West Harrison Street
Chicago, IL 60612

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.

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Hazardous and Solid Waste, Inorganic

1010

Ignitability

1311

TCLP (Organic and Inorganic)

1312

Synthetic Precipitation Leaching Procedure

6020

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

7000B

Lead

7196A

Chromium VI

7420

Lead

7470A

Mercury

7471A

Mercury

9012A

Cyanide

9020B

TOX (Total Organic Halides)

9023

EOX-Extractable Organic Halides

9040C

Hydrogen Ion (pH)

9045C

Hydrogen Ion (pH)

State of Illinois
Environmental Protection Agency

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STAT Analysis Corporation
2242 West Harrison Street
Chicago, IL 60612

Hazardous and Solid Waste, Inorganic	*9045D*	
Hydrogen Ion (pH)		
9066		
Phenolics		
9095A		
Paint Filter		
9095B		
Paint Filter		
Chapter 7/9012A		
Reactive Cyanide		
Chapter 7/9034		
Reactive Sulfide		
Hazardous and Solid Waste, Organic		
8015B		
Diesel range organics (DRO)	Ethylene glycol	Gasoline range organics (GRO)
8015C		
Diesel range organics (DRO)	Gasoline range organics (GRO)	
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	Methoxychlor
Toxaphene		
8081B		
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	Methoxychlor
Toxaphene		
8082		
PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260		
8082A		
PCB-1016	PCB-1221	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260		
8260B		

State of Illinois
Environmental Protection Agency

Certificate No.: 002618

Awards the Certificate of Approval

STAT Analysis Corporation
2242 West Harrison Street
Chicago, IL 60612

Hazardous and Solid Waste, Organic	*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	1,3-Dichloropropane
1,4-Dichlorobenzene	1,4-Dioxane	2,2-Dichloropropane
2-Butanone (Methyl ethyl ketone, MEK)	2-Chloroethyl vinyl ether	2-Chlorotoluene
2-Hexanone	2-Methyl-1-propanol (Isobutyl alcohol)	2-Nitropropane
4-Chlorotoluene	4-Methyl-2-pentanone (Methyl isobutyl ketone, I	Acetone
Acrolein (Propenal)	Acrylonitrile	Benzene
Bromobenzene	Bromochloromethane	Bromodichloromethane
Bromoform	Bromomethane	Carbon disulfide
Carbon tetrachloride	Chlorobenzene	Chlorodibromomethane (Dibromochlorometha
Chloroethane	Chloroform	Chloromethane
cis-1,2-Dichloroethene	cis-1,3-Dichloropropene	Dibromomethane
Dichlorodifluoromethane	Dichloromethane (Methylene chloride)	Diethyl ether
Ethyl acetate	Ethyl ether	Ethylbenzene
Isopropylbenzene	Methyl ethyl ketone	Methyl isobutyl ketone
Methyl-t-butyl ether	m-Xylene	Naphthalene
n-Butanol	n-Butylbenzene	o-Xylene
p-Isopropyltoluene	p-Xylene	sec-Butylbenzene
Styrene	tert-Butylbenzene	Tetrachloroethene
Toluene	trans-1,2-Dichloroethene	trans-1,3-Dichloropropene
Trichloroethene	Trichlorofluoromethane	Trichlorotrifluoroethane
Vinyl acetate	Vinyl chloride	Xylenes (Total)
8270C		
1,2,4-Trichlorobenzene	1,2-Dichlorobenzene	1,2-Diphenylhydrazine
1,3-Dichlorobenzene	1,4-Dichlorobenzene	1,4-Dinitrobenzene
1-Methylnaphthalene	2,4,5-Trichlorophenol	2,4,6-Trichlorophenol
2,4-Dichlorophenol	2,4-Dimethylphenol	2,4-Dinitrophenol
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene
2-Chlorophenol	2-Methylnaphthalene	2-Methylphenol
2-Nitroaniline	2-Nitrophenol	3,3'-Dichlorobenzidine
3-Nitroaniline	4,6-Dinitro-2-methylphenol	4-Bromophenyl phenyl ether
4-Chloro-3-methylphenol	4-Chloroaniline	4-Chlorophenyl phenyl ether
4-Methylphenol	4-Nitroaniline	4-Nitrophenol
Acenaphthene	Acenaphthylene	Aniline
Anthracene	Benzidine	Benzo(a)anthracene
Benzo(a)pyrene	Benzo(b)fluoranthene	Benzo(g,h,i)perlyene
Benzo(k)fluoranthene	Benzoic acid	Benzyl alcohol
Bis(2-chloroethoxy) methane	Bis(2-chloroethyl) ether	Bis(2-chloroisopropyl) ether
Bis(2-ethylhexyl) phthalate	Butyl benzyl phthalate	Carbazole
Chrysene	Dibenz(a,h)anthracene	Dibenzofuran
Diethyl phthalate	Dimethyl phthalate	Di-n-butyl phthalate
Di-n-octyl phthalate	Diphenylamine	Fluoranthene

STAT Analysis Corporation
2242 West Harrison Street
Chicago, IL 60612

Hazardous and Solid Waste, Organic	*8270C*	Fluorene
Hexachlorobenzene	Hexachlorobutadiene	Hexachlorocyclopentadiene
Hexachloroethane	Indeno(1,2,3-cd) pyrene	Isophorone
m-Cresol (3-Methylphenol)	Naphthalene	Nitrobenzene
N-Nitrosodimethylamine	N-Nitrosodi-n-propylamine	N-Nitrosodiphenylamine
o-Cresol (2-Methylphenol)	p-Cresol (4-Methylphenol)	Pentachlorophenol
Phenanthrene	Phenol	Pyrene
Pyridine		
8270D		
1,2,4-Trichlorobenzene	1,2-Dichlorobenzene	1,2-Diphenylhydrazine
1,3-Dichlorobenzene	1,4-Dichlorobenzene	1,4-Dinitrobenzene
1-Methylnaphthalene	2,4,5-Trichlorophenol	2,4,6-Trichlorophenol
2,4-Dichlorophenol	2,4-Dimethylphenol	2,4-Dinitrophenol
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene
2-Chlorophenol	2-Methylnaphthalene	2-Methylphenol (o-Cresol)
2-Nitroaniline	2-Nitrophenol	3,3'-Dichlorobenzidine
3-Nitroaniline	4,6-Dinitro-2-methylphenol	4-Bromophenyl phenyl ether
4-Chloro-3-methylphenol	4-Chloroaniline	4-Chlorophenyl phenyl ether
4-Nitroaniline	4-Nitrophenol	Acenaphthene
Acenaphthylene	Aniline	Anthracene
Benzidine	Benzo(a)anthracene	Benzo(a)pyrene
Benzo(b)fluoranthene	Benzo(g,h,i)perlyene	Benzo(k)fluoranthene
Benzoic acid	Benzyl alcohol	Bis(2-chloroethoxy) methane
Bis(2-chloroethyl) ether	Bis(2-chloroisopropyl) ether	Bis(2-ethylhexyl) phthalate
Butyl benzyl phthalate	Carbazole	Chrysene
Dibenz(a,h)anthracene	Dibenzofuran	Diethyl phthalate
Dimethyl phthalate	Di-n-butyl phthalate	Di-n-octyl phthalate
Diphenylamine	Fluoranthene	Fluorene
Hexachlorobenzene	Hexachlorobutadiene	Hexachlorocyclopentadiene
Hexachloroethane	Indeno(1,2,3-cd) pyrene	Isophorone
m-Cresol (3-Methylphenol)	Naphthalene	Nitrobenzene
N-Nitrosodimethylamine	N-Nitrosodi-n-propylamine	N-Nitrosodiphenylamine
o-Cresol (2-Methylphenol)	p-Cresol (4-Methylphenol)	Pentachlorophenol
Phenanthrene	Phenol	Pyrene
Pyridine		
8321A		
2,4,5-T	2,4,5-TP (Silvex)	2,4-D
2,4-DB	Dalapon	Dicamba
Dichlorprop	Dinoseb	MCPA
MCPP		
Wastewater, Inorganic		
SM2320B,20Ed		
Alkalinity		
SM2340B,20Ed		
Hardness		
SM2510B,20Ed		

State of Illinois
Environmental Protection Agency

Certificate No.: 002618

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2242 West Harrison Street
Chicago, IL 60612

Wastewater, Inorganic	*SM2510B,20Ed*	Specific Conductance
SM2540C,20Ed		
Residue (TDS)		
SM2540D,20Ed		
Residue (TSS)		
SM2540G,18Ed		
Residue (% solids)		
SM4500Cl-G,20Ed		
Chlorine		
SM4500CN-CE,20Ed		
Cyanide		
SM4500NH3-C,20Ed		
Ammonia		
SM4500NO3F,20Ed		
Nitrate	Nitrate-Nitrite (sum)	Nitrite
SM4500P-E,20Ed		
Phosphorus		
SM5210B,20Ed		
Biochemical Oxygen Demand (BOD)	Carbonaceous Biochemical Oxygen Demand (C	
USEPA120.1		
Specific Conductance		
USEPA160.1		
Residue (TDS)		
USEPA160.2		
Residue (TSS)		
USEPA1664RA		
Oil and Grease		
USEPA200.8R5.4		
Aluminum	Antimony	Arsenic
Barium	Beryllium	Boron
Cadmium	Chromium	Cobalt
Copper	Hardness (calc.)	Iron
Lead	Manganese	Molybdenum
Nickel	Selenium	Silver
Thallium	Tin	Vanadium
Zinc		
USEPA245.1R3.0		
Mercury		
USEPA410.4R2.0		
Chemical Oxygen Demand (COD)		
USEPA415.1		
Total Organic Carbon (TOC)		
Wastewater, Organic		

State of Illinois
Environmental Protection Agency

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Chicago, IL 60612

Wastewater, Organic

USEPA624

1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane	1,1,2-Trichloroethane
1,1-Dichloroethane	1,1-Dichloroethene	1,2-Dichlorobenzene
1,2-Dichloroethane	1,2-Dichloropropane	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2-Chloroethylvinyl ether	Acrolein (Propenal)
Acrylonitrile	Benzene	Bromodichloromethane
Bromoform	Bromomethane	Carbon tetrachloride
Chlorobenzene	Chloroethane	Chloroform
Chloromethane	cis-1,3-Dichloropropene	Dibromochloromethane
Dichloromethane (Methylene chloride)	Ethylbenzene	Methyl tert-butyl ether (MTBE)
Tetrachloroethene	Toluene	trans-1,2-Dichloroethene
trans-1,3-Dichloropropene	Trichloroethene	Trichlorofluoromethane
Vinyl chloride	Xylenes (total)	

USEPA625

1,2,4-Trichlorobenzene	1,2-Dichlorobenzene	1,3-Dichlorobenzene
1,4-Dichlorobenzene	2,4,5-Trichlorophenol	2,4,6-Trichlorophenol
2,4-Dichlorophenol	2,4-Dimethylphenol	2,4-Dinitrophenol
2,4-Dinitrotoluene (2,4-DNT)	2,6-Dinitrotoluene (2,6-DNT)	2-Chloronaphthalene
2-Chlorophenol	2-Methyl-4,6-dinitrophenol	2-Nitrophenol
3,3'-Dichlorobenzidine	4-Bromophenyl phenyl ether	4-Chloro-3-methylphenol
4-Chlorophenyl phenyl ether	4-Nitrophenol	Acenaphthene
Acenaphthylene	Anthracene	Benzidine
Benzo(a)anthracene	Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene	Benzyl butyl phthalate
Bis(2-chloroethoxy) methane	Bis(2-chloroethyl) ether	Bis(2-ethylhexyl) phthalate
Chrysene	Dibenz(a,h)anthracene	Diethyl phthalate
Dimethyl phthalate	Di-n-butyl phthalate	Di-n-octyl phthalate
Fluoranthene	Fluorene	Hexachlorobenzene
Hexachlorobutadiene	Hexachlorocyclopentadiene	Hexachloroethane
Indeno(1,2,3-cd) pyrene	Isophorone	Naphthalene
Nitrobenzene	N-Nitrosodimethylamine	N-Nitrosodi-n-propylamine
N-Nitrosodiphenylamine	Pentachlorophenol	Phenanthrene
Phenol	Pyrene	

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899

May 17, 2011

Dr. Pinaki Banerjee
HI-TEK Environmental, Inc. dba.
STAT Analysis Corporation
2242 W. Harrison
Chicago, IL 60612

NVLAP Lab Code: 101202-0

Dear Dr. Banerjee:

I am pleased to inform you that continuing accreditation for specific test methods in Bulk Asbestos Fiber Analysis (PLM) is granted to your organization under the National Voluntary Laboratory Accreditation Program (NVLAP). This accreditation is effective until June 30, 2012, provided that your organization continues to comply with accreditation requirements contained in the NVLAP Procedures.

Your Certificate of Accreditation is enclosed along with a statement of your Scope of Accreditation. You may reproduce these documents in their entirety and announce your organization's accreditation status using the NVLAP symbol and/or term in business publications, the trade press, and other business-oriented literature. Accreditation does not relieve your organization from observing and complying with any applicable existing laws and/or regulations.

We are pleased to have you participate in NVLAP and look forward to your continued association with this program. If you have any questions concerning your NVLAP accreditation, please direct them to Hazel M. Richmond, Program Manager, Laboratory Accreditation Program, National Institute of Standards and Technology, 100 Bureau Dr. Stop 2140, Gaithersburg, MD 20899-2140; (301) 975-4016.

Sincerely,

Sally S. Bruce

Sally S. Bruce, Chief
Laboratory Accreditation Program

Enclosure(s)

NVLAP® National Voluntary Laboratory Accreditation Program

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

STAT Analysis Corporation
STAT Analysis Corporation
2242 W. Harrison
Chicago, IL 60612
Dr. Pinaki Banerjee
Phone: 312-733-0551 Fax: 312-733-2386
E-Mail: Pbanerjee@STATAnalysis.com
URL: http://www.STATAnalysis.com

BULK ASBESTOS FIBER ANALYSIS (PLM) **NVLAP LAB CODE 101202-0**

NVLAP Code	*Designation / Description*
18/A01	EPA-600/M4-82-020: Interim Method for the Determination of Asbestos in Bulk Insulation Samples

2011-07-01 through 2012-06-30
Effective dates

Sally S. Bruce
For the National Institute of Standards and Technology

NVLAP-01S (REV. 2005-05-19)

United States Department of Commerce
National Institute of Standards and Technology

NVLAP®

Certificate of Accreditation to ISO/IEC 17025:2005

NVLAP LAB CODE: 101202-0

STAT Analysis Corporation
Chicago, IL

is accredited by the National Voluntary Laboratory Accreditation Program for specific services, listed on the Scope of Accreditation, for:

BULK ASBESTOS FIBER ANALYSIS

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communique dated January 2009).

2011-07-01 through 2012-06-30
Effective dates

Sally S. Bruce
For the National Institute of Standards and Technology

NVLAP-01C (REV. 2009-01-28)

Appendix H – Field Audit Corrective Action Form

QUALITY CONTROL FIELD AUDIT CORRECTIVE ACTION REPORT
Cherryvale Brownfields Project
EPA 7 Brownfields Assessment
No. 14107010

Property ID: _____

Property Name: _____

Terracon

Auditor Name: _____ **Title:** _____

Signature: _____ **Date/Time:** _____

Activity Audited (Give specific QAPP, Checklist or TSOP reference): _____

Position/Name With Immediate Responsiblity For Compliance: _____

Check and Initial As Appropriate:

- ☐ **Field Activity**
- ☐ **Management Activity**
- ☐ **File/Record Activity**
- ☐ **Found Compliant**
- ☐ **Found Non-Compliant**
- ☐ **Corrected, Copy To QA**

Describe Briefly: _____

Corrective Action Response (Describe in detail the remedy, including response consistent with Terracon Management Plan for reporting. Use Back If Needed):

Reported To: _____ Date/Time: _____

Project Title: _____ Response ID: _____

Check and Initial As Appropriate:

- ☐ Critical Measurement
- ☐ Qualitative Measurement
- ☐ Data Loss or Can't Be Repeated
- ☐ Entered To Field Logbook