Quality Assurance Project Plan

Downstream of Hayton Millpond Dam
Chilton, Calumet County, Wisconsin

January 2021
Revision 0

BRRTS No. 02-08-281506

Prepared For:

Tecumseh Products Company

Prepared By:

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230 W. Monroe Street, Suite 630
Chicago, IL 60606

TRC Project No: 320928.0000

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Approvals:

1/12/2021

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Date

1/12/2021

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Pace Analytical Services, LLC, Quality Assurance Officer

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List of Acronyms

APM	Analytical Project Manager
CFR	Code of Federal Regulations
DCBP	decachlorobiphenyl
DQO	Data Quality Objectives
FB	Field Blank
GC/ECD	Gas Chromatography with Electron Capture Detection
GPS	Global Positioning System
HARP	Hayton Area Remediation Project
ID	Identification
LCS	Laboratory Control Samples
LD	Laboratory Duplicate
mg/kg	Milligram per Kilogram
MS/MSD	Matrix Spike/Matrix Spike Duplicate
PAS	Pace Analytical Services, LLC
PCBs	Polychlorinated Biphenyls
PM	Project Manager
QA	Quality Assurance
QA/G-4	*Guidance for the Data Quality Objective Process* (USEPA guidance document)
QM	Quality Manual
QA/QC	Quality Assurance/Quality Control
QA/R-5	*Requirements for QA Project Plans* (USEPA requirements document)
QAPP	Quality Assurance Project Plan
QC	Quality Control
%R	Percent Recovery
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SIWP	Site Investigation Work Plan
SOP	Standard Operating Procedure
SOW	Scope of Work
SWAC	Surface-Area Weighted Average Concentration
Tecumseh	Tecumseh Products Company
TCMX	2,4,5,6-tetrachloro-m-xylene
TRC	TRC Environmental Corporation
TSCA	Toxic Substances Control Act
µg/kg	Micrograms per Kilogram
µg/L	Micrograms per Liter
USEPA	United States Environmental Protection Agency
WDNR	Wisconsin Department of Natural Resources

TABLE OF CONTENTS

TABLES

FIGURES

APPENDICES

1.0 Project Management

1.1 Introduction

TRC Environmental Corporation (TRC) has prepared this Quality Assurance Project Plan (QAPP) for the sampling activities downstream of the Hayton Millpond dam (Dam).

In November 2018, Wisconsin Department of Natural Resources (WDNR), Tecumseh Products Company (Tecumseh), and TRC Environmental Corporation (TRC) (for limited purposes) executed a Negotiated Agreement (BRRTS #02-08-281506) (Negotiated Agreement), in which Tecumseh agreed to certain response actions and obligations (WDNR, 2018). In accordance with Section III.K. of the Agreement, on January 8, 2019, Tecumseh submitted a sampling and analysis plan (SAP) to characterize the nature and extent of polychlorinated biphenyls (PCBs) below the Dam (Figure 1 and Figure 2). The WDNR reviewed the SAP and provided comments in a letter dated November 18, 2020. Comment #4 of the response required the update and expansion of the project QAPP to cover all data collection methods. A Site Investigation Work Plan (SIWP) and QAPP were required to be re-submitted within 60 days (January 15, 2021).

The Site includes the Hayton Area Remediation Project (HARP) and areas downstream of the Dam at the Hayton Millpond where hazardous substances attributable to the former Tecumseh manufacturing facility may have migrated (WDNR, 2018). TRC has an approved QAPP document to cover sampling and remediation activities within HARP (TRC, 2017). The area downstream of the Dam is located more than 8.5 miles downstream from the potential source, a former manufacturing facility in New Holstein, Wisconsin. Because the sample types and data quality objectives (DQOs) required for investigation of the South Branch Manitowoc River downstream of the Dam are different than those required for the HARP site remediation activities, TRC has elected to prepare this QAPP as a separate stand-alone QAPP for the downstream investigation.

This QAPP was prepared using the general guidance of the United States Environmental Protection Agency (USEPA) Requirements Quality Assurance Project Plans (QA/R-5) (USEPA, December 2002) and associated USEPA national and Region V guidance (USEPA, March 2001). QAPP revisions are submitted for informational purposes only to WDNR and are not specifically required by the Negotiated Agreement.

This QAPP Revision 0 describes the project objectives and organization, functional activities, and Quality Assurance (QA)/ Quality Control (QC) protocols that will be used to achieve the required DQOs. Specific protocols for sampling, sample handling and storage, sample numbering, chain-of-custody, and laboratory and field analyses to be performed as part of the site investigation of areas downstream of the Dam are also described in applicable SIWPs. The QA/QC procedures are in general accordance with applicable technical standards, WDNR requirements, regulations, and guidance. The procedures and methods presented in this QAPP provide for adequate data precision, accuracy, representativeness, comparability, and completeness for the investigation activities.

This QAPP is applicable for work to be performed by TRC and its representatives on the South Branch Manitowoc River downstream of the Dam. This QAPP is intended to support the sampling described in the 2021 SIWP (TRC, 2021), as well as any subsequent sampling programs, if any, of areas downstream of the Dam, with only minor revisions or attachments. This QAPP may be modified in the future as other sampling programs or projects are identified or defined.

1.2 Project Organization and Responsibilities

WDNR is the lead regulatory authority and is overseeing all aspects of work carried out as part of the investigation work downstream of the Dam. USEPA, although not a party to the Negotiated Agreement, has oversight and approval responsibilities through the federal Toxic Substances Control Act (TSCA). No prior sample results collected from sediment downstream of the Dan had concentrations greater than 50 parts per million (ppm). Tecumseh has the primary responsibility for investigation activities. TRC has been retained by Tecumseh to implement the investigation activities. The specific responsibility for each team member is described in detail below.

1.2.1 RA Management and Oversight Responsibilities

- **TRC Program Manager (PM)** – Chris Harvey, PE, TRC, Chicago, Illinois. The PM has the responsibility for ensuring that the project meets WDNR's objectives. The PM will report project status to the WDNR and is responsible for the overall management of the project, including all technical and financial aspects of the work.

- **TRC Quality Assurance Officer (QA Officer)** – Bruce Iverson, PE, TRC, Madison, Wisconsin. The QA Officer will remain independent of direct job involvement and day-to-day operations and will have direct access to the TRC PM to resolve any QA dispute. The QA Officer has sufficient authority to stop work on the investigation as deemed necessary in the event of serious QA issues. The QA Officer will perform QA audits on the sampling activities, provide QA technical assistance to the sampling team, and will be responsible to coordinate data verification. The QA Officer will review and approve QA plans and procedures.

- **TRC Field Samplers** – Field staff for this project are drawn from TRC's pool of qualified resources. The PM will utilize staff to gather and analyze data and to provide supporting materials. The designated technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

- **WDNR PM** – William "Bill" Fitzpatrick, WDNR – Division of Water, Watershed Management, Madison, Wisconsin. The WDNR PM is the lead person for WDNR and is responsible for technical direction, review and approval of all aspects of work performed. The WDNR PM is the lead person for communication with the TRC team and is also responsible for coordinating communications with USEPA (if required), other state agencies, and other WDNR staff, as appropriate.

1.2.2 Laboratory Responsibilities

The laboratory assigned with responsibility for chemical analysis of environmental samples is Pace Analytical Services, LLC (PAS) located in Green Bay, Wisconsin. PAS is certified in the State of Wisconsin. The PAS certification, Quality Manual (QM), and applicable standard operating procedures (SOPs) are provided in Appendix A.

- **Pace Analytical Services, LLC Analytical Project Manager (APM)** – Laurie Woelfel, PAS, Green Bay, Wisconsin, will designate a chemist to be responsible for management of the analytical requirements for sample analysis and will report directly to the APM. The APM will provide a complete interface with TRC from initial project specifications to final deliverables.
- **Pace Analytical Services, LLC Analytical QA Officer** – Kate Verbeten, PAS, Green Bay, Wisconsin, has the overall responsibility for data before it leaves the laboratory. The Analytical QA Officer will communicate data issues through the APM, oversee laboratory QA and QC documentation, conduct a detailed data review, implement laboratory corrective actions, and, if required, define laboratory QA and QC procedures and prepare SOPs.

1.3 Project Target Schedule

The investigation downstream of the Dam will be initiated during the spring/early summer of 2021, after WDNR's approval of the SIWP. Future phases of investigation will be completed on an as-needed basis.

1.4 Project Background and Description

Historically, PCBs from a former manufacturing facility (from a transformer release) were potentially discharged into a storm water outfall that discharges into a portion of the watershed of Jordan Creek that includes drainage ditches in farm fields and which eventually flows into Pine Creek. The ditches drain east to Jordan Creek, which flows generally northeast approximately one mile to a confluence with Pine Creek. Pine Creek flows north and northwest through agricultural areas approximately six miles to the Hayton Millpond, where it joins the South Branch Manitowoc River. Pine Creek enters Hayton Millpond from the south, the South Branch Manitowoc River enters from the northwest, and the Dam is located on the northeast end of the Millpond, where the South Branch Manitowoc River continues to the east (Figure 1).

In August 2015, Tecumseh completed a reconnaissance study downstream of the Dam at the request of the WDNR in its letter dated January 15, 2015 (WDNR, 2015). The methods and means used in this reconnaissance study were established in a WDNR approved SAP (WDNR, 2013). The reconnaissance study area extended from the Dam to approximately 1.5 miles downstream of the Dam in the South Branch Manitowoc River. A surface-area weighted average concentration (SWAC) of 0.53 mg/kg was calculated for the study area. The distribution and range of results indicated low levels of PCBs below the Dam. The SWAC confirmed the effectiveness of the upstream HARP remediation program, that there is no on-going source of contamination, and that there is little PCB-associated risk downstream of the Dam.

Pursuant to the Negotiated Agreement, WDNR required further characterization of the sediment and overbank soil downstream of the Dam, extending from the Dam to approximately 2 miles downstream of the Dam in the South Branch Manitowoc River. Figure 2 shows the extent of the investigation area.

1.5 Quality Objectives and Criteria for Measurement Data

DQOs are qualitative and quantitative statements that define the project objectives, define the appropriate type of data, and specify acceptable probabilities of making decision errors that will be used as the basis for establishing the quality and quantity of data needed for decision making. The DQOs have been developed in general accordance with USEPA *Guidance for Data Quality Objectives Process* (QA/G-4) (USEPA, February 2006). The seven-step DQO process, as applied to the investigation downstream of the Dam, as applicable, is presented below.

1.5.1 Step 1: State the Problem

Following a 2015 reconnaissance study, the WDNR has requested further characterization of sediment conditions and overbank soil downstream of the Dam.

1.5.2 Step 2: Identify the Goals

The objectives of the investigation are as follows:

- To characterize the nature and extent of PCBs in sediment approximately two miles below the Dam.
- To evaluate the general overbank soil conditions and potential source areas below the Dam.
- To expand the reconnaissance level study completed by Tecumseh in August 2015, by integrating the data from this additional study.
- To assess the depth of water and sediment and the depositional environment in the South Branch Manitowoc River downstream from the Dam.

1.5.3 Step 3: Identify Information Inputs

The August 2015 reconnaissance study results provides sediment characterization data to approximately 1.5 miles downstream of the Dam.

1.5.4 Step 4: Define the Boundaries of the Project Area

The upstream and downstream boundaries of the project area for the sediment and overbank soil investigation are well defined, as previously described in Section 1.4.

The focus of the sediment investigation is the surface and near surface depositional areas within the channel (soft sediment) and the overbank soil. Soft sediment is defined as the unconsolidated inorganic/organic material that has settled out of surface water into in-channel beds in direct contact with the aquatic environment.

Soil is defined as the unconsolidated mineral or organic material in the overbank that has been subjected to and shows the effect of soil forming factors. The overbank soil sampling area is generally within 5 - 10 feet from the top of bank and will include floodplain sample locations depending on the geomorphic surface.

1.5.5 Step 5: Develop the Analytical Approach

Surficial sediment samples and overbank soil samples, as well as any water samples (either investigatory or QC samples) collected during the investigation will be analyzed for PCBs by SW-846 Method 8082, in accordance with the associated SIWP. The soil and sediment data are used to characterize the downstream sediment, calculate the SWAC, and determine the potential need for a remedial action, as appropriate. For solid matrix samples (e.g., soil and sediment), percent solids will be analyzed by ASTM D2974-87 to allow for PCB concentrations to be reported on a dry weight basis to maintain consistency.

1.5.6 Step 6: Specify Performance or Acceptance Criteria

Potential decision errors may be associated with field sample variability and sample collection procedures that do not represent the site's characteristics, or laboratory analytical procedures that do not meet the laboratory's required QA/QC process.

The Field Samplers will follow consistent methods to minimize potential errors in sample collection, handling, preparation and decontamination. Analytical errors are limited through the use of appropriate and consistent reporting units, standard operating procedures and regular system calibrations as described in the laboratory QM (Appendix A). In addition, laboratory control samples (LCS) will be analyzed to assess the precision, accuracy, representativeness, and comparability of the analytical results. Laboratory DQOs, expressed in terms of precision, accuracy, and completeness are provided in Table 1, with further detail provided in the laboratory QM (Appendix A).

1.5.7 Step 7: Develop the Plan for Obtaining Data

Historical data from the 2015 reconnaissance study were used to define the project area and select sample locations. Sample locations and frequencies are outlined in the associated SIWP.

In general, sediment samples will be collected at locations targeting the thickest soft sediment deposit at a given location along the river. The core sampler will be advanced through the full thickness of sediment, to refusal, or to a maximum depth of 3 feet below the sediment-surface water interface. If 12 inches or more of soft sediment are retrieved, the top 12-inches of sediment will be composited and submitted to the laboratory for analysis. If less than 12 inches of sediment are recovered, the full thickness of soft sediment will be composited for analysis.

Overbank soil samples will be collected to evaluate the general overbank soil conditions and potential source areas. Overbank soil samples will target the 0- to 6-inch interval below ground surface (bgs) initially. Deeper overbank samples, if deemed necessary based on the shallow sample results, will be collected on 12-inch intervals beginning at a depth of 6-inches bgs (i.e., 6- to 18-inches, followed by 18- to 30-inches, and so on).

1.6 Special Certifications

PAS, a laboratory certified in the State of Wisconsin, shall analyze the samples (sediment, soil, water) in conjunction with this project. Field personnel and contractors working on this project are required to have taken the 40 hour Occupational Safety and Health Administration (OSHA) training specified in 29 Code of Federal Regulations (CFR) 1910.120 and have a corresponding current 8 hour OSHA refresher certification, as applicable.

1.7 Documents and Records

The project team will maintain the project records. These records will contain the final versions of scopes of work, results of samples, copies of written correspondences, and copies of reports. Project records include field documents and laboratory documents. Electronic and hard copies of these files will be maintained for five years beyond completion of the project.

1.7.1 Field Documents and Records

Field documents and records consist of sample logs, chain-of-custody forms, field notes, site photographs, and other appropriate documentation. The field documents and records will be maintained at the TRC office in Chicago, Illinois, during the active portion of the project and for five years beyond completion of the downstream of the Dam project phase.

1.7.2 Laboratory Documents and Records

Laboratory documents and records will be generated and maintained consistent with the PAS QM (Appendix A). At a minimum, PAS will maintain records necessary to comply with WDNR laboratory certification. The information includes records on sample preparation and extraction, instrument signals, and instrument conditions, calibration, calculations and quality control. The laboratory retains records to comply with WDNR certification, and TRC will maintain records for five years beyond completion of the downstream of the Dam project phase.

2.0 Data Generation and Acquisition

2.1 Sample Design Process

A variety of samples are anticipated for the investigation. The individual SIWPs document the samples required and sampling procedures.

2.1.1 Sample Media

Sample media will include soil samples from overbank locations and surficial sediment samples from in-channel sampling locations. Water samples may be necessary for additional investigation and/or for QA/QC analysis (if specified in the associated Work Plan).

Samples will be submitted to the PAS laboratory for analysis of Aroclor congeners and total PCBs (Table 1 and Appendix A).

2.1.2 Sample Locations and Field Positioning

Sample locations will be field located using a hand held, high resolution global positioning system (GPS) device capable of sub-meter accuracy.

2.1.3 Sample Identification and Labels

Each sample will be labeled with unique sample identifications (IDs) that will facilitate tracking and cross-referencing of sample information. The sample identification scheme to be used is described in the SIWP. Example sample identification schemes are described below.

In-Channel Sediment Samples

In-channel samples will have the additional prefix “IC” following the location ID.

- For in-channel samples on the left side of the river:
  - MR IC [#010-499]L
    Example: MR IC 012L
- For in-channel samples on the right side of the river:
  - MR IC [#510-899]R
    Example: MR IC 512R
- For in-channel samples (center of the river):
  - MR IC [#910-999]C
    Example: MR IC 912C

Overbank Soil Samples

- Overbank soil characterization samples on the left side of the river will be named as follows:
  - MR [#010-499]L [interval]
    Example: MR 012L 0-6”
- And on the right side of the river:
  - MR [#510-899]R [interval]
    Example: MR 512R 0-6”

The 3-digit sample number for all in-channel and overbank soil sample locations will be unique for a given sample type (e.g., overbank soil on the left side of the river) and will be labeled in sequence from upstream to downstream (e.g., beginning with “010” for soil on the left bank). The ground surface will be used as the 0” reference for the sample interval.

Water Samples

Investigative water samples are not currently included as an investigation SOW. If collected, investigative water samples will be given a unique sample identifier as defined in the associated SIWP.

2.1.4 Decontamination

Non-dedicated equipment used for sample collection or sample processing will be new or cleaned before its initial use in the field, and will be cleaned again before use at each subsequent sampling site (and between sample intervals). Equipment subject to this decontamination procedure includes, but is not limited to, the following:

- Coring tools (e.g., pistons or core barrels)
- Shovels
- Augers
- Scoops, spatulas, and mixing bowls (if re-used)

The general procedure for decontaminating field equipment is as follows:

- Scrape off as much loose material as possible.
- Disassemble the equipment, as appropriate.
- Wash with detergent/potable water solution.
- Rinse thoroughly with deionized (DI) or distilled water.
- Allow equipment to air dry prior to next use.

- Wrap equipment for transport with inert material (aluminum foil or plastic wrap) to prevent direct contact with potentially contaminated material.

Field decontamination of sampling equipment will take place at a designated location on-site or within the sample processing area. Decontamination will be performed in 5-gallon buckets, and will be managed as IDW as specified in the SOW. Decontamination water will be changed out for new, clean solutions at a minimum of once per sampling day.

2.1.5 Sample Container, Preservation, and Holding Time Requirements

Sample preservatives and containers shall be prepared and used as necessary to comply with laboratory method requirements for analytes of interest (see Table 2).

2.2 Sampling Methods

This section describes the anticipated sampling procedures as well as the equipment, supplies, sample containers, preservation and holding time requirements, sample volumes, and decontamination procedures.

2.2.1 General Sampling Guidelines

The following protocols will be employed during sample collection activities downstream of the Dam:

- Prior to arriving at the site, field sampling equipment will be examined to verify that it is in good operating condition. After each use, non-dedicated sampling equipment will be washed (i.e., decontaminated) with a laboratory grade soap and rinsed in clean, distilled or deionized water.
- Sample preservatives and containers shall be prepared and used as necessary to comply with laboratory method requirements for analytes of interest (see Table 2).
- Field sampling crew members will use a new pair of disposable latex or nitrile gloves for each sample location, for each sample collected or processed, and will change them as appropriate when torn or soiled.
- Field QC samples will be collected and submitted to the laboratory at a frequency specified in the associated SIWP, and in accordance with requirements outlined in Wisconsin Administrative Code (WAC) Chapter NR 716.13(6).
- The sampling generated wastes (i.e., sediment, soil, etc.) will be collected, containerized, and labeled for proper transport and disposal.
- Sample labels will be completed at the time of sample collection, noting the site identification, sample location, sample interval (as appropriate), preservative (as appropriate), sample analysis, and sample collection date.
- The sampling procedures will be recorded in the field notes.

- If the listed sampling or processing procedures cannot be performed or require modification, then a Corrective Action Form will be initiated and steps will be taken to rectify the nonconformance issues as appropriate. The Corrective Action Form is included in Appendix B. The appropriate project personnel will be notified of any material change that warrants the completion of a Corrective Action Form.

2.2.2 Sample Collection

In-Channel Sediment Sample Collection

Sediment core samples will be collected using clear plastic (PVC, lexan, polycarbonate, or equivalent) core tubes. The core tubes will ordinarily be 2-inches in diameter, but may range in diameter from 1.5-inches to 3-inches in diameter depending on the sampling conditions (e.g., sediment type and water depth). The core tube may be used in conjunction with a piston coring assembly, a steel core sampler, or simply be deployed by hand, depending on the field conditions.

At each sampling location, a water depth measurement will be recorded prior to collecting the core sample. This may be achieved by using a core tube or rod pre-marked in 0.1-foot increments, or a weighted tape measure, or similar device. The water depth measuring device will be lowered through the water column until in contact with the sediment surface, and the water depth, estimated to the nearest 0.1 foot, will be recorded.

Once the water depth is recorded, the core tube will then be pushed by hand through the entire thickness of soft sediment and into the underlying soil until refusal is encountered, or to a maximum of 3 feet below the sediment/surface water interface. The penetration depth will be recorded. The sample core will be extracted from the sediment, capped, labeled, maintained in a vertical orientation, and transported to shore for processing. If soft sediment is not present; or the core recovery at the time of retrieval is less than 12 inches and does not appear representative of sediment conditions, up to three attempts may be made to collect a representative core sample at the sample location.

Physical data collected at each location will include the following:

- The water depth;
- The distance that the core is pushed into the sediments;
- The thickness of soft sediment;
- The conditions of refusal (physical impediment or resistance);
- The visual description of the deposit; and
- The recovery length.

Overbank Soil Sample Collection

Overbank soil samples will be collected using either a spade, a hand auger, a push tube sampler, or equivalent. The sample contacting portion of the sampling equipment will be stainless steel, or if collected with a coring tool, a clear plastic (e.g., PVC, polycarbonate, or equivalent) sleeve may be used. Initial soil samples will be collected from a depth of 0 to 6 inches below grade. If deeper samples are required by the SOW, they will be collected at 12-inch intervals beginning at a depth of 6-inches below grade. Overbank soil samples will be collected directly into a vessel for homogenization or for transport to the sample processing location.

2.2.3 Sample Processing

Once retrieved from the sample location, the sediment, soil, and water samples will be processed, containerized, and transported to the analytical laboratory in accordance with the applicable SIWP, disposal facility, or WPDES permit requirements. Sediment/soil sample containers, preservation requirements, and holding time requirements applicable to this project are summarized on Table 2.

2.3 Sample Handling, Chain-of-Custody, and Shipping

Field personnel are responsible for the care and custody of samples until they are transferred to the onsite laboratory or shipped, as necessary. As few people as possible should handle the samples. At the time of collection, sample labels and chain-of-custody forms will be completed by the field sampling team using waterproof ink. The label will include the project number, unique sample identification number, collection date, and sample type. A representative chain-of-custody form is given in Appendix B.

Samples will be accompanied by a properly completed chain-of-custody (COC) form during each step of custody transfer and shipment. When physical possession of samples is transferred, both the individual relinquishing the samples and the individual receiving them will sign, date, and record the time of transfer on the COC form.

Samples will be shipped directly to the laboratories by a TRC employee, an overnight commercial courier, or a laboratory-supplied courier service. In the case of sample shipment by an overnight commercial courier, a package tracking number will serve as an extension of the COC form while the samples are in transit. The COC forms will be sealed inside the sample cooler within a clear plastic bag taped to the inner top of the cooler and the custody seals, if used, will be completed on the outside of the cooler prior to shipment. Commercial couriers are not required to sign off on the custody forms since the forms are sealed inside the cooler prior to shipment so any custody seal remains intact.

The original COC form will accompany the samples at all times. A copy of all COC forms submitted to the laboratory will be retained by the sampler along with field records/logbooks documenting sample collection and will be placed in the project files.

If at the completion of sampling, the samples are not shipped directly from the field or point of collection to the analytical laboratory, the samples will be temporarily stored in an iced cooler at a secure location (e.g., locked vehicle, residence, office). Access to the secure location and

transfer of the sample containers for laboratory delivery shall only be provided by a TRC employee and such sample transfer shall be recorded on the COC form.

2.4 Analytical Methods and Procedures

The sediment, soil, and water samples collected for chemical analyses during this investigation will be analyzed using the methods provided in Table 1. Target reporting limits for the matrices of interest are also provided in Table 1. The following sections describe the analytical methods that will be used. Corresponding SOPs for these analyses are included in Appendix A.

2.4.1 Total PCB in Sediment, Soil, and Water

Sediment, soil, and water samples collected for laboratory analysis by PAS during the investigation, will be analyzed for PCBs using USEPA Method SW-846 Method 8082.

2.4.2 Percent Solids in Sediment/Soil

Percent solids of sediment/soil samples will be determined gravimetrically at the PAS laboratory by drying aliquots of the samples that are collected in the field. The PAS SOP for this procedure, which is taken from ASTM D2974-87 will be followed. Results will be reported as percent dry weight. Appendix A contains the PAS SOP for percent solids or total solids. These results will be used to calculate the PCB results back to a dry weight basis.

2.5 Quality Control Requirements

Specific procedures related to field quality assurance samples, and laboratory quality assurance, namely calibrations, matrix spikes, surrogate spikes, method blanks, laboratory control samples, laboratory duplicate and matrix spike duplicates are described in the following sections. The individual laboratory SOPs provided in Appendix A, also describe specific QC elements and acceptance criteria that apply to each parameter.

2.5.1 Field Quality Assurance/Quality Control (QA/QC) Samples

Four types of QA/QC samples may be collected during the course of the investigation:

- Field duplicates;
- Field equipment blanks;
- Matrix spike/matrix spike duplicates; and
- Temperature blanks.

QA/QC samples will be collected as required by WAC Chapter NR 716.13(6) and as specified in the associated SIWP. Each of the QA/QC sample types and collection frequencies (if required for the SOW) are described below.

Field Duplicates

Blind field duplicate samples, prepared by splitting a single homogenous sample into two separate sets of containers, will be used to evaluate sampling precision. Points where duplicate samples are to be collected will be selected by the field personnel and will be submitted as blind duplicates to the laboratory (i.e., field duplicates are separate study samples that are submitted to the analytical laboratory without distinguishing identification). Duplicate samples will be numbered sequentially beginning with "DUP-001." The duplicate sample ID number and the associated primary sample ID will be recorded in the field logbook and/or field data sheet. Blind duplicate samples will be collected only for water matrix samples (not for solid matrices) at a frequency of one for every 10 (or fewer) primary samples collected. Duplicate samples are not required for soil samples under WAC Chapter 716.13(6)(b).

Field Equipment Blank (Rinsate Blank)

Field equipment blanks (or "rinsate blank") consisting of analyte-free water may be collected and submitted to the analytical laboratory to assess the quality of the data resulting from the field sampling program. The water source for the blanks will consist of deionized or distilled water from an off-site source. Field equipment blanks are analyzed to check for procedural contamination at the site that may cause sample contamination. Field equipment blanks are samples collected in the field by rinsing apiece of non-dedicated sampling equipment (e.g., steel bowls, Geoprobe® samplers, or sampling pumps) that has just been decontaminated with analyte-free water or other blank matrix, and then transferring this water to the proper sample bottles. If included in the applicable SOW, field equipment blanks will be collected at a frequency of one for every 10 (or fewer) primary samples that are collected with the non-dedicated equipment. Equipment blanks will be numbered sequentially starting with FB-001 (FB-001, FB-002, FB-003, etc.), and may have the specific piece of equipment appended to the sample ID (e.g., "FB-001-bowl"). Equipment blanks will not be collected from dedicated or disposable field equipment, and they are not required under WAC Chapter NR 716.13(6)(b) for soil samples.

Matrix Spike/Matrix Spike Duplicate

Additional sample volume for matrix spike and matrix spike duplicate (MS/MSD) samples will not be collected or specified by the field sampling team unless requested by the laboratory. The laboratory will select one sample per analytical batch (20 analytical samples) to perform a MS/MSD as required by the analytical methods. A matrix spike and matrix spike duplicate (MS/MSD) are representative but randomly chosen client samples that have known concentrations of analytes of interest added to the samples prior to sample preparation and analysis. They are processed along with the primary (un-spiked) sample collected from the same location. The purpose of the MS/MSD is to document the accuracy and precision of the method for that specific sample. Control charts are maintained that are indicative of typical MS/MSD recoveries of 'real' samples rather than laboratory-controlled samples. The MS/MSD data serves as an indication of the problems that may be associated with a specific sample or sample matrix, as some materials can interfere with certain analytes. MS/MSD samples typically consist of a triple volume of an existing sample. MS/MSD samples will be collected at a frequency requested by the laboratory and as sample volume permits. MS/MSD samples will be identified by placing "MS/MSD" as the suffix of a sample (e.g. "MW-12 MS/MSD").

Temperature Blanks

The condition of each cooler will be evaluated upon receipt at the laboratory. Samples received on ice are considered preserved at the correct temperature (4°C, ± 2°). Temperature blanks may also be analyzed to assess whether the sample temperature was maintained during sample transport, especially in the case that the ice has all melted. Temperature blanks consist of a sample container, generally polyethylene, filled with tap water. One temperature blank will be transported with each cooler containing sample containers.

2.5.2 Initial and Continuing Calibration

The compliance requirements for satisfactory instrument calibration ensure that the instrument is capable of producing acceptable quantitative data. They consist of an initial calibration to demonstrate that the instrument is performing acceptably throughout the analytical working range before project samples are analyzed, and continuing calibration checks that document that the initial calibration is still valid and that satisfactory maintenance and day-to-day adjustment of the instrument have been achieved. Initial and continuing calibration checks apply to the PCB analyses of sediment/soil and water samples conducted during the investigation.

Specific control criteria and corrective action requirements for initial and continuing calibration checks are given in the laboratory QM and respective method SOPs given in Appendix A.

2.5.3 Method Blanks

A method blank will be analyzed by the laboratory at a frequency of one blank per 20 environmental samples or, in the event that an analytical batch consists of less than 20 samples, one method blank will be analyzed per sampling event. Laboratory distilled and analyte-free water or clean sand will be used for the method blank. Method blanks will be carried through the entire analytical procedure and apply to the PCB in sediment/soil and water samples and percent solids analyses conducted during the investigation.

The specific acceptance criteria for method blanks for each procedure can be found in the respective SOP given in Appendix A.

2.5.4 Matrix Spike/Matrix Spike Duplicate and Laboratory Control Samples

MS/MSD and LCS samples for PCBs will be analyzed at a minimum frequency of one per 20 environmental samples. A LD sample may be substituted for a MS/MSD pair. At least one Aroclor will be used for the total PCB (8082) matrix spikes. Acceptance criteria are described in the PAS SOP provided in Appendix A. Percent spike recoveries will be used to evaluate analytical accuracy while relative percent difference between duplicates will be used to assess precision.

2.5.5 Surrogate Standards

Surrogate spikes are used in PCB analyses. Every blank, QC, and environmental sample analyzed for PCB by GC/ECD will be spiked with decachlorobiphenyl (DCBP) and 2,4,5,6-tetrachloro-m-xylene (TCMX), the surrogates specified in the SOP, prior to extraction. If a sample concentration is high and requires substantial (greater than 10x) dilution in order to quantify, its surrogate recovery will not be calculated and the resulting data will be flagged for "Non Reportable Surrogate Recovery" due to sample dilution.

2.5.6 Corrective Action

Any team member may identify a need for corrective action. The corrective action process consists of identifying a problem, acting to eliminate the problem, monitoring the effectives of the corrective action, verifying that the problem has been eliminated, and documenting the corrective action. A Corrective Action form for field activities is included in Appendix B. The laboratory corrective action process is described in the laboratory QM (Appendix A).

2.6 Instrument/Equipment Testing, Inspection and Maintenance

This section describes the procedures for maintaining the accuracy of the instruments and measuring equipment that is used for field measurements and laboratory analyses. These devices are calibrated, or the calibrations are verified, prior to each use or according to a prescribed schedule.

2.6.1 Field Instruments/Equipment

Equipment used during field sampling, including scoops, bowls, sampling probes, and augers, will be examined prior to use to make sure it is clean and in good operating condition.

A hand held GPS unit will be used to locate sample locations. The GPS unit will be handled according to the manufacturer's recommendations and recalibrated on a regular maintenance interval or as determined necessary by the Field Samplers to provide accurate and reproducible results. Damaged equipment or inaccurate measurements will be recorded on field notes at the time they are noticed and replacement equipment will be acquired for subsequent field activities.

2.6.2 Laboratory Instruments/Equipment

Calibration of laboratory equipment will be performed according to the procedures specified in the laboratory QM and respective SOPs provided in Appendix A. Written documentation of calibration repairs or replacement of instruments will be maintained by the laboratory personnel. These records will be kept at the laboratory and will be available for audit. The laboratory will maintain service contracts with instrument vendors, or train laboratory staff to perform repairs with the appropriate equipment.

2.7 Data Reduction, Validation and Reporting

2.7.1 Data Reduction

Data reduction includes all activities that convert instrument responses into reportable results and may involve mathematical calculations, compound identification, and summary statistics. For all investigation parameters, calculations are performed by computer programs. The initial data reduction is the responsibility of the analyst or field person operating the analytical instrument. General data reduction duties at this level are as follows:

- Calculate spike recoveries and precision for duplicates.
- Identify QC data (method blanks, spikes, duplicates, LCS) for review by senior level staff.
- Check transcription of sample identification numbers on raw data records.
- Calculate final concentrations for environmental samples, accounting for dilution factors extraction volumes or masses, and percent solids.

At PAS, after the primary analyst has performed the data review checklists, the results are submitted to a peer reviewer who reviews 100% of the data entered including 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 peer review includes analyst-generated calculations. The APM 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.

PAS raw data, certificates pertaining to calibration and maintenance of equipment, raw data from instrumentation, quality control documents and logbooks are stored, in either paper files or electronically in a secure location, under the unique PAS project number that has been assigned. Both types of records are retained at PAS for five years after the analysis is completed, unless a longer period is mandated by regulatory requirements.

2.7.2 Data Validation/Usability

Data validation/usability of any or all analytical parameters may be undertaken by one or more of the entities participating in the investigation program, at their sole discretion. Validation/usability of analytical data includes checks for data consistency by looking for comparability of duplicate analyses, potential sample contamination as indicated by the results of field blank or laboratory blank sample analyses, adherence to accuracy and precision acceptance criteria, transmittal errors, and anomalously high or low concentrations. The following is a description of the specific procedures to be used to assess the level of precision, accuracy, and completeness during the routine sample analyses.

Precision

Precision is a measurement of reproducibility of repetitive measurements and will be assessed by comparing the analytical results of MS/MSD pairs and LD samples for PCB analyses. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses using the formula below:

$$RPD = (R1 - R2) / ((R1 + R2) /2) \times 100$$

Where:

R1 = value of the first result
R2 = value of the second result

Accuracy

Accuracy is a measurement of correctness using spike recovery calculations, including random and systemic errors, and will be assessed by calculating the percent recovery of MS/MSD and LCS samples and comparing it to the method acceptance criteria. Percent recovery (%R) will be calculated using the following equation:

$$\%R = ((SSR - SR) / SA) \times 100$$

Where:

SSR = spiked sample result
SR = sample result or background concentration
SA = spike added concentration

Completeness

Completeness will be assessed by comparing the number of valid or useable results following data validation, to the total number of results generated. The project goal is 90%. The following equation will be used to determine completeness:

$$\%C = (V/T) \times 100$$

Where:

V = number of valid sample results
T = total number of sample results

2.7.3 Data Deliverables and Reporting

Final laboratory data reports for investigation activities will consist of the following deliverables:

- A summary of the analytical methods used and any analytical problems and unusual conditions;
- Dates of sample receipt, extraction/preparation and analysis;
- Method blank summaries;
- Duplicate sample data, surrogate compound recovery data, MS/MSD recovery data, LCS recovery data and all associated control limits;
- Executed chain-of-custody records.

Electronically formatted data deliverables will be utilized to expedite data review and submittal.

3.0 Assessment and Oversight

3.1 Assessment and Response Actions

Assessment and oversight activities may be performed to determine whether the QC measures outlined in this QAPP are implemented and documented. The QA Officer may perform an assessment and oversight to check conformance to the plan.

3.2 Field Audits

Internal field audits may be performed to verify compliance with the QAPP. Audits may include examination of the work area, sampling practices, sample handling, labeling, storage, shipping, and field documentation. If performed, the auditor will provide a summary letter to document the visit and any QC issues not in compliance. If an item is identified, an additional internal audit may be performed at the discretion of the QA Officer.

3.3 Laboratory Audits

The laboratory may conduct internal system audits at the discretion of the Analytical QA Officer as described in the QM (Appendix A). If performed, the Analytical QA Officer prepares and issues an internal audit report identifying the observations and deficiencies that require corrective action, if any. The responsible manager will respond to the internal laboratory audit, the Analytical QA Officer will review the response and if acceptable determine the response is adequate. The Analytical QA Officer will re-examine the area, as appropriate.

In addition, external audits are performed on a regular basis by regulatory agencies to maintain laboratory certifications and by clients to maintain appropriate specific protocols.

4.0 References

40 CFR 136, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. July 1989.

American Society of Testing and Materials. 1993. D 2974-87, Standard Test Method for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils. March/April 1993.

TRC. 2017. Quality Assurance Project Plan for Remedial Investigation. Hayton Area Remediation Project, Operable Units 3/Lower and 4, Chilton, Calumet County Wisconsin. Revision 3. October 2017.

TRC. 2021. Site Investigation Work Plan, Additional Investigation Sampling Plan Downstream of Hayton Millpond Dam. January 2021.

U.S. Environmental Protection Agency. 1993. Manual of Chemical Methods for Pesticides and Devices, 2nd ed. 1993.

U.S. Environmental Protection Agency. 1995. Standard Methods for the Examination of Water and Wastewater, 19th ed. 1995.

U.S. Environmental Protection Agency. 1999. Methods for Chemical Analysis of Water and Wastes, 600/4-79-020. 1983 (as updated 1999).

U.S. Environmental Protection Agency. 2001. EPA Requirements for Quality Assurance Project Plans (Final), EPA QA/R-5. March 2001.

U.S. Environmental Protection Agency. 2002. Guidance for Quality Assurance Project Plans (Final), EPA QA/G-5. December 2002.

U.S. Environmental Protection Agency. 2006. Guidance for the Data Quality Objectives Process, EPA QA/G-4. February 2006.

U.S. Environmental Protection Agency. 2007. Test Methods for Evaluating Solid Waste, SW-846, Revision 1. 2007.

U.S. Environmental Protection Agency New England. 2013. Environmental Data Review Supplement For Regional Data Review Elements and Superfund Specific Guidance/Procedures. April 2013.

WDNR, Tecumseh Products, and TRC. 2004. Consent Order No. 2004-COEE-010; Facility ID No. WID006116529. 2004.

WDNR. 2013. Letter from WDNR to Tecumseh, Conditional Approval of Hayton Area Remediation Project Reconnaissance Sampling Plan Downstream of Hayton Millpond Dam. July 1, 2013.

WDNR. 2015. Letter from WDNR to Tecumseh, PCB Sampling Downstream of Hayton Mill Pond Dam, Hayton Area Remediation Project. January 15, 2015.

WDNR, Tecumseh Products, and TRC. 2018. Negotiated Agreement; BRRTS #02-08-281506.

Table 1: Project Goals for Precision, Accuracy, and Completeness for Laboratory Measurements

Parameter	Matrix	Method[1]	Reportable Units	Laboratory Method Detection Limit	Laboratory Reporting Limit	Precision Goal (%RPD)	Accuracy Goal (%R)	Completeness Goal (%)
Percent Solids	Sediment/ Soil	ASTM D2794-87	% dry weight	0.1 weight/weight	0.1 weight/weight	± 10	NA	90
PCB Congeners and Total PCB[3]	Sediment/ Soil	SW-846 8082	mg/kg or µg/kg dry weight	15.22 µg/kg	50.0 µg/kg	≤ 50%[2]	± 30	90
PCB Congeners and Total PCB[3]	Water	SW-846 8082	µg/L	0.112 µg/L	0.5 µg/L	≤ 30%[2]	± 30	90

Footnotes:

[1] References: USEPA 40 CFR Part 136 and SW-846, Third Edition, both as amended.

[2] References: *Environmental Data Review Supplement For Regional Data Review Elements and Superfund Specific Guidance/Procedures,* U.S. EPA New England, April 2013.

[3] PCB congeners are included in Appendix A

Notes:

%RPD = relative percent difference

PCB = Polychlorinated biphenyls

%R = percent recovery

Table 2: Sample Container, Preservation, and Holding Time Requirements

Parameter	Matrix	Method[1]	Sample Container and Size (minimum)	Preservation	Maximum Holding Time[2]
Percent Solids	Sediment/Soil	ASTM D2794-87	4 oz. glass or plastic jar	4° ± 2°C	7 days
PCB - Total	Sediment/Soil	USEPA 8082	2 oz. wide mouth glass jar	4° ± 2°C	14 days [3]
PCB - Total	Water	USEPA 8082	2 1-liter amber glass	4° ± 2°C	7 days [4]

Footnotes:

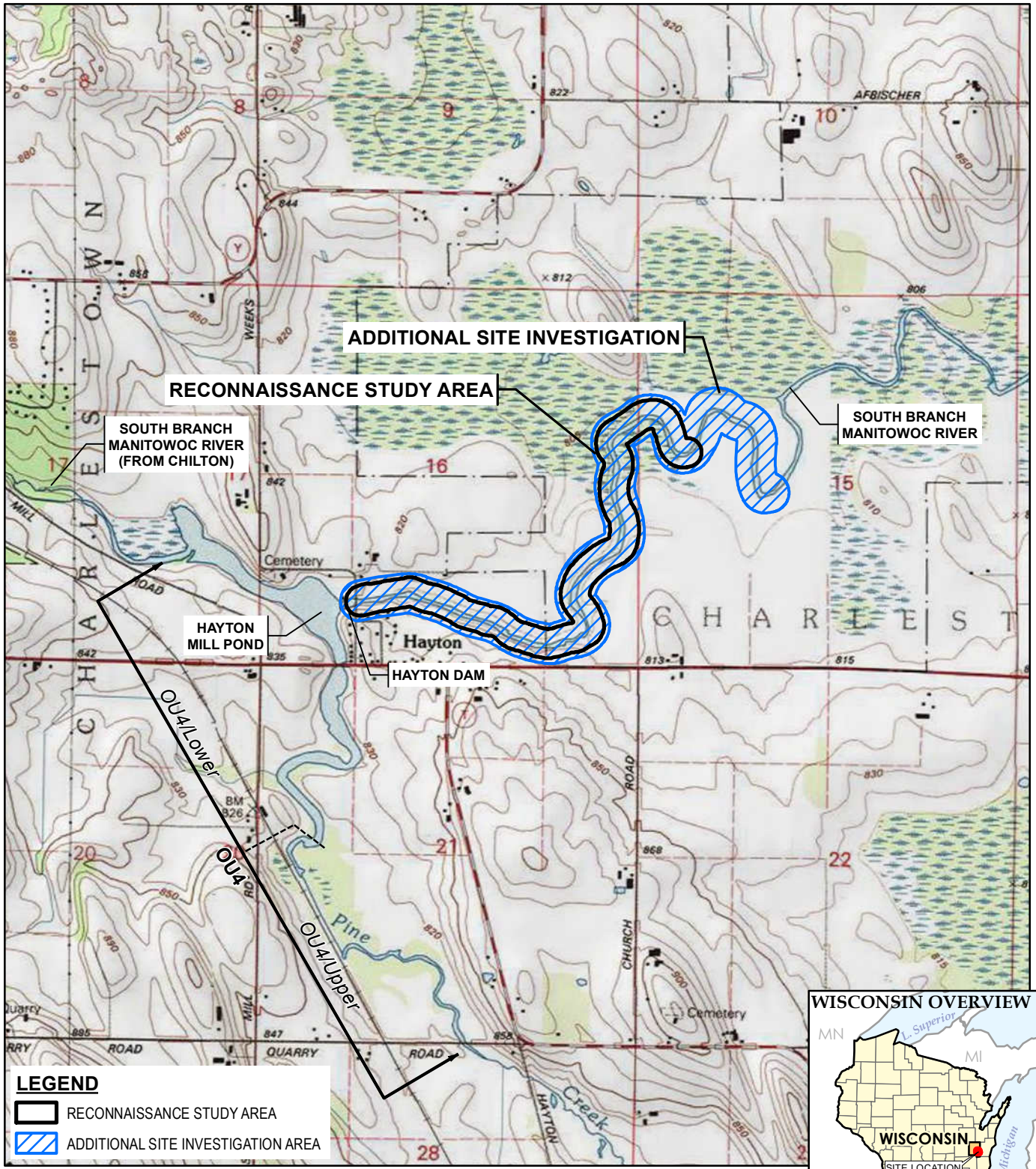
[1] References: USEPA 40 CFR Part 136 and SW-846, Third Edition, both as amended

[2] From date of sample collection.

[3] The laboratory recognizes the SW846 Chapter 4 hold time of none, and uses a 1 year hold time for extraction and extracts.

[4] The laboratory follows 40CFR Part 136 hold time of 1 year until extraction, 1 year after extraction.

TRC - GIS



BASE MAP FROM USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE SERIES.

N

1 " = 2,000 '
1:24,000

0 2,000 4,000
FEET

TRC
708 Heartland Trail
Suite 3000
Madison, WI 53717
Phone: 608.826.3600

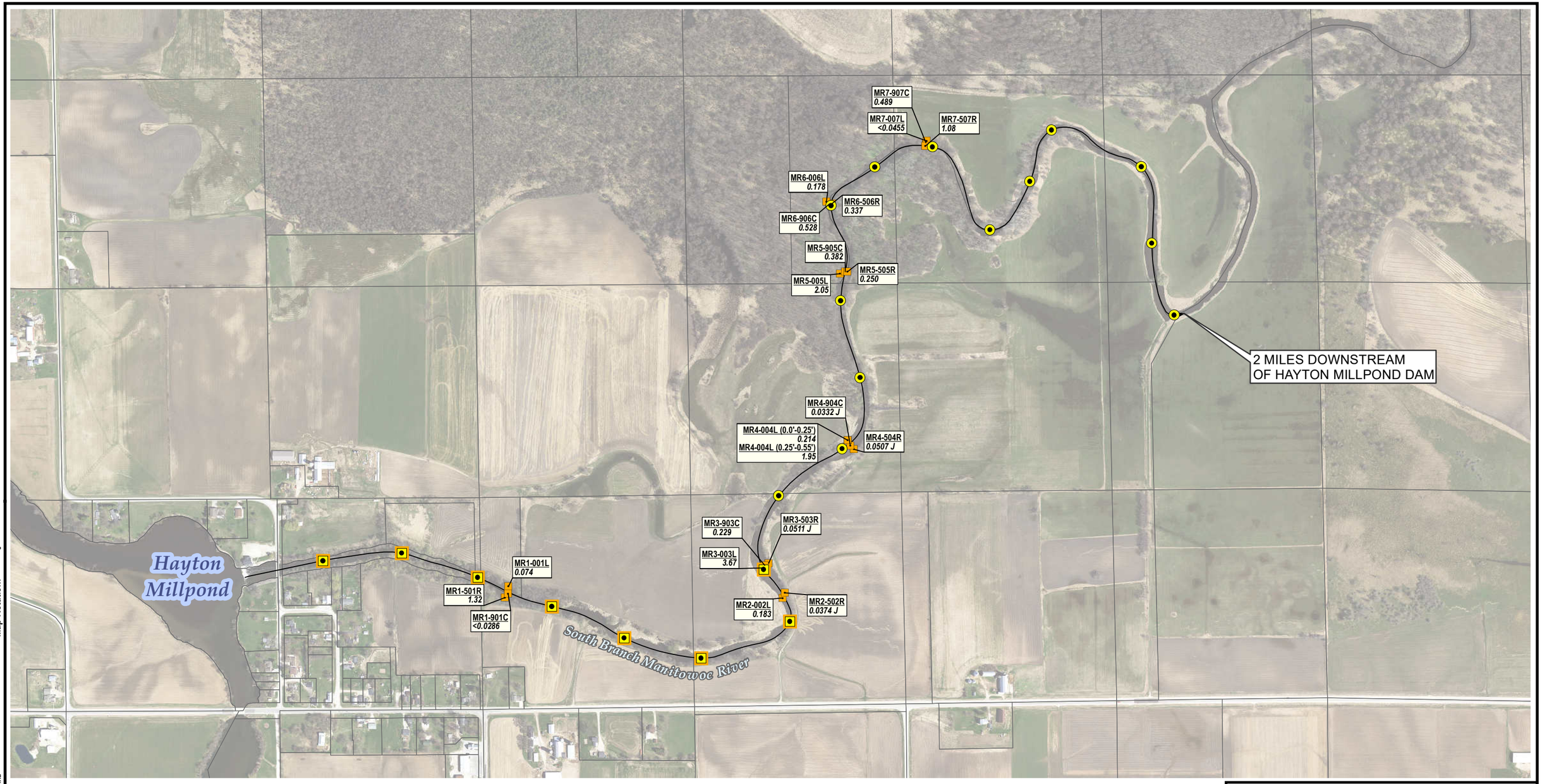
DOWNSTREAM HAYTON MILL POND DAM
ADDITIONAL SITE INVESTIGATION

SITE LOCATION MAP

DRAWN BY:	J. PAPEZ
APPROVED BY:	C. HARVEY
PROJECT NO:	320928
FILE NO.	320928-002.mxd
DATE:	JANUARY 2019

FIGURE 1

Coordinate System: NAD 1983 HARN WISCRS Calumet County Feet (Foot US)
Map Rotation: 0
Plot Date: 12/20/2018, 10:26:54 AM by JPAPEZ -- LAYOUT: ANSI B (11"x17")
Path: E:\Tecumseh\HARP\2018_320928\320928-001.mxd



LEGEND

- TRC SEDIMENT SAMPLING/TRANSECT LOCATION (AUGUST 2015)
- IN-CHANNEL TRANSECT LOCATION
- TRANSECT WITH OVERBANK SOIL SAMPLES

NOTES

1. BASE MAP IMAGERY FROM CALUMET COUNTY 2014.

N

0 600 1,200 Feet

1 " = 600 '
1:7,200

PROJECT:
DOWNSTREAM HAYTON MILL POND DAM ADDITIONAL SITE INVESTIGATION

TITLE:
DOWNSTREAM SAMPLING

DRAWN BY:	J. PAPEZ	PROJ NO.:	320928
CHECKED BY:	J. RICE		
APPROVED BY:	C. HARVEY		**FIGURE 2**
DATE:	DECEMBER 2018		

TRC
708 Heartland Trail, Suite 3000
Madison, WI 53717
Phone: 608.826.3600
www.trcsolutions.com

FILE NO.: 320928-001.mxd

Appendix A: PAS Laboratory Documentation

Pace Analytical®

ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-MAN-GBAY-0001 **Revision:** 01

Document Title: Quality Manual

Department(s): Quality

Date Information

Effective Date: 23 Jan 2020

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-MAN-GBAY-0001 **Revision:** 01

Title: Quality Manual

All dates and times are in Central Time Zone.

ENV-MAN-GBAY-0001-Rev.01 Quality Manual

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Kate Verbeten (007119)	Manager - Quality	15 Jan 2020, 02:39:18 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Nils Melberg (007142)	General Manager 2	16 Jan 2020, 10:19:55 AM	Approved
Brian Basten (007101)	Manager	20 Jan 2020, 02:21:34 PM	Approved
Christopher Haase (007121)	Manager	21 Jan 2020, 01:40:55 PM	Approved
Scott Turner (007177)	Manager	23 Jan 2020, 01:56:20 PM	Approved
Chad Rusch (007163)	Manager	23 Jan 2020, 04:10:46 PM	Approved

TITLE PAGE

LABORATORY QUALITY MANUAL

Prepared for:

Pace Analytical Services, LLC
1241 Bellevue Street, Suite 9
Green Bay, WI 54302
Phone: 920-469-2436
(AKA: Green Bay)

Pace Analytical Services, LLC
870 East Higgins Road, Suite 143
Schaumburg, IL 60173
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Pace Analytical Services, LLC
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Madison, WI 53719
Phone: 608-232-3300

Pace Analytical Services, LLC
37727 Professional Center Drive, Suite 115D
Livonia, MI 48154
Phone: 313-378-0219

Manual Approval Signatories

Approval of this manual by managerial personnel is recorded on the Signature Manifest located before the Title Page of this manual.

The individuals listed below represent the management team that was in place on the effective date of this version of the manual for the following location:

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

Each of the following individuals is a signatory for the manual for the location listed above. The application of their signature to the manual signifies their commitment to communicate, implement, and uphold the requirements, policies and procedures specified in this manual and their commitment to continuously improve the effectiveness of the quality management system based on customer feedback and internal assessment.

Name[1]	Title	Address[2]	Phone[2]
Karl Anderson	Senior General Manager	7726 Moller Rd Indianapolis, IN 46268	317-228-3119
Nils Melberg	General Manager		920-321-9437
Kate Verbeten	Quality Manager		920-321-9426
Brian Basten	Client Services Manager		920-321-9411
Kate Verbeten	Health & Safety, however named.		920-321-9426
Chad Rusch	Inorganic Department Manager[3]		920-321-9438
Christopher Haase	SVOA Department Manager[3]		920-321-9453
Scott Turner	VOA Department Manager[3]		920-321-9425

[1] *Members of the local management team are subject to change during the life-cycle of this document version.*
[2] *Include if different from the physical address and phone number of the facility.*
[3] *This individual serves as an Acting Technical Manager for TNI for one or more fields of accreditation.*

Table of Contents

1.0 PURPOSE AND SCOPE

1.1 Purpose

This quality manual (manual) outlines the quality management system and management structure of the laboratories and service centers affiliated with Pace Analytical Services, LLC (PAS). A laboratory is defined by PAS as any PAS facility, however named, that provides testing, sampling, or field measurement services. When the term 'laboratory" is used in this manual, the term refers to all locations listed on the Title Page of this manual and in Section 4.1.3 unless otherwise specified.

The PAS quality management system is also referred to as the quality program throughout this document. In this context, the phrase "quality management system" and "quality program" are synonymous.

The quality management system is the collection of policies and processes established by PAS management to consistently meet customer requirements and expectations, and to achieve the goals to provide PAS customers with high quality, cost-effective, analytical measurements and services.

The quality management system is also intended to establish conformance[1] and compliance with the current versions of the following international and national quality system standards:

- ISO/IEC 17025: *General requirements for the competence of testing and calibration laboratories*
- NELAC/TNI Standard Volume 1: *Management and Technical Requirements for Laboratories Performing Environmental Analysis*

[1]The statement of conformity to these Standards pertains only to testing and sampling activities carried out by the laboratory at its physical address, in temporary or mobile facilities, in-network, or by laboratory personnel at a customer's facility.

In addition to the international and national standards, the quality management system is designed to achieve regulatory compliance with the various federal and state programs for which the laboratory provides compliance testing and/or holds certification or accreditation. When federal or state requirements do not apply to all PAS locations, the requirements for compliance are provided in addendum to this manual or in other documents that supplement the manual. Customer-specific project and program requirements are not included in the manual in order to maintain client confidentiality.

- A list of accreditation and certifications held by each laboratory associated with this manual is provided in Appendix A.
- A list of analytical testing capabilities offered by each laboratory associated with this manual is provided in Appendix B.

1.2 Scope and Application

This manual applies to each of the PAS locations listed on the Title Page and in Section 4.1.3.

The manual was prepared from a quality manual template (template) created by PAS corporate quality personnel. The template outlines the minimum requirements PAS management considers necessary for every PAS laboratory, regardless of scope of services or number of personnel, to establish in order to maintain a quality management system that achieves the objectives of PAS's Quality Policy (See 4.2.2). In this regard, the template is the mechanism used by the corporate officers (a.k.a. 'top management') to communicate their expectations and commitment for the PAS quality program to all PAS personnel.

The laboratory also has the responsibility to comply with federal and state regulatory and program requirements for which it provides analytical services and holds certification or accreditation. When those requirements are more stringent than the template, the requirements for compliance are provided in addendum to this manual or in other documents that supplement the manual. This document structure maintains consistency in the presentation of the quality management system across the network while providing the laboratory a mechanism to describe and achieve compliance requirements on a program basis.

1.2.1 Quality Manual Template

The quality manual template is developed by the Corporate Quality Director with contribution and input from corporate quality personnel and the corporate officers. Approval of the template by the corporate officers (aka "top management") confirms their commitment to develop and maintain a quality management system appropriate for the analytical services offered by the organization and to communicate their expectations of the quality program to all personnel.

The template and instructions for use of the template are released by corporate quality personnel to quality assurance manager(s) responsible for each laboratory (Local QA). Local QA uses the template to prepare the laboratory's manual by following the instructions provided. Since the template provides the minimum requirements by which all PAS locations must abide, the laboratory may not alter the font, structure or content of the template except where specified by instruction to do so. As previously stated, program specific requirements are provided in addendum or in documents that supplement this manual.

The template is reviewed by corporate quality personnel every two years and updated if needed. More frequent review and revision may be necessary to manage change, to maintain conformance and compliance to relevant standards, or to meet customer expectations.

See standard operating procedure (SOP) ENV-SOP-CORQ-00015 *Document Management and Control* (most current revision or replacement) for more information.

1.2.2 Laboratory Quality Manual

The manual is approved and released to personnel under the authority of local management. The manual is reviewed annually and location specific information is updated, if needed. More frequent review and revision may be necessary when there are significant changes to the organizational structure, capabilities, and resources of the laboratory. Review and revision of the manual is overseen by local QA. If review indicates changes to the main body of the manual are necessary to maintain conformance and compliance to relevant standards, or to meet customer expectations, local QA will notify corporate quality personnel to initiate review and/or revision of the template.

See SOP ENV-SOP-CORQ-00015 *Document Management and Control* (most current revision or replacement) for more information.

1.2.3 References to Supporting Documents

The template and the manual includes references to other laboratory documents that support the quality management system such as policies and standard operating procedures (SOPs). These references include the document's document control number and may include the document title.

This information is subject to change. For example, an SOP may be converted to a policy or the document's title may change. For these types of administrative changes, the manual and template are updated to reflect the editorial change during the document's next scheduled review/revision cycle or the next time a new version of the document is released, whichever is sooner.

Local QA maintains a current list of controlled documents used at each PAS location to support the quality management system. This list, known as the Master List, lists each document used by document control number, title, version, effective date, and reference to any document(s) that the current version supersedes. When there is a difference between the template and/or manual and the Master List, the document information in the Master List takes precedence. The current Master List is readily available to personnel for their use and cross-reference. Parties external to the laboratory should contact the laboratory for the most current version.

2.0 REFERENCES

References used to prepare this manual include:

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, most current version.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, current version.
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, current version.
- "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.
- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.
- "NIOSH Manual of Analytical Methods", U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health, most current version.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (Sep 1986).
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.
- Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.
- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.

- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.
- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, most current version.
- National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.
- ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories- most current version.

  The following are implemented by normative reference to ISO/IEC 17025:

  - ISO/IEC Guide 99, *International vocabulary of metrology –Basic and general concepts and associated terms*
  - ISO/IEC 17000, *Conformity assessment— Vocabulary and general principles*
- Department of Defense Quality Systems Manual (QSM), most current version.
- TNI (The NELAC Institute) Standard- most current version applicable to each lab.
- UCMR Laboratory Approval Requirements and Information Document, most current version.
- US EPA Drinking Water Manual, most current version.

3.0 TERMS AND DEFINITIONS

Refer to Appendix C for terms, acronyms, and definitions used in this manual and in other documents used by the laboratory to support the quality management system.

4.0 MANAGEMENT REQUIREMENTS

4.1 Organization

4.1.1 Legal Identity

Pace Analytical Services, LLC is authorized under the State of Minnesota to do business as a limited liability company.

4.1.1.1 Change of Ownership

If there is a change of ownership, if a location goes out of business, or if the entire organization ceases to exist, Pace Analytical Services, LLC ensures that regulatory authorities are notified of the change within the time-frame required by each state agency for which the location is certified or accredited.

Requirements for records and other business information are addressed in the ownership transfer agreement or in accordance with appropriate regulatory requirements, whichever takes precedence.

4.1.2 Compliance Responsibility

Laboratory management has the responsibility and authority to establish and implement procedures and to maintain sufficient resources necessary to assure its activities are carried out in such a way to meet the compliance requirements of the quality management system.

4.1.3 Scope of the Quality Management System

The quality management system applies to work carried out at each location covered by this manual including permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

The permanent and mobile facilities to which this manual applies includes:

Name	Pace Analytical Services, LLC
Address:	1241 Bellevue Street, Suite 9
City, State, Zip	Green Bay, WI 54302
Phone Number	920-469-2436
Service Type:	Laboratory

4.1.4 Organization History and Information

Founded in 1978, Pace Analytical Services, LLC (PAS) is a privately held scientific services firm operating one of the largest full service contract laboratory and service center networks in the United States. The company's network offer inorganic, organic and radiochemistry testing capabilities; specializing in the analysis of trace level contamination in air, drinking water, groundwater, wastewater, soil, biota, and waste.

With over 90 laboratories and services centers in the contiguous US and in Puerto Rico, the network provides project support for thousands of industry, consulting, engineering and government professionals.

Pace delivers the highest standard of testing and scientific services in the market. We offer the most advanced solutions in the industry, backed by truly transparent data, a highly trained team, and the service and support that comes from four decades of experience.

4.1.4.1 Organization Structure

Each location maintains a local management structure under the oversight and guidance of corporate personnel. Local management is responsible for making day-to-day decisions regarding the operations of the facility, implementing the quality management system, upholding the requirements of the quality program, and for supervision of personnel.

Local management is provided by a General Manager I (GMI), General Manager II (GMII), Quality Manager (QM), Client Services Manager (CSM), Information Technology (IT) Manager, Department Managers (DM) and/or Department Supervisors (DS), however named.

Some locations may also have any one of the following management positions: Operations Manager (OM). When the location does not have an OM, technical management is provided jointly by the GMI, GMII, QM, DM, and DS.

The GMI (or GMII), however named reports to a Regional Director - Operations (RDO), who is responsible for the management of multiple laboratories and service centers within a geographical region, and who reports directly to the Chief Operating Officer (COO). The QM has indirect reporting relationship to the Corporate Director of Quality.

Refer to the organization charts provided in Appendix D to view the management structure, reporting relationships, and the interrelationships between positions.

4.1.5 Management Requirements

4.1.5.1 Personnel

The laboratory is staffed with administrative and technical personnel who perform and verify work under the supervision of managerial personnel.

- Technical personnel include analysts and technicians that generate or contribute to the generation of analytical data and managerial personnel that oversee day to day supervision of laboratory operations. Including the reporting of analytical data and results, monitoring QA/QC performance, and monitoring the validity of analysis to maintain data integrity and reliability.
- Administrative personnel support the day-to-day activities of the laboratory.
- IT personnel maintain the information technology systems and software used at the laboratory.
- Client services personnel include project managers and support staff that manage projects.
- Managerial personnel make day-to-day and longer term decisions regarding the operations of the facility, supervise personnel, implement the quality management system and uphold the requirements of the quality program.

All personnel regardless of responsibilities are expected to carry out their duties in accordance with the policies and processes outlined in this manual and in accordance with standard operating procedures (SOPs) and other quality system documents. The laboratory's policies and procedures are designed for impartiality and integrity. When these procedures are fully implemented, personnel remain free from undue pressure and other influences that adversely impact the quality of their work or data.

4.1.5.1.1 Key Personnel

Key personnel include the management positions that have the authority and responsibility to plan, direct, and control, activities of the division (corporate) or the laboratory.

The following tables list key personnel positions by PAS job title and the position's primary deputy:

Key Personnel: Corporate

Key Personnel	Primary Deputy
Chief Executive Officer	Chief Operating Officer
Chief Operating Officer	Chief Executive Officer
Chief Compliance Officer	Quality Director
Corporate Quality Director	Chief Compliance Officer
Health and Safety Director	Chief Compliance Officer
IT Director	LIMS Administrator, however named.

Key Personnel: Laboratory

Key Personnel	Primary Deputy
Regional Director - Operations	Chief Operating Officer or as designated.
General Manager I/ General Manager II	Regional Director - Operations
Quality Manager	Corporate Quality Manager or as designated.
Client Services Manager	General Manager I or II
Local IT	Corporate IT Director or as designated.
Department Manager	General Manager I or II
Manager[1] Acting Technical Manager TNI	Quality Manager
Operations Manager[1]	General Manager I or II

[1] *Position may not be staffed at each location.*

Some state certification programs require the agency to be notified when there has been a change in key personnel. Program-specific requirements and time-frames for notification by agency, are tracked and upheld by local QA, when these requirements apply.

4.1.5.2 Roles and Responsibilities

The qualifications, duties, and responsibilities for each position are detailed in job descriptions maintained by PAS's corporate Human Resource's Department (HR).

The following summaries briefly identify the responsibility of key personnel positions in relation to the quality management system.

Chief Executive Officer (CEO): The CEO has overall responsibility for performance of the organization and endorses the quality program. Working with corporate and laboratory management, the CEO provides the leadership and resources necessary for PAS locations to achieve the goals and objectives of the quality management system and quality policy statement.

Chief Operating Officer (COO): The COO oversees all aspects of operations management including, strategic planning, budget, capital expenditure, and management of senior management personnel. In this capacity, the COO provides leadership and resources necessary to help top management at each PAS location achieve the goals and objectives of the quality management system and quality policy statement.

Chief Compliance Officer (CCO): The CCO oversees the quality assurance and environmental health and safety programs (HSE) for each business unit. The CCO is responsible for planning and policy development for these groups to ensure regulatory compliance and to manage risk. The position provides leadership and guidance necessary for all PAS locations to achieve the goals and objectives of the quality and HSE programs.

The CCO also serves as the Ethics Officer (ECO). The ECO develops the Ethics and Data Integrity Policy and Training Program, and provides oversight for reporting and investigation of ethical misconduct to maintain employee

confidentiality during the process. The ECO provide guidance and instruction for follow-up actions necessary to remedy the situation and deter future recurrence.

Corporate Director of Quality: The Corporate Director of Quality is responsible for developing and maintaining the PAS quality program under guidance and assistance from the CEO, COO, and CCO. This position helps develop corporate quality policy and procedure and analyzes metric data and other performance indicators to assess and communicate the effectiveness of the quality program to top management. The position provides leadership and guidance for implementation of the quality program across all PAS locations.

Corporate Director of Information Technology: The Corporate Director of IT oversees the systems and processes of information technology used to support the quality program. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity and security of electronic data.

Regional Director – Operations (RDO): The RDO has full responsibility for administrative and operations management and performance of a group of PAS laboratories and service centers. Working with the COO and local laboratory management, the SGM provides leadership, guidance and resources, including allocation of personnel, necessary to achieve the goals of PAS quality program.

General Manager I (GMI) / General Manager II (GMII): The GMI or GMII is responsible for the overall performance and administrative and operations management of a PAS location and associated service center(s). This position is responsible to provide leadership and resources, including allocation and supervision of personnel, necessary for the location to implement and achieve the goals of the PAS quality program. In this capacity, the position assures laboratory personnel are trained on and understand the structure and components of the quality program defined in this manual as well as the policies and procedures in place to implement the quality management system.

The GMI/GMII of NELAC/TNI Accredited laboratories are also responsible for the designation of technical personnel to serve as acting technical managers for TNI for the fields of accreditation held by the laboratory (See Section 4.1.5.2.2) and for notifying the accreditation body (AB) of any extended absence or reassignment of these designations.

Quality Manager (QM): The QM oversees and monitors implementation of the quality management system and communicates deviations to laboratory management. The QM is independent of the operation activities for which they provide oversight and has the authority to carry out the roles and responsibilities of their position without outside influence.

Additionally, in accordance with the TNI Standard, the QM:

- serves as the focal for QA/QC and oversees review of QC data for trend analysis;

- evaluates data objectively and perform assessments without outside influence;
- has document training and experience in QA/QC procedures and the laboratory's quality system;
- has a general knowledge of the analytical methods offered by the laboratory;
- coordinates and conducts internal systems and technical audits;
- notifies laboratory management of deficiencies in the quality system;
- monitors corrective actions;
- provides supports to technical personnel and may serve as the primary deputy for the acting TNI Technical Manager(s).

Client Services Manager (CSM): The CSM oversees project management personnel. This position is responsible for training and management of client facing staff that serve as the liaison between PAS and the customer to ensure that projects are successfully managed to meet the expectations and needs of PAS customers. This position is also responsible for sharing positive and negative customer feedback with laboratory management so that this information may be used to improve the quality program.

Local IT Manager, however named: Local IT managers are responsible for maintaining the IT systems used to support the quality program. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity and security of electronic data.

Department Manager (DM): The DM is responsible for administrative and operations management and implementation of the quality management system in the work area he/she oversees. These responsibilities include but are not limited to: training and supervision of personnel, monitoring work activity to maintain compliance with this manual, SOPs, policies and other instructional documents that support the quality management system; method development, validation and the establishment and implementation of SOPs to assure regulatory compliance and suitability for intended purpose; monitoring QA/QC performance, proper handling and reporting of nonconforming work, purchasing of supplies and equipment adequate for use, maintaining instrumentation and equipment in proper working order and calibration, and general maintenance of administrative and technical processes and procedures established by the laboratory.

Operations Manager (OM): The OM is responsible for management of production and/or other duties assigned by the GM or SGM.

4.1.5.2.1 **Acting Technical Manager (TNI Accreditation):**

For PAS locations that are NELAC/TNI accredited:

The TNI Standard specifies requirements for the qualification and duties of technical personnel with managerial responsibility. These requirements are associated in the Standard to the designation

'technical manager(s), however named'. These responsibilities may be assigned to multiple individuals and are not associated with any specific job title.

For PAS, these TNI requirements for personnel that provide technical oversight correlate with PAS's job descriptions for Department Manager or Supervisor. However, the duties may be assigned to any PAS employee that meets the TNI specified qualifications.

Personnel assigned this designation retain their PAS assigned job title. The job title may be appended with *"acting as technical manager for TNI"* and the technology or field of accreditation for which the employee is approved, if necessary.

When TNI Accreditation Bodies (AB) refer to these employees as 'technical manager' or 'technical director' on the official certificate or the scope of accreditation, this reference is referring to their approval to carry out duties of the 'technical manager, however named' as specified in the TNI Standard.

In accordance with the TNI Standard, the acting Technical Manager(s) for TNI are responsible for monitoring the performance of QC/QA in the work areas they oversee.

If the absence of any employee that is approved as acting technical manager for TNI exceeds 15 calendar days, the duties and responsibilities specified in the TNI Standard are reassigned to another employee that meets the qualifications for the technology or field of accreditation or they are assigned to the position's deputy, the quality manager.

4.1.5.3 Conflict of Interest

A conflict of interest is a situation where a person has competing interests. Laboratory management looks for potential conflict of interest and undue pressures that might arise in work activities and then includes countermeasures in policies and procedures to mitigate or eliminate the conflict.

See policy COR-POL-0004 *Ethics Policy* for more information.

4.1.5.4 Confidentiality

Laboratory management is committed to preserving the confidentiality of PAS customers and confidentiality of business information.

Procedures used by the laboratory to maintain confidentiality include:

- A Confidentiality Agreement which all employees are required to sign at the time of employment and abide by the conditions of throughout employment;
- Record retention and disposal procedures that assure confidentiality is maintained;

- Physical access controls and encryption of electronic data; and
- Protocol for handling Confidential Business Information (CBI).

Client information obtained or created during work activities is considered confidential and is protected from intentional release to any person or entity other than the client or the client's authorized representative information provided to PAS, except when the laboratory is required by law to release confidential information to another party, such as a regulatory agency or for litigation purposes. In which case, the laboratory will notify the client of the release of information and the information provided.

The terms of client confidentiality are included in PAS Standard Terms and Conditions (T&C). With the acceptance of PAS Terms and Conditions and/or the implicit contract for analytical services that occurs when the client sends samples to the laboratory for testing, the client authorizes PAS to release confidential information when required.

See policy COR-POL-0004 *Ethics Policy* for more information.

4.1.5.5 Communication

Communication is defined as the imparting or exchanging of news and information. Effective (good) communication occurs when the person(s) you are exchanging information with actively gets the point and understands it.

4.1.5.5.1 Workplace Communication

Good communication in the workplace is necessary to assure work is done correctly, efficiently, and in accordance with client expectations.

Instructions for how to carry out work activities are communicated to personnel via written policy, standard operating procedures, and standard work instructions.

Information about laboratory performance (positive and negative) and ideas for improvement are communicated using various communication channels such as face to face meetings, video conferencing, conference calls, email, memoranda, written reports, and posters.

4.1.5.5.2 External Communication

Communication with external parties such as customers, vendors, business partners, and regulatory agencies takes place every day.

Laboratory management ensure personnel learn to communicate in professional and respectful ways in order to build strong relationships, and learn to communicate effectively to avoid misunderstanding.

4.2 Quality Management System

4.2.1 Quality Management System Objectives

The objectives of the laboratory's quality management system are to provide clients with consistent, exemplary professional service, and objective work product that is of known and documented quality that meets their requirements for data usability and regulatory compliance.

Objective work product is analytical services, data, test results, and information that is not influenced by personal feeling or opinions. The quality of being objective is also known as 'impartiality'.

4.2.1.1 Impartiality

The laboratory achieves and maintains impartiality by implementing and adhering to the policies and processes of the quality management system, which are based on industry accepted standards and methodologies.

The laboratory's procedures for handling nonconforming work (See 4.9), corrective and preventive actions (See 4.11) and management review (See 4.15) are the primary mechanisms used to identify risk to impartiality and to prompt actions necessary to eliminate or reduce the threat when risk to impartiality is suspected or confirmed.

4.2.1.2 Risk and Opportunity Assessment

Risks are variables that make achieving the goals and objectives of the quality management system uncertain. An opportunity is something that has potential positive consequences for the laboratory.

Laboratory personnel manage risks and opportunities on a daily basis by carrying out the processes that make up the quality management system. Some of the ways in which the quality management system is designed to identify, minimize, or eliminate risk on a daily basis include but are not limited to:

- Capability and capacity reviews of each analytical service request to assure the laboratory can meet the customer's requirements;
- Maintenance of accreditation and certification for test methods in multiple states and programs to cover a broad range of jurisdiction for regulatory compliance;
- SOPs and other controlled instructional documents are provided to personnel to eliminate variability in process. These documents include actions to counter risk factors inherent in the process and are reviewed on a regular basis for on-going suitability and relevancy;
- Participation in proficiency testing programs and auditing activities to verify on-going competency and comparability in performance;
- Provision of on-the-job training and established protocol for quality control (QC) corrective action for nonconforming events;
- An established program for ethics, and data integrity;

- Tiered data review process;
- Culture of continuous improvement;
- Monitoring activities to assess daily and long term performance; and
- Annual critical review of the effectiveness of the quality management system.

PAS also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by Pace to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction. PAS's lean programs and activities help to mitigate risk because they generate a collective understanding of vulnerabilities and utilize group-effort to develop and implement solutions at all levels.

Risk and opportunities may also be formally identified using specific risk and opportunity assessment methods such as SWOT Analysis (Strength, Weakness, Opportunity, Threats) and 3-Stage Impact/Probability Grids.

4.2.1.3 Communication of the Quality Management System

This manual is the primary mechanism used by laboratory management to communicate the quality management system to laboratory personnel.

To assure personnel understand and implement the quality program outlined in the manual:

- All laboratory personnel are required to sign a Read and Acknowledgement Statement to confirm the employee has: 1) been informed of the manual by laboratory management, 2) has access to the manual, 3) has read the manual 4) understands the content of the manual, and 5) agrees to abide by the requirements, policies and procedures therein.
- Personnel are informed that the manual provides the "what" of the quality management system. The "how to" implementation of the quality management system is provided in policy, SOPs, standard work instructions, and other controlled instructional documents.

4.2.2 Quality Policy Statement

The quality policy of the laboratory is to provide customers with data of known and documented quality fit for their intended purpose. The laboratory achieves this policy by implementing the quality management system defined in this manual, by following industry accepted protocol for analytical testing and quality assurance and quality control (QA/QC) activities, by conformance with published and industry accepted testing methodologies, and by compliance with international and national standards for the competency and/or accreditation of testing laboratories.

Intrinsic to this policy statement is each of the following principles:

- The laboratory will provide customers with reliable, consistent, and professional service. This is accomplished by making sure the laboratory has the resources necessary to maintain capability and capacity; that staff are trained and competent to perform the tasks they are assigned; that client-facing staff are trained and prepared to find solutions to problems and to assist customers with their needs for analytical services. Customer feedback, both positive and negative, is shared with personnel and used to identify opportunities for improvement.
- The laboratory maintains a quality program that complies with applicable, state, federal, industry standards for analytical testing and competency.

  ISO/IEC 17025 and the TNI (The NELAC Institute) Standard is used by PAS to establish the minimum requirements of the PAS quality program.

  ISO/IEC 17025 is a competency standard that outlines the general requirements for the management system for calibration and testing laboratories. It is the primary quality system standard from which other quality system standards, such as the TNI Standard, are based. The TNI Standard are consensus standards that provides management and technical requirements for laboratories performing environmental analysis.
- Laboratory management provides training to personnel so that all personnel are familiar with the quality management system outlined in this manual and that they understand that implementation of the quality management system is achieved by adherence to the organization's policies and procedures.
- Laboratory management continuously evaluates and improves the effectiveness of the quality management system by responding to customer feedback, and other measures of performance, such as but not limited to: the results of internal/external audits, proficiency testing, metrics, trend reports, and annual and periodic management reviews.

4.2.2.1 Ethics Policy / Data Integrity Program

PAS has established a comprehensive ethics and data integrity program that is communicated to all PAS employees in order that they understand what is expected of them. The program is designed to promote a mindset of ethical behavior and professional conduct that is applied to all work activities.

The key elements of the PAS Ethics / Data Integrity Program include:

- Ethics Policy (COR-POL-0004);
- Ethics Compliance Officer;
- Standardized data integrity training course taken by all new employees on hire and a yearly refresher data integrity training course for all existing employees;
- Policy Acknowledgement Statements that all PAS personnel, including contract and temporary, are required to sign at the time of employment and again during annual refresher training to document the employee's commitment and obligation to abide by the company's standards for ethics, data integrity and confidentiality;

- SOPs that provide instructions for how to carry out a test method or process to assure tasks are done correctly and consistently by each employee;
- On the Job Training;
- Data integrity monitoring activities which include, but are not limited to, secondary and tertiary data review, internal technical and system audits, raw data audits, data mining scans, and proficiency testing; and
- Confidential reporting process for alleged ethics and data integrity issues.

All laboratory managers are expected to provide a work environment where personnel feel safe and can report unethical or improper behavior in complete confidence without fear of retaliation. Retaliation against any employee that reports a concern is not tolerated.

PAS has engaged Lighthouse Services, Inc. to provide personnel with an anonymous reporting process available to them 24 hours a day/7 days per week. The alert line may be used by any employee to report possible violations of the company's ethics and data integrity program. When using the reporting process, the employee does need to specify the location of concern and when reporting by email, also include the company name. Messages are collected, documented, reviewed, and will be followed up on by the Ethics Compliance Officer to resolve the matter. Investigations concerning data integrity are kept confidential.

Lighthouse Compliance Alert Lines:

English Speaking US & Canada	(844) 940-0003
Spanish Speaking North America	(800) 216-1288
Internet	www/lighthouse-services.com/pacelabs
Email	reports@lighthouse-services.com

4.2.3 Management Commitment: Quality Management System

Evidence of management's commitment for the development, maintenance, and on-going improvement of the quality management system is provided by the application of their signature of approval to this manual. Their signature confirms they understand their responsibility to implement the quality management system outlined in this manual, to communicate the quality program to personnel, and to uphold requirements of the program during work activities.

4.2.4 Management Commitment: Customer Service

Management communicates the importance of meeting customer and regulatory requirements to personnel by training personnel on the quality management system outlined in this manual, implementing the quality management system outlined in this manual, and upholding these requirements for all work activities.

4.2.5 Supporting Procedures

Documents that support this manual and quality management system are referenced throughout this manual. The structure of the document management system is outlined in

SOP ENV-SOP-CORQ-0015 *Document Management and Control* (most current revision or replacement) and summarized in the following subsections.

4.2.5.1 Quality Management System Document Structure

Documents associated with the quality management system are classified into document types that identify the purpose of the document and establish how the document is managed and controlled.

Document types are ranked to establish which documents takes precedence when there is an actual or perceived conflict between documents and to establish the hierarchal relationships between documents. The ranking system also provides information to document writers and reviewers to assure downline documents are in agreement with documents of higher rank. Project specific documents are not ranked because client specific requirements are not incorporated into general use documents in order to maintain client confidentiality.

PAS Quality Management System Documents: Internal

Document Type	Purpose
Quality Manual	Outlines the laboratory's quality management system and structure and how it works for a system including policy, goals, objectives and detailed explanation of the system and the requirements for implementation of system. Includes roles and responsibilities, relationships, procedures, systems and other information necessary to meet the objectives of the system described.
Policy	Provide requirements and rules for a PAS process and is used to set course of actions and to guide and influence decisions. Policy describes the "what", not the "how".
Standard Operating Procedure	Provide written and consistent set of instructions or steps for execution of a routine process, method, or set of tasks performed by PAS. Includes both fundamental and operational elements for implementation of the systems described in PAS manual(s). Assures that activities are performed properly in accordance with applicable requirements. Designed to ensure consistency, protect EHS of employees and environment, prevent failure in the process and ensure compliance with company and regulatory requirements. SOPs describes the "how" based on policy.
Standard Work Instruction	Provide step by step visual and/or written instruction to carry out a specific task to improve competency, minimize variability, reduce work injury and strain, or to boost efficiency and quality of work (performance). SWI are associated with an SOP unless the task described is unrelated to generation of or contribution to environmental data or analytical results.
Template	Pre-formatted document that serves as a starting point for a new document.
Guide	Provide assistance to carry out a task. Most often used for software applications.
Form	Used for a variety of purposes such as to provide a standardized format to record observations, to provide information to supplement an SOP.

PAS Quality Management System Documents: External

Certificate	Lists parameters, methods, and matrices for which the laboratory is certified/accredited to perform within the jurisdiction of the issuing regulatory agency or accreditation body.
Reference Document	Provide information, protocol, instructions, and/or requirements. Issued by the specifier. Examples include quality system standards such as ISO/IEC, TNI, DoD and published referenced methods such as Standard Methods, ASTM, SW846, EPA, and federal and state regulatory bodies.
Project Document	Provides requirements necessary to meet individual client expectations for intended use of data. Examples include: project quality assurance plans (QAPP), client program technical specifications, contracts, and other agreements.

Document Hierarchy

Rank	Document
1	Reference Documents
2	Corporate Manual
3	Corporate Policy
4	Corporate SOP
5	Corporate SWI, Templates & Forms
6	Laboratory Manual
7	Laboratory SOP
8	Laboratory SWI, Templates, & Forms
NA	Project Documents[4]

4.2.6 Roles and Responsibilities

The roles and responsibilities of technical management and of the quality manager are provided in section 4.1.5.1.2.

4.2.7 Change Management

When significant changes to the quality management system are planned, these changes are managed by corporate quality personnel to assure that the integrity of the quality management system is maintained.

4.3 Document Control

4.3.1 General

The laboratory's procedures for document control are provided in SOP ENV-SOP-CORQ-0015 *Document Management and Control* (most current revision or replacement).

The documents that support the quality management system include internally generated documents such as manuals, policies, standard operating procedures, standard work instructions, forms, guides, and templates and external source documents such as but not limited to, regulations, standards, reference methods, manuals, and project-specific documents.

The laboratory uses electronic document management software (eDMS) to carry out the procedures of the SOP. eDMS automates the process for unique document identification, version control, approval, access, and archival.

4.3.2 Document Approval and Issue

Documents that are part of the quality management system are reviewed by qualified personnel and approved by laboratory management prior by to release for general use.

Local QA maintains a master list of controlled documents used at the laboratory. The master list includes the document control number, document title, and current revision status and is made available to personnel for their reference.

Only the approved versions of documents are available to personnel for use. The eDMS system does not allow user access to draft versions of documents except to personnel assigned to work on the draft. eDMS also restricts access to archived documents except to authorized users, such as local QA, in order to prevent the use of obsolete documents.

See SOP ENV-SOP-CORQ-0015 *Document Management and Control* (most current revision or replacement) for more information.

4.3.3 Document Review and Change

Unless a more frequent review is required by regulatory, certification or accreditation program. The laboratory formally reviews documents at least every two years to ensure the document remains current, appropriate, and relevant.

Documents are also informally reviewed every time the document is used. Personnel are expected to refer to and follow instructions in controlled documents when they carry out their work activities. Consequently, any concerns or problems with the document should be caught and brought to the attention of laboratory management on an on-going basis.

Documents are revised whenever necessary to ensure the document remains usable and correct. Older document versions and documents no longer needed are made obsolete and archived for historical purposes.

The laboratory does not allow hand-edits to documents. If an interim change is needed pending re-issue of the document, the interim change is communicated to those that use the document using a formal communication channel, such as SOP Change in Progress form, email, or memorandum.

The document review, revision, and archival process is managed by local QA at the location from which the document was released using the procedures established in SOP ENV-SOP-CORQ-0015 *Document Management and Control* (most current revision or replacement).

4.4 Analytical Service Request, Tender, and Contract Review

The laboratory's management and/or client service personnel perform thorough reviews of requests and contracts for analytical services to verify the laboratory has the capability, capacity, and resources necessary to successfully meet the customer's needs. These review procedures are described in laboratory SOP ENV-SOP-GBAY-0006, *Sample Management and Review of Analytical Requests* (most current revision or replacement).

The procedures in this SOP(s) are established to ensure that:

- The laboratory understands the purpose of data collection in order to ensure the test methods requested are appropriate for the intended use of the data and capable of meeting the client's data quality objectives;

- The laboratory and any subcontractor has the capability, capacity, and resources to meet the project requirements and expectations within the requested time frame for delivery of work product;
- Any concerns that arise from review are discussed and resolved with the client; and
- The results of review and any correspondence with the client related to this process and/or any changes made to the contract are recorded and retained for historical purposes.

Capability review confirms that the in-network laboratories and any potential subcontractors hold required certification/accreditation for the test method, matrix, and analyte and verifies the laboratory can achieve the client's target compound list and data quality objectives (DQOs) for analytical sensitivity and reporting limits, QA/QC protocol, and hardcopy test report and electronic data deliverable (EDD) formats.

Capacity review verifies that the in-network laboratories and any potential subcontractors are able to handle the sample load and deliver work production within the delivery time-frame requested.

Resource review verifies that the laboratory and any potential subcontractors have adequate qualified personnel with the skills and competency to perform the test methods and services requested and sufficient and proper equipment and instrumentation needed to perform the services requested.

4.5 Subcontracting and In-Network Work Transfer

The terms 'subcontract' and "subcontracting" refers to work sent to a business external to Pace Analytical Services, LLC (PAS) and the term 'subcontractor' refers to these external businesses, which are also called vendors.

Work transferred within the PAS network is referred to as interregional work orders (IRWO) and network laboratories are referred to as IRWO or network laboratory.

The network of PAS laboratories offers comprehensive analytical capability and capacity to ensure PAS can meet a diverse range of client needs for any type of project. If the laboratory receives a request for analytical services and it cannot fulfill the project specifications, the laboratory's client services team will work with the client to place the work within the PAS network. When it is not possible to place the work within network, the laboratory will, with client approval, subcontract the work to a subcontractor that has the capabilities to meet the project specifications and can meet the same commitment agreed on between the laboratory and the client. Some client programs require client consent even for IRWO work transfer, and when this applies, the client services team obtains consent as required. The laboratory retains the record of client notification and their consent in the project record for historical purposes.

Whenever work is transferred to a subcontractor or an IRWO laboratory, the laboratory responsible for management of the project verifies each of these qualifications:

- The subcontractor or IRWO laboratory has the proper accreditation/certifications required for the project and these are current; and
- The use of the subcontractor or IRWO laboratory is approved by the client and/or regulatory agency, when approval is required. Record of approval is retained in the project record.

When possible, the laboratory selects subcontractors that maintain a quality management system similar to PAS and that complies with ISO/IEC 17025 and the TNI Standard(s).

PAS also evaluates and pre-qualifies subcontractors as part of company's procurement program. The complete list of approved vendors is maintained by the corporate procurement department and is made available to all PAS locations. Pre-qualification of a subcontractor does not replace the requirement for the placing laboratory to verify the capability, capacity, and resources of any selected subcontractor on a project-specific basis to confirm the subcontractor can meet the client's needs.

For both subcontracting and in-network work transfer, the project specifications are always communicated to the subcontractor or the IRWO laboratory by the project manager so that the laboratory performing the work is aware of and understands these requirements.

The procedures for subcontracting are outlined in laboratory SOP ENV-SOP-GBAY-0005, *Subcontracting Samples* (most current revision or replacement).

4.6 Purchasing Services and Supplies

Vendors that provide services and supplies to the laboratory are prequalified by corporate procurement personnel to verify the vendor's capability to meet the needs of PAS. These needs include but are not limited to: competitive pricing, capacity to fill purchase orders, quality of product, customer service, and business reputation and stability. The records of vendor evaluation and the list of approved vendors is maintained by the corporate procurement department.

The laboratory may purchase goods and services from any supplier on the approved vendor list.

The specifications (type, class, grade, tolerance, purity, etc.) of supplies, equipment, reagents, standard reference materials and other consumables used in the testing process are specified in SOPs. The SOP specifications are based on the governing requirements of the approved reference methods and any additional program driven regulatory specification, such as drinking water compliance. All requisitions for materials and consumables are approved by the department supervisor to confirm the purchase conforms with specified requirements. After approval the requisition is handled by the laboratory's designated purchasing agent. On receipt, the product is inspected and verified before use, when applicable.

The laboratory's procedure for the purchase of services and supplies is specified in laboratory SOP ENV-SOP-GBAY-0145, *Laboratory Supply Procedures* (most current revision or replacement).

4.7 Customer Service

Project details and management is handled by the laboratory's customer service team. Each customer is assigned a Project Manager (PM) that is responsible for review of contract requirements and handling laboratory to customer communication about the project status.

4.7.1 Commitment to Meet Customer Expectations

The laboratory cooperates and works closely with our customers to ensure their needs are met and to establish their confidence in the laboratory's capability to meet their needs for analytical services and expectations for service.

Each customer's project is handled by a project manager (PM) that is the customer's primary point of contact. The PM gathers information from the customer to ensure the details of

their request are understood. After samples are received, the PM monitors the progress of the project and alerts the customer of any delays or excursions that may adversely impact data usability. Laboratory supervisors are expected to keep the PM informed of project status and any delays or major issues, so that the PM can keep the client informed.

PAS also has a team of subject matter experts (SME) available to provide customers with advice and guidance and any other assistance needed. SME are selected by top management based on their knowledge, experience, and qualifications.

The laboratory encourages customers to visit the laboratory to learn more about the laboratory's capabilities, observe performance and to meet laboratory personnel.

PAS customers expect confidentiality. Laboratory personnel will not divulge or release information to a third party without proper authorization unless the information is required for litigation purposes. See Section 4.1.5.3 of this manual and policy COR-POL-0004 *Ethics Policy* for more information on the laboratory's policy for client confidentiality.

4.7.2 Customer Feedback

The laboratory actively seeks positive and negative feedback from customers through surveys and direct communication. Information from the client about their experience working with the laboratory and their satisfaction with work product is used to enhance processes and practices and to improve decision making. Customer feedback is communicated to laboratory management and corporate personnel in monthly reports and analyzed yearly during management review (See 4.15) to identify risk and opportunity. Corrective, preventive, or continuous improvement actions are taken based on nature of and/or feedback trends.

Also see sections 4.9, 4.10, 4.11, 4.12, 4.14, and 4.15 for more information about how customer feedback is managed by the laboratory and used to enhance the quality management system.

4.8 Complaints

Complaints provide opportunities to improve processes and build stronger working relationships with our clients.

The laboratory's complaint resolution process includes three steps. First, handle and resolve the complaint to mutual satisfaction. Second, perform corrective action to prevent recurrence (See 4.11). Third, record and track the complaint and use these records for risk and opportunity assessment and preventive action (See 4.12)

4.9 Nonconforming Work

4.9.1 Definition of Nonconforming Work

Nonconforming work is work that does not conform to customer requirements, standard specifications, laboratory policies and procedures, or that does not meet acceptance criteria.

The discovery of non-conforming work comes come from various sources which include, but are not limited to:

- results of quality control samples and instrument calibrations;
- quality checks on consumables and materials;

- general observations of laboratory personnel;
- data review;
- proficiency testing;
- internal and external audits;
- complaints and feedback;
- management review and reports; and
- regulatory and certification and accreditation actions.

The way in which the laboratory handles nonconforming work depends on the significance and impact (risk) of the issue. Some issues may simply require correction, others may require investigation, corrective action (See 4.11) and/or data recall (See 4.16). When the laboratory releases data and test results associated with nonconforming QC and acceptance criteria test results are qualified or non-conformances are noted in the final analytical report to apprise the data user of the situation. (See 5.10)

Nonconforming work also includes unauthorized departure from laboratory policies, procedures and test methods. Authorized departures are explained in the following subsections. Situations that do not conform to these conditions are considered unauthorized departure(s).

4.9.1.1 Authorized Departure from SOP

An authorized departure from a test method SOP is one that has been reviewed and approved by the Department Manager, Technical Manager, Acting Technical Manager for TNI, Quality Manager, or the General Manager. Review is conducted to confirm the departure does not conflict with regulatory compliance requirements for which the data will be used or does not adversely affect data integrity. The departure may originate from client request or may be necessary to overcome a problem.

An authorized departure from administrative or process-oriented SOP is typically necessary to correct an error in the SOP. These departure requests are reviewed and pre-approved by the local QA Manager. Documentation of SOP departures and approval decisions are retained by the laboratory as evidence that the departure was authorized. When necessary, approved departures from test method SOPs are noted in the final test report to advise the data user of any ramification to data quality.

4.9.1.2 Authorized Departure from Test Methods (Method Modifications)

When test results are associated to a published reference test method, the laboratory's test method SOP must be consistent with the test method. If the test method is mandated for use by a specific regulatory program such as drinking water or wastewater or a certification or accreditation program, such as TNI/NELAC, the SOP must also comply with or include these requirements. If the procedures in the SOP are modified from the test method, these modifications must be clearly identified in the SOP. The conditions under which the laboratory may establish an SOP that is modified from these reference documents, and what is considered a

modification are specified in ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most current revision or replacement).

Modifications that do not meet the requirements of this SOP (ENV-SOP-CORQ-0011) are unauthorized. Client requests to deviate from the test method are handled as client requests to depart from the test method SOP since it is the SOP that the laboratory follows when performing work.

4.9.1.3 Stop Work Authority

Stop Work Authority provides laboratory personnel with the responsibility and obligation to stop work when there is a perceived unsafe condition or behavior that may result in an unwanted event.

All laboratory and corporate personnel have the authority to stop work when needed to preserve data integrity or safety of workers.

Once a stop work order has been initiated and the reason for doing so is confirmed valid; laboratory management is responsible for immediate correction and corrective action (see section 4.10) before resumption of work.

4.10 Continuous Improvement

The laboratory's quality management system is designed to achieve continuous improvement through the implementation of the quality policy and objectives outlined in this manual. Information about the laboratory's activities and performance is gained from many sources such as customer feedback, audits, QC, trend analysis, business analytics, management reports, proficiency testing, and management systems review. This information is subsequently used during the laboratory's corrective action (see section 4.11) and preventive action (see section 4.12) processes and to establish goals and objectives during annual review of the management system (see section 4.15).

PAS also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by Pace to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction.

4.11 Corrective Action

Corrective action is process used to eliminate the cause of a detected nonconformity. It is not the same as a correction. A correction is an action taken to fix an immediate problem. The goal of the corrective action process is to find the underlying cause(s) of the problem and to put in place fixes to prevent the problem from happening again. The corrective action process, referred to as CAPA by PAS, is one of the most effective tools used by the laboratory to prevent nonconforming work, identify risk and opportunity, and improve service to our customers.

The laboratory has two general processes for corrective action:

The process used for actions taken in response to day to day quality control (QC) and acceptance criteria exceptions (nonconformance) that occur during the day to day testing process are called corrections. These events do not usually include formal methods for cause analysis; instead the

reason for the failure is investigated through troubleshooting or other measures. Required actions for correction of routine nonconformance is specified in laboratory SOPs. When corrective action is not taken, cannot be taken, or is not successful, test results associated with the nonconforming work are qualified in the final test report. Documentation of the nonconformance and corrective action taken is documented in the analytical record.

A formal 7 step corrective action process is used when there is a problem or departure from the quality management system, technical activities, or when the extent of a single problem has significant impact on data, regulatory compliance or customer needs. These problems are identified through various activities such as but not limited to: quality control trends, internal and external audits, management review, customer feedback, and general observation.

The laboratory's 7 Step CAPA Process includes:

1) Define the Problem
2) Define the Scope of the Problem
3) Contain the Problem
4) Root Cause Analysis
5) Plan Corrective Action
6) Implement Corrective Action
7) Follow Up / Effectiveness Check

The formal CAPA process may be initiated by any employee. Once the process is initiated it is overseen and coordinated by laboratory management. The CAPA process is documented using an electronic or paper-based system. The CAPA record includes tracking information, dates, individuals involved, those responsible for action plan implementation and follow-up, and timelines and due dates.

For more information about the laboratory's procedure for corrective action, see laboratory SOP ENV-SOP-GBAY-0112, *Corrective and Preventative Action* (most current revision or replacement). Additional explanation about certain aspects of the laboratory's corrective action process are outlined in the next three subsections.

4.11.1 Root Cause Analysis

Root cause analysis (RCA) is the process of investigation used by the laboratory to identify the underlying cause(s) of the problem. Once causal factors are identified, ways to mitigate the causal factors are reviewed and corrective action(s) most likely to eliminate the problem are selected.

The laboratory uses different methods to conduct this analysis. The most common approach is 5-Why, but fishbone diagrams, or even brainstorming may be appropriate depending on the situation. The method used is documented in the CAPA record.

4.11.2 Effectiveness Review

Monitoring corrective actions for effectiveness is shared by laboratory supervisors and quality assurance personnel. Effectiveness means the actions taken were sustainable and appropriate. Sustainable means the change is still in place. Appropriate means the action(s) taken prevented recurrence of the problem since the time corrective action was taken.

The time-frame in which effectiveness review takes place depends on the event and is recorded in the CAPA record with any addition actions that need to be taken.

Corrective action trends are also monitored by laboratory management and used to identify opportunities for preventive action or to gain lessons learned when actions taken were not adequate to solve the problem. See Section 4.12 (Preventive Action) and 4.15 (Management Review) for more information.

4.11.3 Additional Audits

When non-conformances or other problems cast doubt on compliance with the laboratory's policies, procedures, or compliance to regulatory requirements; laboratory management schedules a special audit of the area of activity in accordance with Section 4.14.1 as soon as possible. These special audits are used to determine the scope of the problem and to provide information for the CAPA process. Additional full-scale audits are done when a serious issue or risk to the laboratory's business is identified.

4.12 Preventive Action

Preventive action is an action taken to eliminate the cause of a potential nonconformity and to achieve improvement. Preventive action is a forward thinking process designed to prevent problems opposed to reacting to them (corrective action).

Some examples of preventative action include, but are not limited to:

- Scheduled instrument maintenance (Preventative maintenance)
- Addition of Staff and Equipment
- Professional Development Activities
- Implementation of New Technology

The laboratory looks for opportunities for preventive action from a variety of sources including but not limited to: employee idea's, customer feedback, business partners input, trend analysis, business analytics, management reviews, proficiency testing results, lean management events, and risk-benefit analysis.

The process for preventive actions follows the same 7 step process for corrective action except "problem" is replaced with "opportunity", "cause analysis" is replaced with "benefit analysis", and "corrective action" is replaced with "preventive action".

Laboratory management evaluates the success of preventive actions taken in any given year during annual management review. See Section 4.15 for more information.

4.12.1 Change Management

Preventive actions may sometimes result in significant changes to processes and procedures used by the laboratory. Laboratory management evaluates the risks and benefits of change and includes in its implementation of change process, actions to minimize or eliminate any risk. The types of changes for which risk are considered and managed include: infrastructure change, change in analytical service offerings, certification or accreditation status, instrumentation, LIMS changes, and changes in key personnel.

For more information about the laboratory's procedures for preventive action see laboratory SOP ENV-SOP-GBAY-0112, *Corrective and Preventative Action* (most current revision or replacement).

4.13 Control of Records

A record is a piece of evidence about the past, especially an account of an act or occurrence kept in writing or some other permanent form. Laboratory records document laboratory activities and provide evidence of conformity to the requirements established in the quality management system. These records may be hardcopy or electronic on any form of media.

4.13.1 General Requirements

4.13.1.1 Procedure

The laboratory's procedures for control of records is provided in laboratory SOP ENV-SOP-GBAY-0015, *Document Management and Control* (most current revision or replacement).

The procedures in the SOP are established to assure quality and technical records are identified, retained, indexed, and filed to allow for retrieval during the entire retention time frame. During storage, records are kept secure and protected from deterioration. At the end of the retention time, the records are disposed of properly in order to maintain client confidentiality and to protect the interests of the company.

In general, laboratory records fall into three categories: quality, technical, and administrative.

Examples of each are provided in the following table:

Record Type	Includes Records of:
Quality	Documents: Document Types listed in SOP ENV-SOP-CORQ-016 Audits: Internal and External Certificates and Scopes of Accreditation Corrective & Preventive Action Management Review Data Investigations Method Validation Instrument Verification Training Records
Technical	Raw Data Logbooks Certificates of Traceability Analytical Record Test Reports & Project Information Technical Training Records & Demonstration of Capability
Administrative	Personnel Records Finance/Business

4.13.1.2 Record Legibility and Storage

Records are designed to be legible and to clearly identify the information recorded. Manual entries are made in indelible ink; automated entries are in a typeface and of sufficient resolution to be read. The records identify laboratory personnel that performed the activity or entered the information.

Records are archived and stored in a way that they are retrieved. Access to archived records is controlled and managed.

For records stored electronically, the capability to restore or retrieve the electronic record is maintained for the entire retention period. Hardcopy record are filed and stored in a suitable environment to protect from damage, deterioration, or loss. Hardcopy records may be scanned to PDF for retention. Scanned records must be checked against the hardcopy to verify the scan is complete and legible.

Records are kept for a minimum of 10 years unless otherwise specified by the client or regulatory program.

The date from which retention time is calculated depends on the record. In general, the retention time of technical records of original observation and measurement is calculated from the date the record is created. If the technical record is kept in a chronological logbook, the date of retention may be calculated from the date the logbook is archived. The retention time of test reports and project records, which are considered technical records, is calculated from the date the test report was issued. The retention time of quality records is usually calculated from the date the record is archived.

Refer to the laboratory's record management SOP for more information.

4.13.1.3 Security

The laboratory is a secure facility and access to records is restricted to laboratory personnel.

4.13.1.4 Electronic Records

The data systems used to store electronic records is backed up in accordance with laboratory SOP ENV-GBAY-0119 *Data and Records Archival* (most current revision or replacement). Access to archived records stored electronically is maintained by personnel responsible for management of the electronic system.

4.13.2 Technical Records

In addition to the requirements identified in subsections 4.13.1.1 through 4.13.1.4, the requirements in the following subsections also apply to technical records.

4.13.2.1 Description

Technical records are the accumulation of data and information generated from the analytical process. These records may include forms, worksheets, workbooks, checklists, notes, raw data, calibration records, final test reports, and project record. The accumulated record essentially need to provide sufficient detail to historically reconstruct the process and identify the personnel that performed the tasks associated with a test result.

4.13.2.2 Real Time Recordkeeping

Personnel are instructed and expected to always record observations, data, and calculations at the time they are made. Laboratory managers are responsible to assure that data entries, whether made electronically or on hardcopy, are identifiable to the task.

4.13.2.3 Error Correction

Errors in records must never erased, deleted or made illegible. Use of correction fluid, such as white-out is prohibited. In hardcopy records, the error is corrected by a single-strike through the original entry and the new entry recorded alongside or footnoted to allow for readability. Corrections are initialed and dated by the person making the correction. If the correction is not self-explanatory, a reason for the correction is recorded.

For electronic records, equivalent measures of error correction or traceability of changes made is kept. For example, audit trails provide records of change.

Maintenance of proper practices for error correction is monitored through the tiered data review process described in Section 5.9.3. Laboratory records are reviewed throughout the data review process. Individuals performing these reviews flag errors that are not properly corrected and bring these to the attention of the department manager or supervisor of the work area in which the record was generated so that the problem may be addressed and corrected with the individual(s) that did not make the correction properly.

4.14 Audits

The laboratory performs internal systems and technical audits to assess compliance to this manual and to other laboratory procedures, such as policy, SOP and SWI. Since the processed in this manual are based on the relevant quality system standards and regulatory and accreditation/certification program requirements the laboratory provides services for, the internal audits also assess on-going compliance to these programs.

The laboratory is also audited by external parties such as regulatory agencies, customers, consultants and non-government assessment bodies (NGAB).

Information from internal and external audits is used by laboratory management to address compliance concerns and opportunities where improvement will increase the reliability of data.

Deficiencies, observations and recommendations from audits are managed by local QA using the laboratory's formal CAPA process. See Section 4.11 for more information.

4.14.1 Internal Audit

The laboratory's internal audit program is managed by local QA in accordance with a pre-determined audit schedule established at the beginning of each calendar year. The schedule is prepared to assure that all areas of the laboratory are reviewed over the course of the year. Conformance to the schedule is reported to both laboratory management and corporate quality personnel in a monthly QA report prepared by the quality manager.

Although the QA Manager creates the audit schedule, it is the shared responsibility of local QA and laboratory managers to assure the schedule is maintained. Laboratory supervisors cooperate with QA to provide the auditors with complete access to the work area, personnel, and records needed.

Internal audits are performed by personnel approved by the quality manager. In general, personnel may not audit their own activities unless it can be demonstrated that an effective and objective audit will be carried out. 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 laboratory's internal audit program includes:

- System Audits & Method Audits: The purpose of these audits is to determine if daily practice is consistent with laboratory's SOPs and if SOPs are compliant with adjunct policy and procedures. Auditing techniques includes analyst interviews and observation and records review. These audits are performed per the pre-determined schedule.
- Raw Data / Final Test Report Audits: The purpose of these audits is to review raw data and/or a final test reports to verify the final product is consistent with customer/project requirements and supported as compliant to SOPs, reference methods, with test results that are properly qualified when necessary, accurate, and of known and documented quality. The reviews should also identify opportunities for improvement and best practices.
- Special Audits: Special audits are those performed ad hoc to follow up on specific a specific issue such as a client complaint, negative feedback, concerns of data integrity or ethics, or a problem identified through other audits. Special audits may be scheduled or unscheduled. Unscheduled internal audits are conducted whenever doubts are cast on the laboratory's compliance with regulatory requirements or its own policies and procedures. These unscheduled internal audits may be conducted at any time and may be performed without an announcement to laboratory personnel.

When observations and findings from any audit (internal or external) cast doubt on the validity of the laboratory's testing results, the laboratory takes immediate action to initiate investigate the problem and take corrective action. (Also see 4.11 and 4.16)

The laboratory's internal audit program and auditing procedures are further described in laboratory SOP ENV-SOP-GBAY-0108, *Internal and External Audits* (most current revision or replacement).

4.14.1.1 Corporate Compliance Audit

The laboratory may also be audited by corporate quality personnel to assess the laboratory's compliance to the company's quality management program and to evaluate the effectiveness of implementation of the policies and procedures that make up the quality management system. The purpose of the compliance audit is to identify risks and opportunities and to assist laboratory management achieve the goals and objectives of the company's quality program.

4.15 Management Review

The laboratory's management team formally reviews the management system on an annual basis to assess for on-going suitability and effectiveness and to establish goals, objectives, and action plans for the upcoming year.

At a minimum, following topics are reviewed and discussed:

- The on-going suitability of policies and procedures including HSE (Health, Safety and Environment) and waste management;

- Reports from managerial and supervisory personnel including topics discussed at regular management meetings held throughout the year;
- The outcome of recent internal audits;
- Corrective and preventive actions;
- Assessments by external bodies;
- The results of interlaboratory comparisons or proficiency tests;
- Changes in the volume and type of the work;
- Customer and personnel feedback, including complaints;
- Effectiveness of improvements / preventive actions made since last review;
- Internal and external issues of relevance and risk identification;
- A review of the status of actions from prior management reviews; and
- Other relevant factors, such as quality control activities, resources, and staff training.

The discussion and results of this review are documented in a formal report prepared by laboratory management. This report includes a determination of the effectiveness of the management system and its processes; goals and objectives for improvements in the coming year with timelines and responsibilities, any other need for change. See laboratory SOP ENV-SOP-CORQ-0005, *Review of Laboratory Management System* (most current revision or replacement) for more information.

Goals and action items from annual management systems review are shared with employees to highlight focus areas for improvement in addition to areas in which the laboratory has excelled.

4.16 Data Integrity

The laboratory's procedures for data integrity reviews are described in SOP ENV-SOP-CORQ-0010 *Data Recall* (most current revision or replacement).

Customers whose data are affected by these events are notified in a timely manner, usually within 30 days of discovery. Some accreditation programs also require notification to the accreditation body (AB) within a certain time-frame from date of discovery when the underlying cause of the issue impacts accreditation. The laboratory follows any program or project specific client notification requirements for notification, when applicable.

5.0 TECHNICAL REQUIREMENTS

5.1 General

Many factors contribute to the correctness and reliability of the technical work performed by the laboratory. These factors are fall under these general categories:

- Human Performance
- Facility and Environmental Conditions
- Test Method Performance and Validation

- Measurement Traceability
- Handling of Samples

The impact of each of these factors varies based on the type of work performed. To minimize negative effects from each these factors, the laboratory takes into account the contribution from each of these categories when developing test method and process (administrative) SOPs, evaluating personnel qualifications and competence, and in the selection of equipment and supplies used.

5.2 Personnel

5.2.1 Personnel Qualifications

The laboratory's program for personnel management is structured to ensure personnel are selected, qualified, and competent to perform the roles and responsibilities of their position based on education, experience, and training.

Qualifications, duties, responsibilities, and authorities of each position are specified in job descriptions maintained by corporate HR (See Section 5.2.4). These job descriptions provide the general basis for the selection of personnel for hire and are used by the laboratory to communicate to personnel the duties, responsibilities, and authorities of their position.

The term "personnel" refers to individuals employed by the laboratory directly as full-time, part-time, or temporary, and individuals employed by the laboratory by contract, such as through an employment agency. The term "personnel" is used interchangeably with the term "employee" throughout this manual. For purposes of this manual, these terms are equivalent.

The personnel management program is structured to establish and maintain records for each of the following:

- Selection of personnel;
- Training of personnel;
- Supervision of personnel;
- Authorization of personnel; and
- Monitoring Competence of personnel.

5.2.1.1 Competence

Competence is the ability to apply a skill or series of skills to complete a task or series of tasks correctly within defined expectations.

Competence for technical personnel authorized by PAS to provide opinion and interpretation of data to customers also includes the demonstrated ability to:

- Apply knowledge, experience, and skills needed to safely and properly use equipment, instrumentation, and materials required to carry out testing and other work activities in accordance with manufacturer specifications and laboratory SOPs;
- Understand and apply knowledge of general regulatory requirements necessary to achieve regulatory compliance in work product; and

- Understand the significance of departures and deviations from procedure that may occur during the analytical testing process and the capability and initiative to troubleshoot and correct the problem, document the situation and decision making process, and to properly qualify the data and analytical results.

The laboratory's requirements for the competence of personnel (education, qualification, work experience, technical skills, and responsibilities) are specified in job descriptions created by management and kept by human resources (HR). The job description provides the basis for the selection of personnel for each position.

An employee is considered competent when he/she has completed required training.

The policies and standard operating procedures (SOPs) for the following topics are established by management as minimum required training for all personnel:

- Ethics and Data Integrity
- Quality Manual
- Safety Manual
- Quality Management System
- Technical Process and Procedure relevant to their job tasks
- Successful Demonstration of Capability (DOC) – Analytical Personnel Only

Personnel are initially authorized competent to independently carry out their assigned duties when required training is complete and documented.

Records of training and qualification provide the record of competence for the individual. Qualification records may include but are not limited to diploma, transcripts, and curriculum vitae (CV).

The on-going competence of each employee is monitored by laboratory management through on-the-job performance. Analytical employees are also required to successfully complete another demonstration capability for each test method performed on an annual basis.

5.2.2 Training

Training requirements are outlined in policies COR-POL-0023 *Mandatory Training Policy*, COR-POL-0004 *Ethics Policy*, and laboratory SOP ENV-SOP-GBAY-0094 *Employee Orientation and Training* (most current revision or replacement). Additional training requirements may also be specified in other documents, such as manuals

5.2.2.1 Training Program and Goals

The laboratory's training program includes 4 elements:

- Identification of Training Needs
- Training Plan Development and Execution

- Documentation and Tracking
- Evaluation of Training Effectiveness

Laboratory management establishes goals and training needs for individual employees based on their role, education, experience, and on-the-job performance.

Training needs for all employees are based on business performance measures that include but are not limited to:

- Quality Control Trends
- Process Error / Rework Trends
- Proficiency Testing Results
- Internal & External Audit Performance
- Management Review Goals

Training is delivered using various methods that incorporate techniques that appeal to the main learning styles: visual, aural, linguistic, and kinesthetic. Techniques include, on-the-job, instructor-led, self-study, eLearning, and blended.

The employee's direct supervisor is responsible for oversight of the employee's training plan and for providing adequate time to the employee to complete training assignments. Both the supervisor and employee are responsible to make sure the employee's training status and training records are current and complete.

The laboratory's QA department monitors the training status of personnel and provides the status to the General Manager (GM or AGM) at least monthly or more frequently, if necessary. The status report is used by laboratory management to identify overdue training assignments, the reasons for the gaps, and to make arrangements for completion.

The following subsections highlight specific training requirements:

5.2.2.1.1 New Hire Training

New hire training requirements apply to new personnel and to existing employee's starting in a new position or different work area.

Required new hire training includes each of the following:

- Ethics and Data Integrity (See 5.2.2.1.3)
- Quality Manual / Quality Management System (See 5.2.2.1.4)
- Safety Manual and any training requirements specified in the manual.
- Policies & SOPs relevant to their job tasks
- Technical personnel that test samples must also successfully complete an initial demonstration of capability (IDOC) for the

test methods performed before independently testing customer samples. (See 5.2.2.1.5). Independent testing means handling of client samples without direct supervision of the work activity by the supervisor or a qualified trainer.

All required training must be current and complete before the employee is authorized to work independently. Until then, the employee's direct supervisor is responsible for review and acceptance of the employee's work product.

5.2.2.1.2 On-Going Training

Personnel receive on-going training in each of the following topics:

- Ethics and Data Integrity (See 5.2.2.1.3)
- Quality Manual / Quality Management System (See 5.2.2.1.4)
- Safety Training
- Changes to Policies & SOPs
- Specialized Training
- Technical employees that carry of testing must also successfully complete on-going demonstration of capability (ODOC) for all test methods performed on an annual basis. (See 5.2.2.1.5)

Personnel are expected to maintain their training status and records of training current and complete and to complete training assignments in a timely manner.

5.2.2.1.3 Ethics and Data Integrity Training

Data integrity training is provided to all new personnel and refresher data integrity training is provided to all employees on an annual basis. Personnel are required to acknowledge they understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment, or civil/criminal prosecution.

The initial data integrity training and the annual refresher training is documented with a signature attendance sheet or other form of documentation to provide evidence that the employee has participated in training on this topic and understand their obligations related to data integrity.

The following topics and activities are covered:

- Policy for honesty and full disclosure in all analytical reporting;
- Prohibited Practices;
- How and when to report data integrity issues;

- Record keeping. The training emphasizes the importance of proper written documentation on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially nonconforming;
- Training Program, including discussion regarding all data integrity procedures;
- Data integrity training documentation;
- In-depth procedures for data monitoring; and
- Specific examples of breaches of ethical behavior such as improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards.

All PAS personnel, including contract and temporary, are required to sign an "Attestation of Ethics and Confidentiality" at the time of employment and during annual refresher training. This document clearly identifies inappropriate and questionable behavior. Violations of this document result in serious consequences, including prosecution and termination, if necessary.

Also see SOP-ENV-COR-POL-0004 *Ethics Policy* for more information.

5.2.2.1.4 Management System Documents Training

PAS Manuals, policies, and SOPs are the primary documents used by regulatory bodies and PAS customers to verify the laboratory's capability, competency, and compliance with their requirements and expectations.

In addition to on-the-job training, employees must have a signed Read and Acknowledgement Statement on record for the laboratory quality manual, and the policies and SOPs relating to his/her job responsibilities. This statement when signed by the employee electronically or by wet signature, confirms that the employee has received, read, and understands the content of the document, that the employee agrees to follow the document when carrying out their work tasks; and the employee understands that unauthorized change to procedures in an SOP is not allowed except in accordance with the SOP departure policy (See 4.9.9.1) and SOP ENV-CORQ-0016 *Standard Operating Procedures and Standard Work Instructions* for more information.

5.2.2.1.5 Demonstration of Capability (DOC)

Technical employees must also complete an initial demonstration of capability (IDOC) prior to independent work on client samples analyzed by the test methods they perform. After successful IDOC, the employee must demonstrate continued proficiency (CDOC) for

the test method on an annual basis. If more than a year has passed since the employee last performed the method; then capability must be re-established with an IDOC.

Demonstration of capability (IDOC and DOC) is based on the employee's capability to achieve acceptable precision and accuracy for each analyte reported by the laboratory for the test method using the laboratory's test method SOP.

Records of IDOC and ODOC are kept in the employee's training file.

For more information, see laboratory SOP ENV-SOP-GBAY-0094, *Employee Orientation and Training* (most current revision or replacement).

5.2.2.2 Effectiveness of Training

The results of the performance measures used to identify training needs are the same measures used by the laboratory to measure effectiveness of the training program. Improvement in key performance measures suggest the training program is successful. (See 5.2.2.1)

Effectiveness of individual employee training is measured by their demonstrated ability to comprehend the training material and apply knowledge and skills gained to their job task. Measurements include but are not limited to:

- Testing of the employee's knowledge of the quality management system, policies, and technical and administrative procedures through various mechanisms, such as quizzes, observation, and interviews.
- Demonstrated ability to convey information correctly and factually in written and verbal communication to internal and external parties.
- Demonstrated ability to carry out tasks in accordance with SOPs and other work instructions.
- Demonstrated ability to make sound decisions based on guidance and information available.
- Demonstrated initiative to seek help or guidance when the employee is unsure of how to proceed.

5.2.3 Personnel Supervision

Every employee is assigned a direct supervisor, however named, who is responsible for their supervision. Supervision is the set of activities carried out by the supervisor to oversee the progress and productivity of the employees that report to them.

General supervisory responsibilities may include but are not limited to:

- Hiring Employees
- Training Employees

- Performance Management
- Development, oversight, and execution of personnel training plans
- Monitoring personnel work product to assure the work is carried out in accordance with this quality manual, policies, SOPs, and other documents that support the quality management system.

5.2.4 Job Descriptions

Job Descriptions that define the required education, qualifications, experience, skills, roles and responsibilities, and reporting relationships for each PAS position are established by top management and kept by corporate HR. PAS laboratories use these job descriptions as the source of positions and job titles for the laboratory. The job descriptions apply to employees who are directly employed by PAS, part-time, temporary, technical and administrative and by those that are under contract with PAS through other means.

The job descriptions include the education, expertise, and experience required for the position and the responsibilities and duties, including any supervisory or managerial duties assigned to the position.

5.2.5 Authorization of Technical Personnel

Laboratory management authorizes technical personnel to perform the technical aspects of their position after it has been verified that the employee meets the qualifications for the position, has successfully completed required training, and the employee has demonstrated capability. After initial authorization, technical personnel are expected to maintain a current and complete training record, demonstrate on-going capability at least annually for each test method performed, and produce reliable results through accurate analysis of certified reference materials, proficiency testing samples, and/or routine quality control samples in order to remain authorized to continue to perform their duties.

Records to support authorization including, education, experience, training, and other evaluations are kept by the laboratory.

5.3 Accommodations and Facilities

5.3.1 Facilities

The laboratory is designed to support the correct performance of procedures and to not adversely affect measurement integrity or safety. Access to the laboratory is controlled by various measures, such as card access, locked doors, main entry. Visitors to the laboratory are required to sign-in and to be escorted by laboratory personnel during their visit. A visitor is any person that is not an employee of the laboratory.

5.3.2 Environmental Conditions

The laboratory is equipped with energy sources, lighting, heating, and ventilation necessary to facilitate proper performance of calibrations and tests. The laboratory ensures that housekeeping, electromagnetic interference, humidity, line voltage, temperature, sound and vibration levels are appropriately controlled to ensure the integrity of specific measurement results and to prevent adverse effects on accuracy or increases in the uncertainty of each measurement.

Environmental conditions are monitored, controlled, and recorded as required by the relevant specifications, methods, and procedures. Laboratory operations are stopped if it is discovered that the laboratory's environmental conditions jeopardize the analytical results.

5.3.3 Separation of Incompatible Activities

The layout and infrastructure of each work area including air handling systems, power supplies, and gas supplies of each laboratory work area is specifically designed for the type of analytical activity performed. Effective separation between incompatible work activities is maintained. For example, sample storage, preparation, and chemical handling for volatile organic analysis (VOA) is kept separate from semi-volatile organic (SVOA).

The laboratory separates samples known or suspected to contain high concentration of analytes from other samples to avoid the possibility for cross-contamination. If contamination is found, the source of contamination is investigated and resolved in accordance with laboratory SOPs.

5.3.4 Laboratory Security

Security is maintained by controlled access to the building and by surveillance of work areas by authorized personnel. Access is controlled to each area depending on the required personnel, the sensitivity of the operations performed, and possible safety concerns. The main entrance is kept unlocked during normal business hours for visitors, and is continuously monitored by laboratory staff. All visitors must sign a visitor's log, and a staff member must accompany them during the duration of their stay.

5.3.5 Good Housekeeping

The laboratory ensures good housekeeping practices in work areas to maintain a standard of cleanliness necessary for analytical integrity and personnel health and safety. Minimally, these measure include regular cleaning of the work area. Where necessary, areas are periodically monitored to detect and resolve specific contamination and/or possible safety issues.

5.4 Test Methods

5.4.1 General Requirements

The laboratory uses test methods and procedures that are appropriate for the scope of analytical services the laboratory offers.

Instructions on the use and operation of equipment and sample handling, preparation, and analysis of samples are provided in SOPs. The instructions in SOPs may be supplemented with other documents including but not limited to, standard work instructions (SWI), manuals, guides, project documents and reference documents.

These documents are managed using the procedures described in SOP ENV-SOP-CORQ-0015 *Document Management and Control* (most current revision or replacement) and SOP ENV-SOP-CORQ-0016 *Standard Operating Procedures and Standard Work Instructions* (most current revision or replacement).

Deviations to test method and SOPs are allowed under certain circumstances. See sections 4.9.1.1 and 4.9.1.2 for more information.

5.4.2 Method Selection

The test methods and protocols used by the laboratory are selected to meet the needs of the customer, are appropriate for the item tested and intended use of the data, and to conform with regulatory requirements when regulatory requirements apply.

In general, the test methods offered are industry accepted methods published by international, regional, or national standards. The laboratory bases its procedure on the latest approved edition of a method unless it is not appropriate or possible to do so or unless regulatory requirements specify otherwise.

The laboratory confirms that it can perform the test method and achieve desired outcome before analyzing samples (see section 5.4.5). If there is a change in the published analytical method, then the confirmation is repeated.

When a customer does not specify the test method(s) to be used, the laboratory may suggest test methods that are appropriate for the intended use of the data and the type of samples to be tested. The laboratory will also inform customers when test methods requested are considered inappropriate for their purpose and/or out of date. This discourse takes place during review of analytical service requests (See Section 4.4).

5.4.3 Laboratory Developed Methods

A laboratory developed method is a method developed from scratch (no published source method), a procedure that modifies the chemistry from the source method, or a procedure that exceeds the scope and application of the source method.

Laboratory developed methods must be validated prior to use (see section 5.4.5) and the procedure documented in a test method SOP.

The requirements for non-standard methods (Section 5.4.4) also apply to laboratory developed methods.

5.4.4 Non-standard Methods

A non-standard method is a method that is not published or approved for use by conventional industry standards for the intended purpose of the data. Non-standard methods must be validated prior to use (see section 5.4.5) and the procedure developed and documented in a test method SOP.

At a minimum, the following information must be included in the procedure:

- Title / Identification of Method;
- Scope and Application;
- Description of the type of item to be analyzed;
- Parameters or quantities and ranges to be determined;
- Apparatus and equipment, including technical performance requirements;
- Reference standards and reference materials required;
- Environmental conditions required and any stabilization period needed
- Description of the procedure, including:

- Affixing identification marks, handling, transporting, storing and preparing of items;
- Checks to be made before the work is started;
- Verifying equipment function and, where required, calibrating and/or adjusting the equipment before each use;
- Method of recording the observations and results;
- Any safety measures to be observed;
- Criteria and/or requirements for approval/rejection;
- Data to be recorded and method of analysis and presentation; and
- Uncertainty or procedure for estimating uncertainty.

Use of a non-standard method for testing must be agreed upon with the customer. The agreement, which is retained by the laboratory in the project record, must include the specifications of the client's requirements, the purpose of testing, and their authorization for use of the non-standard method.

5.4.5 Method Validation

5.4.5.1 Validation Description

Validation is the process of conformation and the provision of objective evidence that the stated requirements for a specific method/procedure are fulfilled.

The laboratory's requirements and procedures for method validation are outlined in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most current revision or replacement).

5.4.5.2 Validation Summary

All test methods offered by the laboratory are validated before use to confirm the procedure works and the data and results achieved meet the goals for the method. The extent of validation performed is based on technology and other factors as defined in the validation SOP (ENV-SOP-CORQ-0011).

Results of validation are retained are kept in accordance with the laboratory's SOP ENV-SOP-GBAY-0119, *Data and Records Archival*, for retention of technical records (most current revision or replacement).

The need to repeat validation is assessed by laboratory management when there are changes to the test method.

5.4.5.3 Validation of Customer Need

Laboratory management reviews the results of test method validation, which include accuracy, precision, sensitivity, selectivity, linearity, repeatability, reproducibility, robustness, and cross-sensitivity, against general customer needs to ensure the laboratory's procedure for the test method will meet those needs.

The review procedure is detailed in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most current revision or replacement).

The following subsections highlight some of these concepts:

5.4.5.3.1 Accuracy

Accuracy is the degree to which the result of a measurement, calculation, or specification conforms to the correct value or a standard. When the result recovers within a range from the known value (control limit); the result generated using the laboratory's test method SOP is considered accurate.

5.4.5.3.2 Precision

Precision refers to the closeness of two or more measurements to each other. It is generally measured by calculating the relative percent difference (RPD) or relative standard deviation (RSD) from results of separate analysis of the same sample. Precision provides information about repeatability, reproducibility, and robustness of the laboratory's procedure.

5.4.5.3.3 Limits of Detection (LOD) (Chemistry)

The LOD is the minimum result which can be reliably discriminated from a blank with a predetermined confidence level. The LOD establishes the limit of method sensitivity and is also known as the detection limit (DL) or the method detection limit (MDL).

Values below the LOD cannot be reliably measured and are not reported by the laboratory unless otherwise specified by regulatory program or test method.

The LOD is established during method validation and after major changes to the analytical system or procedure that affect sensitivity are made.

The laboratory's procedure for LOD determination is detailed in laboratory SOP ENV-SOP-GBAY-0106, *Determination of the LOD and LOQ* (most current revision or replacement). The SOP complies with 40 CFR 136 Appendix B or the current industry approved and accepted guidance for this process.

5.4.5.3.4 Limits of Quantitation (LOQ) and Reporting Limit (RL)

The LOQ is the minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence. The LOQ is established at the same time as the LOD. The laboratory's procedure for determination and verification of the LOQ is detailed in laboratory ENV-SOP-GBAY-0106, *Determination of the LOD and LOQ* (most current revision or replacement).

The LLOQ is the value of the lowest calibration standard. The LOQ establishes the lower limit of quantitation.

The LOQ and LLOQ represent quantitative sensitivity of the test method.

- The LOQ must always be equal to or greater than the LLOQ and the LLOQ must always be greater than the LOD.
- Any reported value (detect or non-detect) less than the LLOQ is a qualitative value.

The RL is the value to which the presence of a target analyte is reported as detected or not-detected. The RL is project-defined based on project data quality objectives (DQO). In the absence of project specific requirements, the RL is usually set to the LOQ or the LLOQ. Depending on the relationship of the RL to the LLOQ or LOQ, both the RL value may be or quantitative.

For more information, refer to laboratory SOP ENV-SOP-GBAY-0106, *Determination of the LOD and LOQ* (most current revision or replacement).

5.4.5.3.5 Linearity

Linearity is a mathematical concept applied to calibration models that employ multiple points to establish a calibration range used for quantitative analysis. Linearity is measured differently based on the calibration model. In general, if linearity is demonstrated than the slope of the response of standards are sufficiently close to one another. The accuracy of the linear regression and non-linear curves is verified by checking percent error or relative standard error (RSE), which is the process of refitting calibration data back to the model to determine if the results are accurate. For linear curves that use average calibration or response factor, error is measured by relative standard difference (RSD).

Linearity also establishes the range of quantitation for the test method used which directly impacts the sensitivity of the test method and uncertainty in measurement results. As previously noted, the LLOQ establishes the lower limit of quantitation. Similarly, the upper range of linearity establishes the upper limit of quantitation. In general, results outside of this range are considered qualitative values. However, some inorganic methods allow for extension of the linear range above the upper limit of quantitation when accuracy at this value is verified.

Linearity can also be used to establish repeatability, reproducibility, and robustness of the laboratory's test method. When linearity is demonstrated using a specific calibration model during method validation, then use of this same calibration model to achieve linearity on a day to day basis confirms the laboratory's method is repeatable, reproducible, and robust.

5.4.5.3.6 Demonstration of Capability (DOC)

The DOC performed during method validation confirms that the test method acceptable precision and accuracy. The procedure used for DOC for method validation is the same as described in section 5.2.2.1.5 for demonstration of analyst capability.

5.4.6 Measurement Uncertainty

The laboratory provides an estimate of uncertainty in testing measurements when required or on client request. In general, the uncertainty of the test method is reflected in the control limits used to evaluate QC performance. (See 5.9.1.1.10). ISO/IEC supports this concept with language that reads when a well-recognized test method specifies limits to the values of the major source of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory has satisfied the requirements on analytical uncertainty by following the test method and reporting instructions.

When measurement uncertainty cannot be satisfied through control limits, the laboratory will provide a reasonable estimation of uncertainty. A reasonable estimation is based on knowledge of method performance and previous experience. When estimating the analytical uncertainty, all uncertainty components which are of importance in the given situation are taken into account.

5.4.7 Control of Data

The laboratory has policies and processes in place to assure that reported data is free from calculation and transcription errors, that quality control is reviewed and evaluated before data is reported, and to address manual calculation and integration.

5.4.7.1 Calculations, Data Transfer, Reduction and Review

Whenever possible, calculations, transfer of data, and data reduction are performed using validated software programs. (See 5.4.7.2)

If manual calculations are necessary, the results of these calculations are verified during the data review process outlined in section 5.9.3.

5.4.7.1.1 Manual Integration

The laboratory's policy and procedures for manual integration are provided in SOP ENV-SOP-CORQ-0006 *Manual Integration* (most current revision or replacement).

This SOP includes the conditions under which manual integration is allowed and the requirements for documentation.

Required documentation of manual integration includes:

- complete audit trail to permit reconstruction of before and after results;
- identification of the analyst that performed the integration and the reason the integration was performed; and

- the individual(s) that reviewed the integration and verified the integration was done and documented in compliance with the SOP.

5.4.7.2 Use of Computers and Automated Acquisition

Whenever possible the laboratory uses software and automation for the acquisition, processing, recording, reporting, storage, and/or retrieval of data.

Software applications developed by PAS are validated by corporate IT for adequacy before release for general use. Commercial off the shelf software is considered sufficiently validated when the laboratory follows the manufacturer or vendor's manual for set-up and use. Records of validation are kept by the corporate information technology (IT) group or by the local laboratory, whichever group performed the validation.

The laboratory's process for the protection of data stored in electronic systems include:

- Individual user names and passwords for Laboratory Information Management Systems (LIMS) and auxiliary systems used to store or process data.
- Employee Training in Computer Security Awareness
- Validation of spreadsheets used for calculations to verify formulas and logic yield correct results and protection of these cells to prevent unauthorized change.
- Operating system and file access safeguards
- Protection from Computer Viruses
- Regular system backup; and testing of retrieved data

The laboratory's process for software development and testing process includes:

- Verification the software application works as expected and is adequate for use and fulfills compliance requirements, such as the need to record date/time of data generation.
- Change control to assure requests for changes are reviewed and approved by management before the change is made.
- Communication channels to assure all staff are aware of changes made.
- Version Control and maintenance of historical records.

These procedures are detailed in laboratory SOPs ENV-SOP-GBAY-0105, *Software Validation* (most current revision or replacement).

5.5 Equipment

5.5.1 Availability of Equipment

The laboratory is furnished with all equipment and instrumentation necessary to correctly perform the tests offered in compliance with the specifications of the test method and to achieve the accuracy and sensitivity required.

5.5.2 Calibration

Equipment and instrumentation is checked prior to use to verify it performs within tolerance for its intended application.

Laboratory management is made aware of the status of equipment and instrumentation and any needs for either on a daily basis. This information is obtained during laboratory walkthroughs (LDM) that are conducted as part of the laboratory's lean program.

5.5.2.1 Support Equipment

The laboratory confirms support equipment is in proper working order and meets the specifications for general laboratory use prior to placement in service and with intermediate checks thereafter. Equipment that does not meet specifications is removed from service until repaired or replaced. Records of repair and maintenance activities are maintained.

Procedures used to carry out and record these checks are outlined laboratory SOP ENV-SOP-GBAY-0115, *Support Equipment* (most current revision or replacement).

5.5.2.2 Analytical Instruments

Analytical instruments are checked prior to placement in service in accordance with SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.* After the initial service date, the calibration of instruments and verification calibration is performed in accordance with local test method SOPs.

The calibration procedures in the test method SOPs comply with the requirements for acceptable calibration practices outlined in corporate document ENV-SOT-CORQ-0026 *Acceptable Calibration Practices*, the reference methods, and any applicable regulatory or program requirements.

5.5.3 Equipment Use and Operation

Equipment is operated and maintained by laboratory personnel that are trained on the test method SOP. Up-to-date instructions and procedures for the use and maintenance of analytical equipment are included in SOPs and/or supplemental documents such as standard work instructions (SWI) or instrument manuals which are made readily accessible in the work area to all laboratory personnel.

5.5.4 Equipment Identification

The laboratory uniquely identifies equipment by serial number or any other unique ID system, when practical. The identifier is included in the equipment list maintained by QA.

5.5.5 Equipment Lists and Records

5.5.5.1 Equipment List

The laboratory maintains a master list of equipment that includes information about the equipment including a description, manufacturer, serial number, date placed in service, condition when received, identity, and the current location in the laboratory. The date of purchase is tracked by the procurement record. The equipment list(s) for each location covered by this manual is provided in Appendix F.

5.5.5.2 Equipment Records

In addition to the equipment list, the laboratory maintains records of equipment that include:

- Verification that equipment conforms with specifications.
- Calibration records including dates, results, acceptance criteria, and next calibration dates.
- Maintenance plan and records
- Records of damage, malfunction, or repair

The laboratory follows an equipment maintenance program designed to optimize performance and to prevent instrument failure which is described in laboratory SOP ENV-SOP-GBAY-0098, *Preventative, Routine, and Non-Routine Maintenance* (most current revision or replacement) or individual test method SOPs.

The maintenance program includes routine maintenance activities which are performed as recommended by the manufacturer at the frequency recommended and non-routine maintenance, which is performed to resolve a specific problem such as degradation of peak resolution, shift in calibration relationship, loss of sensitivity, or repeat failure of instrument performance checks and quality control samples.

Maintenance is performed by laboratory personnel or by outside service providers.

All maintenance activities performed by laboratory personnel are recorded by the individual(s) that performed the activity at the time the maintenance was performed in an instrument maintenance log.

The maintenance record minimally includes the date of maintenance, the initials of the person(s) performing maintenance, a description of the activity performed, why (when the maintenance is non-routine), and the return to analytical control. When maintenance is performed by an external vendor, the laboratory staples the service record into hardcopy maintenance logs or scans the record easy retrieval. The laboratory provides unrestricted access to instrument maintenance logs in order to promotes good instrument maintenance and recordkeeping practices.

If an instrument must be moved, the laboratory will use safe practices for handling and transport to minimize damage and contamination.

5.5.6 Out of Service Protocol

Equipment that has been subjected to overloading, mishandling, gives suspect results, has been shown to be defective, or is performing outside of specified limits is taken out of service and either removed from the work area or labeled to prevent accidental use until it has been repaired and verified to perform correctly.

When analytical equipment is taken out of service, the laboratory examines the potential effect it may have had on previous analytical results to identify any non-conforming work. (See section 4.9).

5.5.7 Calibration Status

The laboratory labels support equipment to indicate calibration status, whenever practicable or otherwise maintains the calibration status in a visible location in the work area. These procedures are described in laboratory SOP ENV-SOP-GBAY-0115, *Support Equipment* (most current revision or replacement).

The calibration status of analytical instruments is documented in the analytical record. Analysts verify on-going acceptability of calibration status prior to use and with instrument performance check standards. These procedures are described in test method SOPs.

5.5.8 Returned Equipment Checks

When equipment or instrument is sent out of the laboratory for service, the laboratory ensures that the function and calibration status of the equipment is checked and shown to be satisfactory before the equipment is returned to service. These procedures are outlined in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most current revision or replacement).

5.5.9 Intermediate Equipment Checks

The laboratory performs intermediate checks on equipment to verify the on-going calibration status. For example, most test method require some form of continuing calibration verification check and these procedures are included in the test method SOP. Periodic checks of support equipment are also performed; see appendix E for more information.

5.5.10 Safeguarding Equipment Integrity

The laboratory safeguards equipment integrity using a variety of mechanisms that include but are not limited to:

- Adherence to manufacture's specification for instrument use so that settings do not exceed manufacturer's recommendation or stress the performance of the equipment.
- Established maintenance programs.
- Transparent maintenance records and unrestricted access to maintenance logs.
- Validation and approval of software before use.
- Audits to confirm instrument settings are consistent with SOPs.
- On-the-job training for safe and proper use of laboratory equipment.

5.6 Measurement Traceability

5.6.1 General

Measurement traceability refers to a property of a measurement result whereby the result can be related to a reference through an unbroken chain of calibration, each contributing to the measurement uncertainty. Traceability requires an established calibration hierarchy of equipment (instruments) used during testing including equipment used for subsidiary measurements. The laboratory assures this equipment is calibrated prior to being put into service and that the reference standard and materials used for calibration are traceable to the international standard of units (SI) or national measurement standard.

When strict traceability to SI units cannot be made, the laboratory establishes traceability with the use of reference standards and equipment obtained from competent supplier that provide calibration certificates and/or certificates of analysis (COA).

5.6.2 Equipment Correction Factors

When correction factors are used to adjust results the laboratory will assure that results in computer software are also updated. For example, if the direct instrument or reading output must be corrected based on preparation factor or concentration factors, laboratory management will assure the corrected result is also updated in the software, whenever possible.

5.6.3 Specific Requirements

5.6.3.1 Requirements for Calibration Laboratories

The laboratory does not offer calibration services to customers.

5.6.3.2 Requirements for Testing Laboratories

The laboratory has procedures in place to verify equipment is calibrated prior to being put into service. (See 5.5.2) and ensures the reference standard and materials used for calibration are traceable to the international standard of units (SI) or national measurement standard. When strict traceability to SI units cannot be made, the laboratory establishes traceability with the use of reference standards and equipment obtained from competent suppliers that provide calibration certificates and/or certificates of analysis (COA).

5.6.4 Reference Standards and Reference Materials

5.6.4.1 Reference Standards

The laboratory uses reference standards of measurement to verify adequacy of working weights and thermometers. The working weight is the weight(s) used for daily balance calibration checks and the working thermometers are used for temperature measurements on a daily basis.

Intermediate checks of the working reference measurement standards are performed to verify adequacy between calibration from an external calibration laboratory. The measurements from working weights and thermometers are compared to measurement taken by the reference standard which is traceable to SI or a national standard. The reference weights and thermometers are used solely for verification

purposes unless the laboratory can prove that daily use does not adversely affect performance of the reference standard.

The laboratory performs intermediate checks of the working weights at least annually.

Working thermometers (glass and digital) are checked against the reference thermometer prior to placement in service to establish a correction factor and then rechecked annually (glass) or quarterly (digital) thereafter.

The calibration of liquid in glass reference thermometers is verified every 5 years and the calibration of digital reference thermometers is verified annually by an ISO/IEC 17025 accredited calibration laboratory or service provider that provides traceability to a national standard.

The calibration of the reference weight(s) is verified every 5 years by an ISO/IEC 17025 accredited calibration laboratory.

If criteria for the intermediate checks or recertification is not acceptable, the impact on previously reported results is evaluated using the process for evaluation of nonconforming work (See 4.9)

See laboratory ENV-SOP-GBAY-0115, *Support Equipment* (most current revision or replacement) for more information about this process.

5.6.4.2 Reference Materials

The laboratory purchases chemical reference materials used (also known as stock standards) from vendors that are accredited to ISO 17034 or Guide 34. Purchased reference materials must be received with a Certificate of Analysis (COA) where available. If a reference material cannot be purchased with a COA, it must be verified by analysis and comparison to a certified reference material and/or there must be a demonstration of capability for characterization. COA are reviewed for adequacy and retained by the laboratory for future reference.

The laboratory procedure for traceability and use of these materials is provided in laboratory SOP ENV-SOP-GBAY-0145, *Laboratory Supply Procedures* (most current revision or replacement).

This SOP includes each of the following requirements:

- Procedures for documentation of receipt and tracking. The record of entry includes name of the material, the lot number, receipt date, and expiration date.
- Storage conditions and requirements. Reference materials must be stored separately from samples, extracts, and digestates.
- Requirements to assure that preparations of intermediate or working solutions are recorded and assigned a unique identification number for tracking. Records of preparation include the lot number of the stock standard(s) used, the type and lot number of the solvent, the formulation, date, expiration date, and the preparer's initials. The lot number of the working standards is recorded in the analytical record to provide traceability to the standard preparation record. The

preparation record provides traceability to the COA, which is traceable to SI or the national measurement standard.

- A requirement that the expiration dates of prepared standards may not exceed the expiration date of the parent standard. Standards, reference materials, and reagents are not used after their expiration dates unless their reliability is thoroughly documented and verified by the laboratory. If a standard exceeds its expiration date and is not re-certified, the laboratory removes the standard and/or clearly designates it as acceptable for qualitative/troubleshooting purposes only. All prepared standards, reference materials, and reagents are verified to meet the requirements of the test method through routine analyses of quality control samples.
- The second source materials used for verification of instrument calibration are obtained from a different manufacturer or different lot from the same manufacturer.
- Procedures to check reference materials for degradation and replacement of material if degradation or evaporation is suspected.
- Procedures for labeling. At a minimum the container must identify the material, the ID of the material and the expiration date. Original containers should also be labeled with date opened.

5.6.4.3 Intermediate Checks

Checks to confirm the calibration status of standards and materials are described in laboratory SOPs. These checks, include use of second source standards and reference materials reserved only for the purpose of calibration checks.

5.6.4.4 Transport and Storage

The laboratory handles and transports reference standards and materials in a manner that protects the integrity of the materials. Reference standard and material integrity is protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials. Standards and reference materials are stored separately from samples, extracts, and digestates. All standards are stored according to the manufacturer's recommended conditions. Temperatures colder than the manufacturer's recommendation are acceptable if it does not compromise the integrity of the material (e.g. remains in liquid state and does not freeze solid). In the event a standard is made from more than a single source with different storage conditions, the standard will be stored according to the conditions specified in the analytical method.

See the applicable analytical SOPs for specific reference material storage and transport protocols.

5.7 Sampling

Sampling refers to the field collection of samples and to subsamples taken by the laboratory for analysis from the field collected sample.

Subsampling procedures are included in the laboratory SOP ENV-SOP-GBAY-0129, *Laboratory Homogenization, Compositing, and Sub-Sampling* (most current revision or replacement) to assure the aliquot used for testing is representative of the field collected sample.

The requirements in the following subsections apply when field sampling is performed by the laboratory.

5.7.1 Sampling Plans and SOPs

When the laboratory performs field collection of samples, sampling is carried out in accordance with a written sample plan prepared by the customer or by the laboratory and by relevant sampling SOPs. These documents are made readily accessible at the sampling location. Sampling plans and SOPs are, whenever reasonable, based on appropriate governing methods and addresses the factors to be controlled to ensure the validity of the analytical results.

5.7.2 Customer Requested Deviations

When the customer requires deviations, additions, or exclusions from the documented laboratory sampling plan and/or procedure, the laboratory records the client's change request in detail with the sampling record, communicates the change to sampling personnel, and includes this information in the final test report.

5.7.3 Recordkeeping

The laboratory assures the sampling record includes the sampling procedure used, any deviations from the procedure, the date and time of sampling, the identification of the sampler, environmental conditions (if relevant), and the sampling location.

5.8 Sample Management & Handling

5.8.1 Procedures

The laboratory's procedures for sample management and handling are outlined in laboratory SOP ENV-SOP-GBAY-0006, *Sample Management and Review of Analytical Requests* (most current revision or replacement).

The procedures in these SOPs are established to maintain the safe handling and integrity of samples from transport, storage, to disposal and during all processing steps in-between; to maintain client confidentiality, and to protect the interests of PAS and its customers.

5.8.1.1 Chain of Custody

All samples received by the laboratory must be accompanied with a Chain of Custody (COC) record. The COC provides information about the samples collected and submitted for testing and documents the possession of samples from time of collection to receipt by the laboratory.

The COC record must minimally include the following information:

- Client name, address, phone number
- Project Reference
- Client Sample Identification (Client ID)

- Date, Time, and Location of Sampling
- Samplers Name or Initials
- Matrix
- Type of container, and total number collected each sample
- Preservatives
- Analyses Requested
- Mode of collection
- Any special instructions
- The date and time and signature of each sample transfer from time of collection to receipt in the laboratory. When the COC is transported inside the cooler, independent couriers do not sign the COC. Shipping manifests and/or air bills are the records of possession during transport.

A complete and legible COC is required. If the laboratory observes that the COC is incomplete or illegible, the client is contacted for resolution. The COC must be filled out in indelible ink. Personnel correct errors by drawing a single line through the initial entry so the entry is not obscured, entering the correct information, and initialing, and dating the change.

5.8.1.2 Legal Chain of Custody

Legal chain of custody is a chain of custody protocol used for evidentiary or legal purposes. The protocol is followed by the laboratory when requested by customer or where mandated by a regulatory program.

Legal chain of custody (COC) protocol establishes an intact, continuous record of the physical possession*, storage, and disposal of "samples" which includes, sample aliquots, and sample extracts/digestates/distillates.

Legal COC records account for all time periods associated with the samples, and identifies all individuals who physically handled individual samples. Legal COC begins at the point established by legal authority, which is usually at the time the sample containers are provided by the laboratory for sample collect or when sample collection begins.

*A sample is in someone's custody if:

- It is in one's physical possession;
- It is in one's view after being in one's physical possession;
- It has been in one's physical possession and then locked or sealed so that no one can tamper with it; and/or
- It is kept in a secure area, restricted to authorized personnel only.

Refer to laboratory SOP ENV-SOP-GBAY-0006, *Sample Management and Review of Analytical Requests* (most current revision or replacement) for more information.

5.8.2 Unique Identification

Each sample is assigned a unique identification number by the laboratory (Lab ID) after the sample has been checked and accepted by the laboratory in accordance with the laboratory's sample acceptance policy (See 5.8.3). the Lab ID is affixed to the sample container using a durable label.

The unique identification of samples also applies to subsamples, and prepared samples, such as extracts, digestates, etc.

The lab ID is linked to the field ID (client ID) in the laboratory's record. Both IDs are linked to the testing activities performed on the sample and the documentation records of the test.

Also see 5.8.4.

5.8.3 Sample Receipt Checks and Sample Acceptance Policy

The laboratory checks the condition and integrity of samples on receipt and compares the labels on the sample containers to the COC record. Any problem or discrepancy is recorded. If the problem impacts the suitability of the sample for analysis or if the documentation is incomplete, the client is notified for resolution. Decisions and instructions from the client are maintained in the project record.

5.8.3.1 Sample Receipt Checks

The following checks are performed:

- Verification that the COC is complete and legible.
- Verification that each sample's container label includes the client sample ID, the date and time of collection and the preservative in indelible ink.
- The container type and preservative is appropriate for each test requested.
- Adequate volume is received for each test requested.
- Visual inspection for damage or evidence of tampering.
- Visual inspection for presence of headspace in VOA vials. (VOA = volatile organic analysis).
- Thermal Preservation: For chemical testing methods for which thermal preservation is required, temperature on receipt is acceptable if the measurement is above freezing but <6°C. For samples that are hand-delivered to the laboratory immediately after sample collection, there must be evidence that the chilling process has begun, such as arrival on ice. The requirements for thermal preservation vary based on the scope of testing performed. For example, for microbiology, temperature on receipt is acceptable if the measurement is <10°C. Refer to the laboratory's SOP for sample receipt for more information.
- Chemical Preservation

- Holding Time: Sample receiving personnel are trained to recognize tests with tests where the holding time is 48 hours or less and to expedite the log-in of these samples. Except for tests with immediate holding times (15 minutes from time of collection or less), when samples are received out of hold, the laboratory will notify the client and request instruction. If the decision is made to proceed with analysis, the final test report will include notation of this instruction.

5.8.3.2 Sample Acceptance Policy

The laboratory maintains a sample acceptance policy in accordance with regulatory guidelines to clearly establish the circumstances in which sample receipt is accepted or rejected. When receipt does not meet acceptance criteria for any one of these conditions, the laboratory must document the noncompliance, contact the customer, and either reject the samples or fully document any decisions to proceed with testing. In accordance with regulatory specifications, test results associated with receipt conditions that do not meet criteria are qualified in the final test report.

All samples received must meet each of the following:

- Be listed on a complete and legible COC.
- Be received in properly labeled sample containers.
- Be received in appropriate containers that identify preservative.
- The COC must include the date and time of collection for each sample.
- The COC must include the test requested for each sample.
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be received within holding time. Any samples received beyond the holding time 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 $\leq 6°C$) unless program requirements or customer contractual obligations mandate otherwise. The cooler temperature is recorded directly on the COC. 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.

5.8.4 Sample Control and Tracking

The samples are controlled and tracked using the Laboratory Information Management System (LIMS). The LIMS stores information about the samples and project. The process of entering information into the LIMS is called login and these procedures are described in laboratory SOP ENV-SOP-GBAY-0006, *Sample Management and Review of Analytical Requests* (most current revision or replacement). After log-in, a label is generated and affixed to each sample container. Information on this label, such as the lab ID, links the sample container to the information in LIMS.

At a minimum, the following information is entered during log-in:

- Client Name and Contact Information;
- The laboratory ID linked to the client ID;
- Date and time of sample collection;
- Date and time of sample receipt;
- Matrix;
- Tests Requested.

5.8.5 Sample Storage, Handling, and Disposal

The laboratory procedures for sample storage, handling and disposal are detailed in laboratory SOPs ENV-SOP-GBAY-0006, *Sample Management and Review of Analytical Requests* and ENV-SOP-GBAY-0126, *Waste Handling and Management* (most current revision(s) or replacement(s).

5.8.5.1 Sample Storage

The samples are stored according to method and regulatory requirements as per test method SOPs. Samples are stored away from all standards, reagents, or other potential sources of contamination and 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.

Refrigerated storage areas are maintained at ≤6°C (but not frozen) and freezer storage areas are maintained at <-10°C (unless otherwise required per method or program). The temperature of each storage area is checked and documented at least once for each day of use. If the temperature falls outside the acceptable limits, then corrective actions are taken and appropriately documented.

The laboratory is 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 taken to the appropriate storage location immediately after sample receipt and login procedures are completed. All sample storage areas have limited access. Samples are removed from storage areas by designated personnel and returned to the storage areas as soon as possible after the required sample quantity has been taken.

5.8.5.2 Sample Retention and Disposal

The procedures used by the laboratory for sample retention and disposal are detailed in laboratory SOP ENV-SOP-GBAY-0126, *Waste Handling and Management* (most current revision or replacement).

In general, unused sample volume and prepared samples such as extracts, digestates, distillates and leachates (samples) are retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

Samples may be stored at ambient temperature when all analyses are complete, the hold time is expired, the report has been delivered, and/or when allowed by the customer or program. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

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.

5.9 Assuring the Quality of Test Results

5.9.1 Quality Control (QC) Procedures

The laboratory monitors the validity and reliability of test results using quality control (QC) samples that are prepared and analyzed concurrently with field samples in the same manner as field samples. QC results are always associated to and reported with the field samples they were prepared and analyzed with from the same preparation or analytical batch. See the glossary for definition of preparation and analytical batch.

The results of QC performed during the testing process are used by the laboratory to assure the results of analysis are consistent, comparable, accurate, and/or precise within a specified limit. When the results are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

Other QC measures performed include the use of certified reference materials (see 5.6.2), participation in interlaboratory proficiency testing (see 5.9.1.1), verification that formulae used for reduction of data and calculation of results is accurate (see 5.9.3), on-going monitoring of environmental conditions that could impact test results (see 5.3.2), and evaluation and verification of method selectivity and sensitivity (see 5.4.5).

QC results are also used by the laboratory to monitor performance statistical trends over time and to establish acceptance criteria when no method or regulatory criteria exist. (see 5.9.1.4).

5.9.1.1 Essential QC

Although the general principles of QC for the testing process apply to all testing, the QC protocol used for each test depends on the type of test performed.

QC protocol used by the laboratory to monitor the validity of the test are specified in test method SOPs. The SOP includes QC type, frequency, acceptance criteria, corrective actions, and procedures for reporting of nonconforming work.

These requirements in the SOP conform to the reference method and any applicable regulations or certification and accreditation program requirement for which results of the test are used. When a project requires more stringent QC protocol than specified in the SOP, project specification is followed. When the project requires less stringent QC protocol, the project specification may be followed as an authorized departure from the SOP when the project specifications meet the requirements in the mandated method and any regulatory compliance requirements for which the data will be used.

The following are examples of essential QC for Chemistry:

5.9.1.1.1 Second Source Standard (ICV/QCS)

The second source standard is a standard obtained from a different vendor than the vendor of the standards used for calibration. It is a positive control used to verify the accuracy of a new calibration relative to the purity of the standards used for calibration. This check is referred to in test method and quality system standards as the initial calibration verification (ICV) or quality control sample (QCS). The second source standard is analyzed immediately after the calibration and before analysis of any samples. When the ICV is not within acceptance criteria, a problem with the purity or preparation of the standards may be indicated.

5.9.1.1.2 Continuing Calibration Verification (CCV)

CCV is to determine if the analytical response has significantly changed since initial calibration. If the response of the CCV is within criteria, the calibration is considered valid. If not, there is a problem that requires further investigation. Actions taken are technology and method specific.

5.9.1.1.3 Method Blank (MB) / Other Blanks

A method blank is a negative control used to assess for contamination during the prep/analysis process. The MB consists of a clean matrix, similar to the associated samples that is known to be free of analytes of interest. The MB is processed with and carried through all preparation and analytical steps as the associated samples.

In general, contamination is suspected when the target analyte is detected in the MB above the reporting limit. Some programs may require evaluation of the MB to ½ the reporting limit or the detection limit. When contamination is evident, the source is investigated and corrections are taken to reduce or eliminate it. Analytical results associated with MB that does not meet criteria are qualified in the final test report.

Other types of blanks that serve as negative controls in the process may include:

- Trip Blanks (VOA)
- Storage Blanks
- Equipment Blanks
- Field Blanks
- Calibration Blanks
- Cleanup Blanks
- Instrument Blanks

5.9.1.1.4 Laboratory Control Sample (LCS)

The LCS is positive control used to measure the accuracy of process in a blank matrix. The LCS is spiked by the laboratory with a known amount of analyte. The spike is a standard solution that is pre-made or prepared from a certified reference standard. The LCS is processed with and carried through all preparation and analytical steps as the associated samples.

When the percent recovery (%R) of the LCS is within the established control limit, sufficient accuracy has been achieved. If not, the source of the problem is investigated and corrected and the procedure may be repeated. Analytical results associated with LCS that does not meet criteria are qualified in the final test report.

5.9.1.1.5 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Matrix spikes measures the effect the sample matrix has on precision and accuracy of the determinative test method. The MS and MSD are replicates of a client sample that is spiked with known amount of target analyte.

Due to the heterogeneity of matrices even of the same general matrix type, matrix spike results mostly provide information on the effect of the matrix to the client whose sample was used and on samples of the same matrix from the same sampling site. Therefore, MS should be client-specific when the impact of matrix on accuracy and precision is a project data quality objective. When there is not a client-specified MS for any sample in the batch, the laboratory randomly selects a sample from the batch; the sample selected at random is called a "batch" matrix spike.

The MS/MSD results for percent recovery and relative percent difference are checked against control limits. Because the performance of matrix spikes is matrix-dependent, the result of the matrix spike is not used to determine the acceptability of the test.

5.9.1.1.6 Sample Duplicate (SD)

A sample duplicate is a second replicate of sample that is prepared and analyzed in the laboratory along another replicate. The SD is used to measure precision.

The relative percent difference between replicates are evaluated against the method or laboratory derived criteria for relative percent difference (RPD), when this criterion is applicable. If RPD is not met, associated test results are reported with qualification.

5.9.1.1.7 Surrogates

Surrogates are compounds that mimic the chemistry of target analytes but are not expected to occur naturally in real world samples. Surrogates are added to each sample and matrix QC samples (MS, MSD, SD) at known concentration to measure the impact of the matrix on the accuracy of method performance. Surrogates are also added to the positive and negative control samples (MB, LCS) to evaluate performance in a clean matrix, and included in the calibration standards and calibration check standards.

The percent recovery of surrogates is evaluated against method-specified limits or statistically derived in-house limits. Project-specific limits and/or program-specific limits are used when required. Results with surrogate recovery out of limits in samples are reported with qualification. Samples with surrogate failures can also be 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.

5.9.1.1.8 Internal Standards

Internal Standards are compounds not expected to occur naturally in field samples. They are added to every standard and sample at a known concentration prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. The laboratory follows specific guidelines for the treatment of internal standard recoveries and further information can be found in the applicable laboratory SOP.

5.9.1.1.9 QC Acceptance Criteria and Control Limits

The QC acceptance criteria are specified in test method SOPs. The criteria in the SOP are based on the requirements in the published test method or regulatory program. When there are no established acceptance criteria, the laboratory develops acceptance criteria in accordance with recognized industry standards.

Some methods and programs require the laboratory to develop and use control limits for LCS, MS/MSD and surrogate evaluation. In

laboratory developed limits are referred to as "in-house" control limits. In-house control limits represent ± 3 Standard Deviations (99% confidence level) from the average recovery of at least 20 data points generated using the same preparation and analytical procedure in a similar matrix.

See laboratory SOP ENV-SOP-GBAY-0116, *Control Charting and Trend Analysis* (most current revision or replacement) for more information.

5.9.1.2 Proficiency Testing (PT)

The laboratory participates in interlaboratory proficiency testing (PT) studies to measure performance of the test method and to identify or solve analytical problems. PT samples measure laboratory performance through the analysis of unknown samples provided by an external source.

The PT samples are obtained from accredited proficiency testing providers (PTP) and handled as field samples which means they are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results during the duration of the study, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

The laboratory initiates an investigation and corrective action plan whenever PT results are deemed unacceptable by the PT provider.

The frequency of PT participation is based on the certification and accreditation requirements held by the laboratory.

5.9.2 QC Corrective Action

When the results of QC are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken per the specifications in the test method SOP. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

5.9.3 Data Review

The laboratory uses a tiered system for data review. The tiered process provides sequential checks to verify data transfer is complete; manual calculations, if performed, are correct, manual integrations are appropriate and documented, calibration and QC requirements are met, appropriate corrective action was taken when required, test results are properly qualified, process and test method SOPs were followed, project specific requirements were met, when applicable, and the test report is complete.

The sequential process includes three tiers referred to as primary review, secondary review, and administrative/completeness review.

Detailed procedures for the data review process are described in laboratory SOP ENV-SOP-

GBAY-0117, *Data Review Process* (most current revision or replacement). The general expectations for the tiered review process are described in the following sections:

5.9.3.1 Primary Review

Primary review is performed by the individual that performed the task. All laboratory personnel are responsible for review of their work product to assure it is complete, accurate, documented, and consistent with policy and SOPs.

Checks performed during primary review include but are not limited to:

- Verification that data transfer and acquisition is complete
- Manual calculations, if performed, are documented and accurate
- Manual integrations, if performed, are documented and comply with SOP ENV-SOP-CORQ-006 *Manual Integration*
- Calibration and QC criteria were met, and/or proper correction and corrective actions were taken, and data and test results associated with QC and criteria exceptions are properly qualified
- Work is consistent with SOPs and any other relevant instructional document such as SWI, program requirements, or project QAPP

5.9.3.2 Secondary Review

Secondary review is performed by qualified peer or supervisor. Secondary review is essentially a repeat of the checks performed during primary review by another person. In addition to the checks of primary review, secondary review includes chromatography review to check the accuracy of quantitative analyte identification.

5.9.3.3 Completeness Review

Completeness review is an administrative review performed prior to release of the test report to the customer. Completeness review verifies that the final test report is complete and meets project specification. This review also assures that information necessary for the client's interpretation of results are explained in the case narrative or footnoted in the test report.

5.9.3.4 Data Audits

In addition to the 3 tier data review process, test reports may be audited by local QA to verify compliance with SOPs and to check for data integrity, technical accuracy, and regulatory compliance. These audits are not usually done prior to issuance of the test report to the customer. The reports chosen for the data audits are selected at random.

If any problems with the data or test results are found during the data audit, the impact of the nonconforming work is evaluated using the process described in Section 4.9.

Also see Section 4.14 for internal audits.

5.10 Reporting

5.10.1 General Requirements

The laboratory reports results of testing in a way that assures the results are clear, and unambiguous. All data and results are reviewed prior to reporting to assure the results reported are accurate and complete.

Test results are summarized in test reports that include all information necessary for the customer's interpretation of the test results. Additional information necessary to clarify the data or disclose nonconformance, exceptions, or deviations that occurred during the analytical process are also reported to the customer in the test report.

The specifications for test reports and electronic data deliverables (EDD) are established between the laboratory and the customer at the time the request for analytical services is initiated. The report specifications include the test report format, protocol for the reporting limit (RL), conventions for the reporting of results less than the limit of quantitation (LOQ), and specification for the use of project or program specific data qualifiers. Information about review of analytical service requests is provided in Section 4.4.

5.10.2 Test Reports: Required Items

Test Reports are prepared by the laboratory at the end of the testing process. The format of the report depends on the level of reporting requested by the customer. The laboratory offers a variety of standardized test report formats and can also can provide custom test report formats, when necessary.

The level of detail required in the test report depends on the customer's needs for data verification, validation, and usability assessments that occur after the laboratory releases the test report to the customer. The test report formats offered by the laboratory provide gradient levels of detail to meet the unique needs of each customer. The laboratory project manager helps the customer select the test report format that best meets their needs. When a specific report format or protocol is required for a regulatory or program compliance, the laboratory project manager must ensure the test report selected meets those requirements.

Every test report issued by the laboratory includes each of the following items:

a) Title

b) Name and phone number of a point of contact from the laboratory issuing the report.

c) Name and address of the laboratory where testing was performed. When testing is done at multiple locations within network (IRWO), the report must clearly identify which network laboratory performed each test and must include the physical address of each laboratory.

d) Unique identification of the test report and an identifier on each page of the report to link each page to the test report and clear identification of the end of the report.

e) The name and address of the customer

f) Identification of test methods used

g) Cross reference between client sample identification number (Sample ID) and the laboratory's identification number for the sample (Lab ID) to provide unambiguous identification of samples.

h) The date of receipt of samples, condition of samples on receipt, and identification of any instance where receipt of the samples did not meet sample acceptance criteria.

i) Date and times of sample collection, receipt, preparation, and analysis.

j) Test results and units of measurement, and qualification of results associated with QC criteria exceptions, and identification of reported results outside of the calibration range.

k) Name, title, signature of the person(s) authorizing release of the test report and date of release.

l) A statement that the results in the test report relate only to the items tested.

m) Statement that the test report may not be reproduced except in full without written approval from the laboratory.

5.10.3 Test Reports: Supplemental Items

5.10.3.1 Supplemental Requirements

The following items are included in the test report when required or relevant:

a) Explanation of departure from test method SOPs including, what the departure was and why it was necessary.

b) Statistical methods used. (Required for Whole Effluent Toxicity)

c) For solid samples, specification that results are reported on a dry weight or wet weight basis.

d) Signed Affidavit, when required by client or regulatory agency.

e) A statement of compliance / non-compliance with requirements or specifications (client, program, or standard) that includes identification of test results that did not meet acceptance criteria.

f) When requested by the client, statement of estimated measurement uncertainty. In general, for environmental testing, estimated uncertainty of measurement is extrapolated from LCS control limits. Control limits incorporate the expected variation of the data derived from the laboratory's procedure. When the control limits are specified by the test method or regulatory program, the control limits represent the expected variation of the test method and/or matrices for which the test method was designed.

g) Opinions and Interpretations.

h) If a claim of accreditation/certification is included in the test report, identification of any test methods or analytes for which accreditation/certification is not held by the laboratory if the accrediting body offers accreditation/certification for the test method/analyte. The fields of accreditation/certification vary between agencies and it cannot be presumed that because accreditation/certification is not held that it is offered or required.

i) Certification Information, including certificate number and issuing body.

5.10.3.2 Test Reports: Sampling Information

The following items are included in the test report when samples are collected by the laboratory or when this information is necessary for the interpretation of test results:

a) Date of Sampling.

b) Unambiguous identification of material samples.

c) Location of sampling including and diagrams, sketches, or photographs.

d) Reference to the sampling plan and procedures used.

e) Details of environmental conditions at time of sample that may impact test results.

f) Any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

5.10.4 Calibration Certificates

The laboratory does not perform calibration activities for its customers and calibration certificates are not offered or issued.

5.10.5 Opinions and Interpretations

The laboratory provides objective data and information to its customers of sufficient detail for their interpretation and decision making. Objective data and information is based solely on fact and does not attempt to explain the meaning (interpret) or offer a view or judgement (opinion). Sometimes the customer may request the laboratory provide opinion or interpretation to assist them with their decisions about the data.

When opinions and interpretations are included in the test report, the laboratory will document the basis upon which the opinions and interpretations have been made and clearly identify this content as opinion or interpretation in the test report.

Examples of opinion and interpretation include but are not limited to:

- The laboratory's viewpoint on how a nonconformance impacts the quality of the data or usability of results.
- The laboratory's judgment of fulfillment of contractual requirements.
- Recommendations for how the customer should use the test results and information.
- Suggestions or guidance to the customer for improvement.

When opinions or interpretations are verbally discussed with the customer, the content of these conversations is summarized by the laboratory and kept in the project record.

5.10.6 Subcontractor Reports

When analytical work has been subcontracted to an organization external to PAS, the test report from the subcontractor is included in its entirety as an amendment to the final test report.

Note: Test results for analytical work performed within the PAS network may be are merged into a single test report. The test report issued clearly identifies the location and address of each network location that performed testing and which tests they performed. (See 5.10.2)

5.10.7 Electronic Transmission of Results

When test results and/or reports are submitted to the customer through electronic transmission, follow the procedures established in this manual for confidentiality and protection of data.

5.10.8 Format of Test Reports

The test formats offered by the laboratory are designed to accommodate each type of analytical test method carried out by the laboratory and to minimize the possibility of misunderstanding or misuse of analytical results. The format of electronic data deliverables (EDD) follow the specifications for the EDD.

5.10.9 Amendments to Test Reports

Test reports that are revised or amended by the laboratory after date of release of the final test report to the customer are issued as a new test report that is clearly identified as an amendment or revision and that includes a reference to the originally issued final test report.

The customer is the organization doing business with PAS external to PAS.

Changes made to test results and data before the final test report is issued to the customer are not amendments or revisions, these are corrections to errors found during the laboratory's data verification and review process,

The laboratory's procedure for report amendments and revision are outlined in laboratory SOP ENV-SOP-GBAY-0120, *Final Report and Data Deliverable Contents* (most current revision or replacement).

6.0 Revision History

This Version: ENV-MAN-GBAY-0001

Section	Description of Change
All	This version is a complete rewrite of the document this version supersedes.

This document supersedes the following documents:

Document Number	Title	Version
ENV-MAN-CORQ-0001	Quality Manual	00
ENV-MAN-GBAY-0001	Quality Manual	00

7.0 APPENDICES

7.1 Appendix A: Certification / Accreditation Listing

The certifications / accreditation lists provided in this manual represent those that were held by the named location on the effective date of this manual. This information is subject to change without notice and must not be considered valid proof of certification or accreditation status. Current certificates are maintained by Local QA and a copy of the certificate is posted to PAS's eDMS Portal for access by all PAS employees. External parties should contact the laboratory for the most current information.

7.1.1 PAS-Green Bay

Authority	Certificate Number	Authority	Certificate Number
Florida Department of Health, Bureau of Laboratories	E87948	North Dakota Department of Health Chemistry Division	R-150
Georgia, Environmental Protection Division	E87948	South Carolina Department of Health and Environmental Control	83006001
Illinois EPA	200050	Texas Commission on Environmental Quality	T104704529-14-1
Kentucky Environmental and Public Protection Cabinet	82	Virginia Department of General Services	5537
Louisiana Department of Environmental Quality	04168	Wisconsin Department of Natural Resources	405132750
Minnesota Department of Health	055-999-334	Wisconsin Department of Agriculture, Trade and Consumer Protection	105-444
New York Department of Health	12064		

7.2 Appendix B: Capability Listing

The capabilities listed in this Appendix were held by the location referenced on the effective date of this manual. This information is subject to change without notice. External parties should contact the laboratory for the most current information.

Table Legend:

- DW = Drinking Water
- NPW = Non-Potable Water
- SCM = Solid and Chemical Materials
- Waste = Non-Aqueous Phase Liquid (NAPL), Oil
- Tissue = Biota and Tissue

7.2.1 PAS-Green Bay

Parameter	Method	Matrices				
		DW	NPW	SCM	Waste	Tissue
Dry Weight	ASTM D2974-87			x	x	x
ASTM Leach	ASTM D3987-85			x		
Flashpoint	EPA 1010A		x	x		
Specific Conductance	EPA 120.1		x			
TCLP Leach	EPA 1311		x	x	x	
SPLP Leach	EPA 1312		x	x	x	
Solids, Total (TS)	SM 2540 B		x			
Solids, Total Dissolved (TDS)	SM 2540 C		x			
Solids, Total Suspended (TSS)	SM 2540 D		x			
Solids, Total Volatile Suspended (TVSS)	SM 2540 E		x			
Solids, Total Volatile (TVS)	EPA 160.4		x	x		
Solids, Volatile Suspended (TVSS)	EPA 160.4		x			
Solids, Total Percent	SM 2540 G			x		
AVS/SEM	EPA 1629			x		
Turbidity	EPA 180.1		x			
Turbidity	SM 2130 B		x			
Ion Chromatography	EPA 300.0	x	x	x		
Ion Chromatography	EPA 9056A		x			
Acidity, Total	EPA 305.1		x			
Acidity, Total	SM 2310 B		x	x		
Alkalinity, Total	EPA 310.2		x			

Parameter	Method	Matrices				
		DW	NPW	SCM	Waste	Tissue
Alkalinity, Total	SM 2320 B		x			
Cyanide, Total	EPA 335.4		x			
Cyanide, Total	EPA 9012B		x	x		
Ammonia, Total	EPA 350.1		x	x		
Total Kjeldahl Nitrogen	EPA 351.2		x	x		
Nitrogen, NO2/NO3	EPA 353.2		x	x		
Phosphorous, Total	EPA 365.4		x	x		
Chemical Oxygen Demand	EPA 410.4		x			
pH	EPA 9040C		x	x		
pH	SM 4500-H+-B		x			
pH	EPA 9045D			x		
Carbon, Total Organic	SM 5310 C		x			
Carbon, Total Organic (Quad/Mod)	EPA 9060A		x	x		
Carbon, Total Organic	Lloyd Kahn			x		
Carbon, Total Organic	Walkley-Black			x		
Paint Filter Liquid Test	EPA 9095A			x		
Iron, Ferrous	HACH 8146		x			
Iron, Ferric Calculation	EPA6010/6020 HACH 8146		x			
Apparent Color	SM 2120 B		x			
Specific Gravity	SM 2710 F		x			
Chromium, Hexavalent	SM 3500-Cr B		x			
Oxygen, Dissolved	SM 4500-O G		x			
Sulfide	SM 4500-S F		x			
Biochemical Oxygen Demand	SM 5210 B		x	x		
Heterotrophic Plate Count	SM 9215B		x	x		
Coliform, Fecal	SM 9222D	x	x			
Coliform, Total	SM 9223		x			
Mercury, Low Level	EPA 1631E		x	x		x
Mercury, Total	EPA 7470A		x			
Mercury, Total	EPA 7471B			x		x
Mercury, Total	EPA 245.1		x			

Parameter	Method	Matrices				
		DW	NPW	SCM	Waste	Tissue
Mercury, Total	EPA 245.6					x
Mercury, Total	EPA 7473					x
ICP-Metals	SW846 6010B /6010C /EPA		x	x		
ICPMS-Metals	SW846 6020 / 6020A /EPA		x	x		x
TPH-Diesel	SW846 8015C/D		x	x	x	
Diesel Range Organics	WI Modified DRO		x	x	x	
Organochlorine Pesticide/ Toxaphene	SW846 8081A / 8081B / EPA 608 / 608.3		x	x		x
Polychlorinated Biphenyls (PCB)	SW846 8082 / 8082A / EPA 608 / 608.3		x	x		x
Polyaromatic Hydrocarbons (PAH)	SW846 8270C– SIM / EPA 625.1		x	x		x
Semi-Volatile Organics	SW846 8270C / EPA 625.1		x	x		
TPH-Gasoline	SW846 8015C		x	x		
Gasoline Range Organics	WI Modified GRO		x	x		
Methane, Ethene, Ethane	SW846 8015C Mod		x			
PVOC	SW846 8021B / EPA 602		x	x		
Volatile Organics	SW846 8260B / EPA 624.1		x	x		

7.3 Appendix C: Glossary

This glossary provides common terms and definitions used in the laboratory. **It is not intended to be a complete list of all terms and definitions used.** The definitions have been compiled mostly from the TNI Standard and DoD QSM. Although this information has been reproduced with care, errors cannot be entirely excluded. Definitions for the same term also vary between sources. When the meaning of a term used in a laboratory document is different from this glossary or when the glossary does not include the term, the term and definition is included or defined in context in the laboratory document.

Term	Definition
3P Program	PAS-The continuous improvement program used by PAS that focuses on Process, Productivity, and Performance.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies.* The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- 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.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.
Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by PAS as every 12 months ± 30 days.
Assessment	TNI- The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	TNI- 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 or the time frame specified by the regulatory program. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/ Expected Data Points)*100
Confirmation	TNI- 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. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
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	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and 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 Calibration Verification Standard (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.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
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.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
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.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence $(1-\alpha)$ that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
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	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.
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.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.

False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
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 sPAS to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.
Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation 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. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).
Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.

In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
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 that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	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 Standard	TNI and DoD- A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
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. For example, hexane (C_6H_{14}) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A body that calibrates and/or tests.

Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
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.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
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	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.

Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- 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)	TNI- 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.
Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, $1-\beta$, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
MintMiner	Program used by PAS to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).

National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	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.
Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).
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.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
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.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.

Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
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.
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)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.

Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.
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.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.

Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-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 (RLVS)	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.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.
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 sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a chain-of-custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected 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. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	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 (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.
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)	TNI- 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)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
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.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
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	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD–A definitive procedure that determines one or more characteristics of a given substance or product.
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.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.

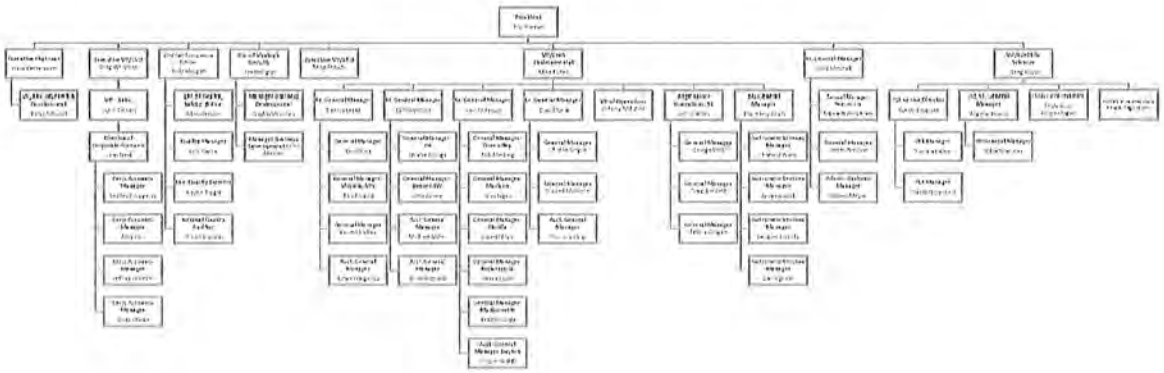
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
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, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where k > 1).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. 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.
Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

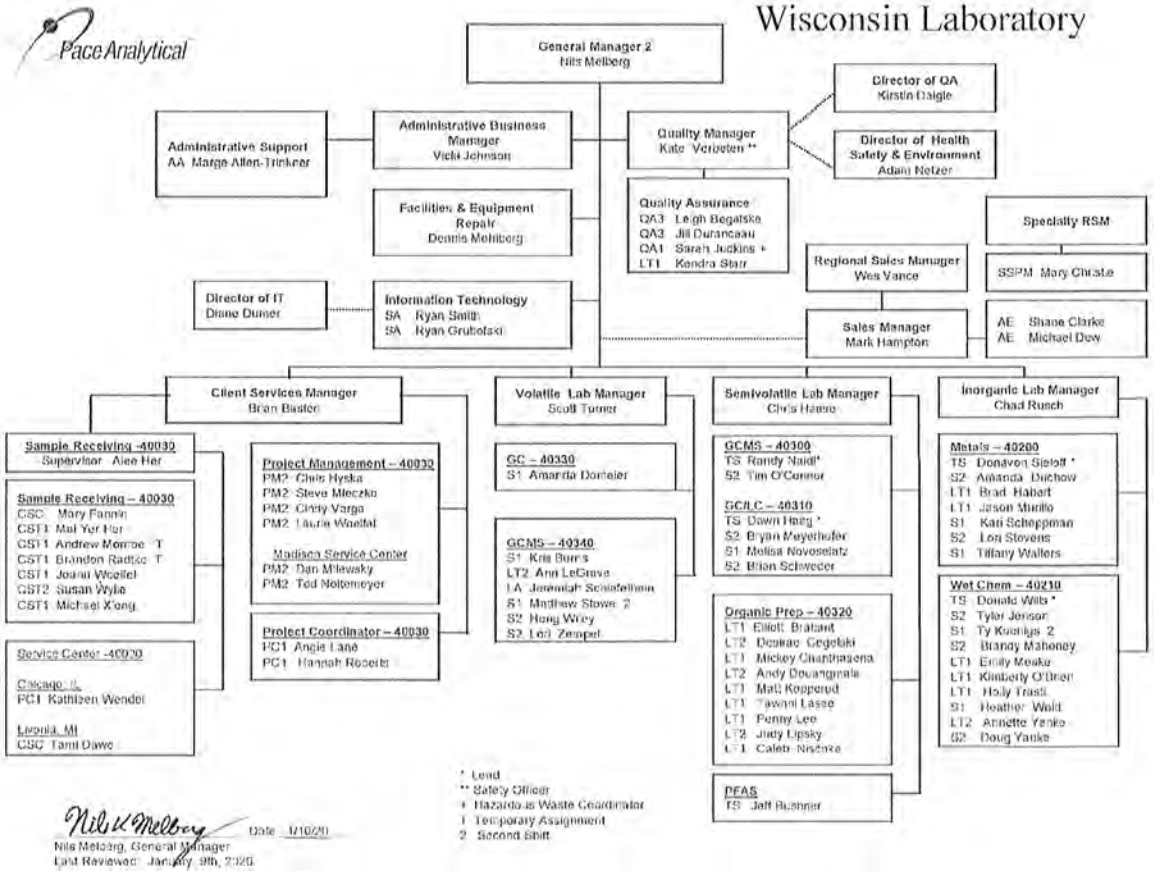
7.4 Appendix D: Organization Chart(s)

7.4.1 PAS – Corporate

Management Staff
December 2019



7.4.2 PAS – Green Bay



7.5 Appendix E: Equipment Listing

The equipment listed represents equipment were held by each location on the effective date of this manual. This information is subject to change without notice. External parties should contact the location for the most current information.

7.5.1 PAS-Green Bay

Equipment List: PAS-Green Bay

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
Quick Trace Mercury Analyzer	Cetac	M-7500	051104QTA	6/15/11	New	Metals Dept	40HG2	At Instrument
Direct Mercury Analyzer	Milestone	DMA-80	10070875	11/3/11	New	Metals Dept	40HG4	On-line
ICPMS	Thermo	X Series 2	01301C	6/11/08	New	Metals Dept	40ICM2	On-line
ICPMS	Thermo	X Series 2	01780C	8/21/10	New	Metals Dept	40ICM3	On-line
ICP	Thermo	ICAP 6500	20073913	10/1/04	New	Metals Dept	40ICP2	On-line
Low Level Mercury	Analytik Jena	Mercur	K170A0130	10/18/10	New	LL Hg	40LHG4	On-line
Low Level Mercury	Cetac	M-8000	111003QM8	8/17/12	New	LL Hg	40LHG5	On-line
GC/FID	Hewlett Packard	5890 Series II	3140A38457	10/1/04	Used	SVOA	40GCS1	At instrument
GC/ECD	Agilent	6890N	US10538012	8/1/10	Used	SVOA	40GCS7	At instrument
GC/ECD	Hewlett-Packard	6890	US00031701	10/1/04	Used	SVOA	40GCS8	At instrument
GC/ECD	Hewlett-Packard	6890	US00021961	10/1/04	Used	SVOA	40GCS9	At instrument
GC/ECD	Agilent	6890	US00040655	10/1/04	Used	SVOA	40GCSB	At instrument
GC/ECD	Hewlett-Packard	6890	US00024921	10/1/04	Used	SVOA	40GCSC	At instrument
GC/FID	Agilent	7890	CN10912008	5/19/09	New	SVOA	40GCSF	At instrument
GC/ECD	Agilent	7890B	CN14043012	3/1/14	New	SVOA	40GCSG	At instrument
GC/ECD	Hewlett-Packard	6890	US10344089	3/1/14	New	SVOA	40GCSH	At instrument
GC/ECD	Agilent	6890	US10443037	3/1/14	New	SVOA	40GCSJ	At instrument
GC/MS	Hewlett-Packard	6890	US81221570	2/1/00	New	SVOA	40MSS2	At instrument
GC/MS	Hewlett-Packard	6890	US00024414	4/1/99	New	SVOA	40MSS4	At instrument
GC/MS	Hewlett-Packard	5890	3310A49571	10/1/04	Used	SVOA	40MSS6	At instrument
GC/MS	Agilent	7890A	CN10752040	8/5/10	New	SVOA	40MSS7	At instrument
GC/MS	Agilent	7890A	CN10705029	9/4/13	New	SVOA	40MSS8	At instrument
GC/MS	Agilent	6890N	US10540022	6/1/14	Used	SVOA	40MSS9	At instrument
GC/MS	Agilent	7890B	15483197	1/14/16	New	SVOA	40MSSA	At instrument
GC/PID/FID	Hewlett-Packard	5890	3310A48054	3/1/92	New	VOA	40GCV1	At instrument

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
GC/PID/FID	Hewlett-Packard	5890	3310A48054	3/1/92	New	VOA	40GCV2	At instrument
GC/PID/FID	Hewlett-Packard	5890	3140A39241	6/1/93	New	VOA	40GCV3	At instrument
GC/PID/FID	Hewlett-Packard	5890	3336A60500	12/1/95	New	VOA	40GCV4	At instrument
GC/PID/FID	Hewlett-Packard	5890	3310A47921	4/1/95	New	VOA	40GCV5	At instrument
GC/FID	Hewlett-Packard	5890	2843A20939	7/1/95	New	VOA	40GCV8	At instrument
GC/MS	Hewlett-Packard	5890	3235A46437	5/14/93	New	VOA	40MSV1	At instrument
GC/MS	Hewlett-Packard	6890	US00032794	10/18/04	New	VOA	40MSV2	At instrument
GC/MS	Agilent	6850	CN10719006	3/6/08	New	VOA	40MSV3	At instrument
GC/MS	Hewlett-Packard	6890	US00025880	11/20/01	New	VOA	40MSV5	At instrument
GC/MS	Hewlett-Packard	6890	US00040707	9/25/02	New	VOA	40MSV7	At instrument
GC/MS	Agilent	6850	CN1065-1003	7/9/07	New	VOA	40MSV8	At instrument
GC/MS	Hewlett-Packard	7890	CN10031128	4/14/10	New	VOA	40MSVA	At instrument
GC/MS	Hewlett-Packard	7890A	CN10811039	2/5/13	New	VOA	40MSVB	At instrument
GC/MS	Hewlett-Packard	7890B	CN13283076	8/12/13	New	VOA	40MSVC	At instrument
GC/MS	Hewlett-Packard	7890B	CN13283076	8/12/13	New	VOA	40MSVD	At instrument
Oxygen Meter	YSI	5000	14F101753	8/20/14	New	Wet Chem.	40WET2	At instrument
Turbidi-meter	Hach	2100P	950400007487	10/1/04	Used	Wet Chem	40WET6	At instrument
Conductivity Meter	Accument	30	C0019471	10/1/04	Used	Wet Chem	40WET7	At instrument
pH Meter	Orion	720A	008577	10/1/04	Used	Wet Chem	40WET8	At instrument
EH Meter	Accumet	AB15	AB81200474	7/15/09	Used	Wet Chem	40WET9	At instrument
pH Meter	Symphony	SB20	00001852	7/12/10	New	Wet Chem	40WETB	At instrument
pH Meter	Orion	720A	006211	10/1/04	Used	Wet Chem	40WETC	At instrument
pH Meter	Corning	320	C3026	10/1/04	Used	Wet Chem	40WETD	At instrument
BOD AutoEZ	Thermo/Orion	10060020	A0117	9/5/12	New	Wet Chem	40WETE	At instrument
pH Meter	Orion Star	A211	X38338	7/25/17	New	Wet Chem	40WETF	At instrument
pH Meter	Orion Star	A211	X38338	7/25/17	New	Wet Chem	40WETG	At instrument
Flashpoint	Tanaka	Apm-8fc	33930	10/17/17	New	Wet Chem	40WETH	At instrument
Direct Reading Spectro-photometer	Hach	DR 2000	960300039446	10/1/04	Used	Wet Chem	40WTA1	At instrument
Ion Chromato-graph	Dionex	DX-120	97040612	10/1/04	Used	Wet Chem	40WTA3	At instrument

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
Apollo	Teknar/ Dohrmann	Apollo 9000	99174002	10/1/04	Used	Wet Chem	40WTA5	At instrument
Fusion	Teledyne	14-9600-100	US08105007	10/1/04	Used	Wet Chem	40WTA7	At instrument
Ion Chromato-graph	Thermo Scientific	ICS-110	12051009	8/3/12	New	Wet Chem	40WTAB	At instrument
Quick Chem 8500 Series II	Lachat	8500 Series 2	120600001428	8/13/12	New	Wet Chem	40WTAC	At instrument
Ion Chromato-graph	Thermo Scientific	ICS-1100	13040963	7/16/13	New	Wet Chem	40WTAD	At instrument
Quik Chem 8500 Series II	Lachat	8500 Series 2	140500001688	6/10/14	New	Wet Chem	40WTAE	At instrument
Ion Chromato-graph	Thermo Scientific	Aquion	160640270	8/19/16	New	Wet Chem	40WTAF	At instrument
Analytik Jena	Analytik Jena	Multi EA 4000	N4-167/N	8/1/17	New	Wet Chem	40WTAG	At instrument
Quik Chem 8500 Series II	Lachat	8500 Series 2	191000002249	12/6/19	New	Wet Chem	40WTAH	At instrument
Omnis Titrator	Metrohm	110010010	001000158371	12/9/19	New	Wet Chem	40WTAI	At instrument
TOC-VWP	Shimadzu	TOC-VWP	H51725600347	12/16/19	New	Wet Chem	40WTAJ	At instrument

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-GBAY-0004 **Revision:** 03

Document Title: Measurement of Percent Moisture in Soils and Solids

Department(s): Client Services

Date Information

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Notes

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Document Number: ENV-SOP-GBAY-0004 **Revision:** 03

Title: Measurement of Percent Moisture in Soils and Solids

All dates and times are in Central Time Zone.

ENV-SOP-GBAY-0004-Rev.03 Measurement of Percent Moisture in Soils and Solids

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Kate Verbeten (007119)	Manager - Quality	02 Sep 2020, 03:18:32 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Nils Melberg (007142)	General Manager 2	02 Sep 2020, 03:33:30 PM	Approved
Christopher Haase (007121)	Manager	02 Sep 2020, 03:37:25 PM	Approved
Alee Her (007671)	Supervisor	08 Sep 2020, 09:39:17 AM	Approved

1.0 Scope And Application

This standard operating procedure (SOP) describes the laboratory procedure for the measurement percent moisture of soils and solid samples based on ASTM D2974-87 Standard Test Methods.

1.1 **Personnel:** The policies and procedures contained in this SOP are applicable to all 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.

1.2 **Parameters:** This SOP applies to percent moisture typically used to correct results of inorganic and organic parameter analysis to dry weight basis.

2.0 Summary of Method

2.1 A sample aliquot is weighed before and after heating to dryness at 103-105°C. The weight loss is calculated as % Moisture.

3.0 Interferences

3.1 Non-representative materials, e.g., leaves and sticks, should be removed from the sample prior to measurement.

3.2 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.

4.0 Definitions

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

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.

5.1.1 Regulated soil samples are to be handled in accordance with Pace SOP: ENV-SOP-GBAY-0121, *Regulated Soil Handling* (current revision or replacement).

5.2 **DO NOT WEAR LATEX OR NITRILE GLOVES WHILE HANDLING HOT TRAYS.**

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when enough information about sample collection is provided with the samples.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory SOP ENV-SOP-GBAY-0007 *Bottle Preparation* (current revision or replacement).

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Solid/Soil	Wide mouth glass or plastic 4-oz container	16 grams	Thermal: ≤6°C Chemical: N/A	30 Days. Note: Analyze as soon as possible to minimize microbiological decomposition of organic solids.

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (most recent revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ≤6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ≤6°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 30 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment:

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
Analytical Balance	A&D	EK-200i	40BALN/ 40BALQ	Electronic with RS-232 output, capable of weighing 0.01g
Analytical Balance	A&D	GH200	40BALP	Electronic with RS-232 output, capable of weighing 0.01g
Analytical Balance	A&D	GH200	40BALU	Electronic with RS-232 output, capable of weighing 0.01g
Drying Oven	VWR	1370 FM	40OVN7	Capable of maintaining temperature at 103-105°C
Drying Oven	VWR	1370 GM	40OVNH	Capable of maintaining temperature at 103-105°C
Computer for Electronic Prep Log				Automated sample weight upload into LIMS

7.2 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	Drierite™
Non-indicating Desiccant	Fisher	Fisher p/n 07-577-3B	Drierite™
Disposable Aluminum Weighing Dishes	Fisher	Fisher p/n 08-732	
Lab Spoons	Fisher	Fisher p/n 14-375-10	Spoonula™
Trays, plastic or metal	NA	NA	

8.0 REAGENTS AND STANDARDS

Not applicable to this SOP.

9.0 PROCEDURE

9.1 **Calibration and Standardization:**

9.1.1 Analytical Balance Calibration

9.1.1.1 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, ENV-SOP-GBAY-0115, *Support Equipment* (current revision or replacement).

9.1.2 Daily Calibration Check

9.1.2.1 Clean the balance and surrounding area prior to starting the daily calibration check.

9.1.2.2 Check the sight level on the balance. If it needs adjusting, level the balance.

9.1.2.3 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.

9.1.2.4 Tare the balance before weighing the NIST certified weights.

9.1.2.5 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 section 9.1.3).

9.1.3 Corrective Action:

9.1.3.1 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.

9.1.3.2 The internal calibration function (if available) of the balance may be used as a means of corrective action.

9.1.3.3 Utilize the internal calibration function and diagnostics. Refer to instrument manual.

9.1.3.4 Contact the QA office for assistance if the balance does not meet the calibration tolerances.

9.1.3.5 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.

9.1.3.6 Record any corrective action. Initial and date all entries in the logbook.

9.2 **Procedure**

9.2.1 Locate the samples to be analyzed, place on a cart and allow samples to warm to ambient temperature prior to processing.

9.2.2 Review location of samples. Soil samples that are collected in regulated domestic areas or that are of foreign origin must be handled in accordance with the Pace SOP: ENV-SOP-GBAY-0121, *Regulated Soil Handling* (most recent revision or replacement).

9.2.3 Determine the number of aluminum weighing pans required for the number of samples to be analyzed plus one for a duplicate (typically 21).

9.2.4 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 projects soil samples.

9.2.5 Review form ENV-FRM-GBAY-0047 *Dry Weight Instructions* (current revision) for information on batching and preparing the electronic prep log.

9.2.6 After batching samples in EPIC Pro print the work list.

9.2.7 Open the Electronic Prep Log. To start a new worksheet, select the template from the list of active templates. You may also search for them by clicking the triangle expanded button and entering in criteria for your search. Then press enter (or click the search button) to perform the search. Once you have the needed template, double click on it.

9.2.8 Now that the template is loaded you need to enter the Batch for your test. You can either drag over the Batch Samples in the order you need them, or you can drag them over and then reorder them using the drag and drop method. Once you have them in the order you need them click the arrows to the right of "Search by Batch" to minimize the space the "Search Samples" section takes up.

9.2.9 Save your data by pressing the disc icon next to the search template button. If this is your first time saving it will ask you to enter a prep group description. This is used by your group to find the Electronic Log you are making. Enter the name of the queue, batch number and lab group (for example, Sample Receiving enters "SR") e.g. PMST######SR, where ###### is the batch number.

9.2.10 Verify balance calibration - refer to section 9.1.1 for balance check procedures and corrective actions. Refer to the balance logbook for the acceptance criteria for the designated balance.

9.2.11 Now that you have your run set-up, you can start entering results.

9.2.12 To use the balance, verify the balance matches the instrument ID and click the balance icon in between the search button and the Auto Post button.

9.2.13 For each sample, label and record the tare weight (to the nearest 0.01 g) for an aluminum weighing dish by placing your curser in the field you wish to put the weight in and pressing the print button on the balance.

9.2.14 Using a clean Spoonula lab spoon, stir the material in the sample container. Transfer at least 11g and up to 16g of the remaining sample to the tared weighing dish.

9.2.15 Weigh the sample and dish, recording to the nearest 0.01g.

9.2.16 Place the samples to dry in the oven overnight at 103-105°C. Check that the oven temperature is recorded on the Electronic Prep Log bench sheet and is within required specifications before placing samples into oven as per Pace SOP ENV-SOP-GBAY-0115 *Support Equipment* (current revision or replacement).

9.2.17 Overnight is a period of time ≥ 8 hours.

9.2.18 Record the oven temperature prior to removal of the samples and verify it is still within the required specifications of 103-105°C. Remove the sample from the oven and allow to cool for 1 minute. If the sample has dried less than 8 hours, it must be placed 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.

9.2.19 After the sample has cooled, weigh the dried residue to the nearest 0.01g. If the sample has been oven dried for at least 8 hours, proceed to section 9.2.21. If dried less than 8 hours, proceed to the next section.

9.2.20 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 9.2.21. If the weight has changed by more than 0.01g or 0.1%, repeat step 9.2.16 until a constant weight (<0.01g or 0.1% change) is achieved.

9.2.21 As weights are entered, Percent Difference and Weight Differences will be calculated when using multiple weights.

9.2.22 You can manually select a weight to use by entering an "M" in the Use test box to override the automatic weight chosen. If you want to manually de-select a weight, enter an "m". The weights are chosen using weight differences being less than 5mg.

9.2.23 Once all the weights have been taken and additional information has been entered your results will be calculated and you will be ready to auto post the data into EpicPro.

9.2.24 Save your data by pressing the disk icon next to the search template button.

9.2.25 If required, you may print or create a PDF of your data by selecting Menu -> Print Landscape.

9.2.26 Verify all the samples you wish to auto post have a Y for select, then press the auto post Button.

9.2.27 Select a few samples randomly and verify that the % Moisture result is being calculated correctly following Section 10 for the calculation.

9.2.28 Once data is auto posted, review for precision. See Section 10.2 for the RPD Calculation.

10.0 Data Analysis And Calculations

10.1 **% Moisture is calculated and in the Electronic Prep Log worksheet using the following equation.**

$$\% \text{ MOISTURE} = (W_W - W_D) * 100\% / W_W$$

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)

10.2 **Relative percent difference (RPD) is calculated as follows:**

$$\%RPD = (S1-S2) * 100\% / ((S1+S2)/2)$$

Where: S1=%Moisture for Sample

S2=%Moisture for Sample Duplicate

11.0 Quality Control and Method Performance

11.1 **Quality Control**

11.1.1 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.

11.1.2 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 affected samples must be re-analyzed.

11.2 **Method Performance**

11.2.1 All applicable personnel must read and understand this SOP with documentation of SOP reviewed maintained in their training files. Additionally, staff must read and understand the Pace SOP: ENV-SOP-GBAY-0121 *Regulated Soil Handling* (most recent revision or replacement) in addition to receiving Regulated Soil Training upon hire and annually thereafter.

11.3 **Analyst Qualifications and Training**

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going

DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-GBAY-0094 *Orientation and Training Procedures* (most recent revision or replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-GBAY-0120 *Data Review and Final Report Processes* (most recent revision or replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

Procedures for handling waste generated during this analysis are addressed in ENV-SOP-GBAY-0125, *Waste Handling and Management* (current revision or replacement).

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (current revision) for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 **Method Modifications for ASTM D2974-87 are as follows:**

14.1.1 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.

14.1.2 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.

14.1.3 Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace Laboratories.

14.1.4 ASTM D2974-87 states to use 50 g of the test specimen; Pace Analytical Services, LLC – Green Bay uses an 11 to 16 g aliquot.

14.1.5 ASTM D2974-87 states to dry the sample for 16 hours, Pace Analytical Services, LLC – Green Bay defines the drying time as overnight, which is a drying time of ≥8 hours. ASTM D2974-87 states to cool the samples in a desiccator. Pace – Green Bay has compiled a study in 2017 demonstrating the equivalency of sample results when cooled in a desiccator versus cooling on the counter when samples have been dried overnight (≥8 hours).

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Not Applicable to this SOP.

17.0 REFERENCES

17.1 Pace Quality Assurance Manual (most current revision or replacement).

17.2 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (current revision or replacement).

17.3 ASTM D 2974-87 Test Method A "Standard Test Methods for moisture, Ash and Organic Matter of Peat and Other Organic Soils", American Society of Testing and Materials, Reapproved 1995.

17.4 EPA Contract Laboratory Program SOW for Inorganic Analysis Doc. ILM 1.03 March 1990.

17.5 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.

18.0 REVISION HISTORY

This Version: ENV-SOP-GBAY-0004-Rev.03

Section	Description of Change
All	Transferred to new format
7.1	Additional balance added to equipment list.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-GBAY-0004	Percent Moisture in Solids	Rev.01
ENV-SOP-GBAY-0004	Percent Moisture in Solids	Rev.02

Pace Analytical®

ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-GBAY-0063 **Revision:** 01

Document Title: Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by 8082

Department(s): SVOA

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Signature Manifest

Document Number: ENV-SOP-GBAY-0063 **Revision:** 01

Title: Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by 8082

All dates and times are in Central Time Zone.

ENV-SOP-GBAY-0063-Rev.01 PCB_8082_608

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Kate Verbeten (007119)	Manager - Quality	08 Oct 2020, 11:32:58 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Christopher Haase (007121)	Manager	08 Oct 2020, 12:08:53 PM	Approved
Nils Melberg (007142)	General Manager 2	08 Oct 2020, 12:12:01 PM	Approved

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of concentrations of polychlorinated biphenyls (PCBs) in extracts in accordance with SW846 Method 8082 and EPA 608. Samples for analysis are prepared by SW846 Method(s) 3510C, 3540C, 3541, and 3580A.

1.1 Target Analyte List and Limits of Quantitation (LOQ)[1]

Aroclor	CAS #	Aqueous (µg/L)	Solid (µg/Kg)	Biota (µg/Kg)
AR1016	12674-11-2	0.5	50	25
AR1221	11104-28-2	0.5	50	25
AR1232	11141-16-5	0.5	50	25
AR1242	53469-21-9	0.5	50	25
AR1248	12672-29-6	0.5	50	25
AR1254	11097-69-1	0.5	50	25
AR1260	11096-82-5	0.5	50	25
AR1262[2]	37324-23-5	0.5	50	25
AR1268[2]	11100-14-4	0.5	50	25
Total PCB	NA	0.5	50	25

1. Values in place as of effective date of this SOP. LOQ are subject to change. For the most up to date LOQ, refer to the LIMS or contact the laboratory.
2. Aroclor(s) 1262 and 1268 only analyzed per client request.

1.2 Applicable Matrices

1.2.1 This method is applicable to most water, solid, sediment, waste and biological 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.

1.3 Personnel

1.3.1 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.

2.0 SUMMARY OF METHOD

2.1 Sample extracts are prepared for analysis by an appropriate sample preparation method. 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.

2.2 Retention time windows, in combination with characteristic elution patterns from a dual-column analysis, are used in the identification of PCBs as Aroclors.

2.3 PCBs are quantified as Aroclor mixtures by comparison of their ECD response on a primary column with a calibration curve(s) constructed from the response(s) of authentic standards.

2.4 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.

3.0 INTERFERENCES

3.1 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.

3.2 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 copper granules or similar procedure described in a separate SOP.

3.3 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.

3.4 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.

4.0 DEFINITIONS

4.1 Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

4.2 **Extract:** A solution of contaminants extracted and concentrated from a sample.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's Quality Manual.

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory SOP ENV-SOP-GBAY-0007 *Bottle Preparation* (most recent revision or replacement).

6.1 The lab provides appropriate bottle ware, including preservative, for requested testing. Where applicable, the bottle ware is demonstrated to be free of target analytes. When bottle ware not originating from the lab is used, the data may be qualified.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	1L amber glass	1 L	Thermal: ≤6° Celsius Chemical: NA	Collection to Extraction: 365 days
Soil/Solid (non-aqueous)	One 8oz wide glass jar or zip-top bag	30g	Thermal: ≤6° Celsius Chemical: NA	Collection to Extraction: 365 days
Biological Tissue	8oz wide glass jar, zip-top bag, aluminum foil	90g	Thermal: ≤6° Celsius (shipped) Thermal: ≤-10° Celsius (lab storage) Chemical: NA	365 days or longer per client request prior to extraction, typical extraction hold time do not apply.
Extracts	2mL amber glass vial	NA	Thermal: ≤6° Celsius	365 days

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (most recent revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, aqueous, solid and leachate samples are stored at ≤6°C until sample preparation; biological tissue samples are stored at ≤-10°C. Prepared samples (extracts, digestates, distillates, other) are stored at ≤6°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Analytical Instrument/Peripherals	EPIC Pro Name
HP 6890 GC	40GCS7
Dual Electron Capture Detector (ECD)	40GCS7
HP 7683 AutoSampler Tray	40GCS7
HP 7683 Injector	40GCS7
Chemstation	40GCS7
HP 6890 GC	40GCS9
Dual Electron Capture Detector (ECD)	40GCS9
HP 7683 AutoSampler Tray	40GCS9
HP 7683 Injector	40GCS9
Chemstation	40GCS9
HP 6890 GC	40GCSB
Dual Electron Capture Detector (ECD)	40GCSB
HP 7683 AutoSampler Tray	40GCSB
HP 7683 Injector	40GCSB
Chemstation	40GCSB
HP 6890 GC	40GCSC
Dual Electron Capture Detector (ECD)	40GCSC
HP 7683 AutoSampler Tray	40GCSC
HP 7683 Injector	40GCSC
Chemstation	40GCSC

*Or equivalent

7.2 Chromatography Supplies

Item*	Vendor*	Model / ID*	Catalog #*	Description
Primary Analytical Column	Restek	Rtx-PesticideCLPesticides	11139	30m, 0.32 mm ID 0.5 µm df
Confirmation Column	Restek	Rtx-CLP Pesticide2	11324	30m, 0.32 mm ID 0.25 µm df
Guard Column	Restek	I.P. Deactivated Guard Column	10045	5m, 0.53 mm ID
Fluorocarbon O-rings	Supelco	Thermo O-ring seal	21004-U	¼"
Vespel/Graphite Ferrules	Restek	0.5mm 0.8mm	20231 20230	1/16" X 0.5 mm ID 1/16" X 0.8 mm ID
Cyclo Uniliner	Restek	NA	22271	4 mm x 6.3 x 78.5 for Agilent GCs
Y Splitter	Restek	Universal angled Y	20404	Press-tight connector
Inlet Seals	Restek	Dual Vespel Ring	212389	Stainless Steel

*Or equivalent

7.3 General Supplies

Supply*	Vendor*	Model / ID*	Catalog#	Description
Gastight Syringes	Fisher	10-µL 25-µL 50-µL 100-µL 250-µL 500-µL 1,000-µL	14-815-1 14-815-29 14-824-30 14-684-100 14-684-102 13-684-106 14-824-25	Hamilton Gastight Syringes
Glass Storage Vials	Fisher	B7921VO	03-377-38	20mL EPA Amber glass
Caps	Fisher	B718524	03-391-12F	PTFE Lined 24-400
Glass Autosampler Vials	MG Scientific	2.0mL	V300-51	Clear Glass
Autosampler vial crimp cap	Fisher	11mm crimp seal	06-406-19B	PTFE lined
Volumetric Flask	Fisher	10mL 50mL 100mL 200mL	20-812D 10-210B 10-210C 10-210D	Class A
Disposable Pasteur pipettes	MG Scientific	5 ¾" 9"	P200-1 P200-2	Glass

*Or equivalent

8.0 Reagents And Standards

8.1 Reagents

Reagent/Standard*	Concentration/ Description*	Manufacturer/Vendor/Item #*	Expiration Date
Hexane	Pesticide Grade or equivalent	Burdick & Jackson / MG Scientific / B&J-217-4	Manufacturer's recommended expiration or 2 years from receipt, whichever is sooner
Acetone	Pesticide Grade or equivalent	Burdick and Jackson / MG Scientific / B&J-010-4	
Nitrogen Gas	99.9999% / Ultra High Purity	Airgas	NA
Helium Gas	99.9999% / Ultra High Purity	Airgas	NA

*Or Equivalent

8.2 Analytical Standards: Prepared from stock standard solutions and are required for initial calibration and continuing calibration checks (Table 8.2.4). 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.

8.2.4 Standard Stock Solutions:

Standard	Concentration	Manufacturer	Catalog #	Expiration Date
Pesticide Surrogate Mix	200µg/mL each in Acetone	Restek Corporation or equivalent	32000	Manufacturer's recommended expiration date for unopened ampulated standards. 1 year after ampule is opened or on expiration date, whichever is sooner.
Aroclor 1016 Mix	1000µg/mL in Hexane		32006	
Aroclor 1221 Mix	1000µg/mL in Hexane		32007	
Aroclor 1232 Mix	1000µg/mL in Hexane		32008	
Aroclor 1242 Mix	1000µg/mL in Hexane		32009	
Aroclor 1248 Mix	1000µg/mL in Hexane		32010	
Aroclor 1254 Mix	1000µg/mL in Hexane		32011	
Aroclor 1260 Mix	1000µg/mL in Hexane		32012	
Aroclor 1262 Mix*	1000µg/mL in Hexane		32409	
Aroclor 1268 Mix*	1000µg/mL in Hexane		32410	
Aroclor 1016	1000µg/mL in Isooctane	Supelco or equivalent	4-8097	
Aroclor 1260	1000µg/mL in Isooctane		4-4809	
*Aroclor(s) 1262 and 1268 only analyzed upon client request				

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 8.5. All standards are prepared in hexane and stored in amber vials with PTFE-lined screw caps at ≤6°C.

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.

8.5 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 or the expiration date listed for the stock source, whichever is sooner.
AR1221 Stock Solution	Aroclor 1221 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1232 Stock Solution	Aroclor 1232 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1242 Stock Solution	Aroclor 1242 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1248 Stock Solution	Aroclor 1248 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1254 Stock Solution	Aroclor 1254 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1262 Stock Solution*	Aroclor 1262 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1268 Stock Solution*	Aroclor 1268 Mix	1000µL	10mL of Hexane	100µg/mL	
AR1660 Stock Solution	Aroclor 1016 Mix Aroclor 1260 Mix	1000µL each	10mL of Hexane	100µg/mL each	
AR1660 ICV Stock Solution	Aroclor 1016 Aroclor 1260	1000µL each	10mL of Hexane	100µg/mL each	
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 months from date of preparation or the expiration date listed for the stock source, whichever is sooner.
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	
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	
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	
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	
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	
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	

Analytical Standard	Standard or Stock Solution Used	Volume of Standard or Stock Used	Final Volume & Solvent Used	Final Concentration	Expiration Date
AR1660-1 Calibration Standard and PRLS	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 50µL TCMX/DCB 100µL	100mL of Hexane	AR1660 0.05µg/mL TCMX/DCB 0.01µg/mL	6 months from date of preparation or the expiration date listed for the stock source, whichever is sooner.
AR1660-2 Calibration Standard	AR1660 Stock Solution TCMX/DCB Stock Solution	AR1660 200µL TCMX/DCB 200µL	100mL of Hexane	AR1660 0.2µg/mL TCMX/DCB 0.02µg/mL	
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	
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	
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	
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	

*Aroclor(s) 1262 and 1268 only analyzed upon client request

8.5.1 Standards will have a label attached to the bottle identifying the following (due to the limited amount of space on some standard vials (i.e., 2 mL autosampler vials), an EpicPro standard label will be attached which does not contain all the following):

8.5.1.1 Name of Solution

8.5.1.2 Pace, LLC. Standard ID Number

8.5.1.3 Pace, LLC. Lab Lot ID (for stock standards and reagents)

8.5.1.4 Preparation Date

8.5.1.5 Preparer's Initials

8.5.1.6 Concentration

8.5.1.7 Expiration Date

9.0 PROCEDURE

9.1 Operating Parameters: Configure the GC/ECD 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.

Instrument Operating Parameters:

GC Column Conditions*	
Carrier Gas	UHP Helium
Carrier Gas Flow Rate:	3.4mL/min
Make-up Gas	UHP Nitrogen
Make-up Gas Flow Rate:	35.0mL/min
Detector Temperature:	300°C
Injector Temperature:	205°C
Injection:	Splitless
GC Temperature Program*	
Initial Temperature	110°C
Initial Time:	0.5 min
Rate 1 :	30.00°C/min
Final Temperature 1	200°C
Final Time 1:	1.00 min
Rate 2:	12.00°C/min
Final Temperature 2:	220°C
Final Time 2:	0.00 min
Rate 3:	30.00°C/min
Final Temperature 3:	305°C
Final Time 3:	6.00 min

*Or Equivalent

9.2 Calibration and Standardization

9.2.1 Initial Calibration:

9.2.1.1 **Analysis of Standards:** The initial calibration includes analysis of a five-point calibration curve of AR1660 at concentrations of 0.05, 0.2, 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, 1262, and 1268 at 0.5µg/mL.

9.2.1.1.1 Other calibration ranges may be substituted to meet expected concentrations of samples to be analyzed. If historical data indicates a specific Aroclor is present (or by client request) a five-point initial calibration may be performed for the Aroclor of concern instead of using the AR1660 mixture. Please see Section 8.5 on instructions of the preparation of Aroclor standards referencing the 5-point calibration for AR 1660.

9.2.1.1.2 Three to ten (preferably seven) peaks must be selected for each Aroclor, except for Aroclor 1221 which only requires a minimum of three peaks. 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.

9.2.1.1.3 The typical batch for initial calibration should include:

Series of 2-3 Primes
Solvent Blank (Hexane)
AR1660-1 (0.05µg/mL)
AR1660-2 (0.2µg/mL)
AR1660-3 (0.5µg/mL)
AR1660-4 (0.8µg/mL)
AR1660-5 (1.0µg/mL)
Solvent Blank (Hexane)
AR1660-3 ICV
AR1660-1 (0.05µg/mL) (CRDL)

9.2.1.1.4 Load Autosampler: Load the autosampler with the appropriate primes, solvent blanks, standards and samples for the batch as it was created.

9.2.1.1.5 Analyze Samples: Analyze all standards.

9.2.1.2 **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.

9.2.1.2.1 Make at least three injections of all analytes of interest over a 72-hour period.

9.2.1.2.2 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.

9.2.1.2.3 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.

9.2.1.2.4 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.

9.2.1.2.5 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 Aroclors. 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 Aroclor 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 Aroclor and does not correspond to a peak from a DDT analog.

9.2.1.3 **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 the Analyte Response.
C_x = Concentration of the analyte in the solution (µg/mL)

9.2.1.4 **SW846 8082 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.**

9.2.1.5 **EPA 608 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 ≤ 10%. If this is the case, linearity can be assumed, and the average RF can be used for quantitation. If the %RSD is >10%, a linear calibration curve may be used if the correlation coefficient is ≥ 0.99. **The results for both columns must meet calibration acceptance criteria.**

9.2.1.6 **Initial Calibration Verification (ICV):** In order to consider the initial calibration acceptable, an ICV standard must be analyzed. For every five-point Initial Calibration, a standard corresponding ICV must also 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.

9.2.2 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.

9.2.2.1 **SW846 8082 Acceptance Criteria:** The percent difference (%D) is determined for every analyte and must be within ±15% 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

9.2.2.1.1 First determine whether the average %D for all of the peaks for each specific Aroclor with a five-point calibration is ≤ 15%. Each individual Aroclor must be evaluated separately. For example, the average %D for all of the peaks used for quantitation of AR1016 must be ≤ 15%. If the Aroclors themselves are acceptable, evaluate the %D for each surrogate. If the %D is ≤ 15 for each individual Aroclor and surrogate, the continuing meets the acceptance criteria.

9.2.2.1.2 If the ending calibration verification standard exceeds 15%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.

9.2.2.2 **EPA 608 Acceptance Criteria:** The percent difference (%D) is determined for every analyte and must be within ±15% of the calibration curve. Calculate percent recovery for each peak using the following equation:

$$\%Recovery = \frac{Observed\ Concentration}{Theoretical\ Concentration} X 100$$

9.2.2.2.1 If the response factor (area/concentration) of the check standard deviates by more than 15% from the initial average response factor, the calibration is considered out of control and analysis must be stopped. If the Aroclors themselves are acceptable, evaluate each surrogate to determine if their recovery is within 15% of the predicted response.

9.2.2.2.2 If the ending calibration verification standard exceeds the 15% recovery 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.

9.2.2.3 **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.

9.2.3 Lower Limit of Quantification Standard (LLOQ): For every five-point Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) must also be analyzed. The LLOQ 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.

9.2.4 See Attachment I: Calibration Acceptance and Verification Criteria and laboratory SOP: ENV-SOP-GBAY-0138, *Calibration Procedures* (most recent revision or replacement) for additional guidance.

9.3 Sample Preparation

9.3.1 Biological Tissue Samples: Tissue samples are prepared according to EPA 3540C and 3541. These procedures are contained in separate standard operating procedures. Refer to SOP number ENV-SOP-GBAY-0066 *Extraction of Biological Samples for Organochlorine Pesticides/PCBs by SW846 3540C* and ENV-SOP-GBAY-0082 *Extraction of PCBs in Tissue Using the Automated Soxhlet* (most recent revision or replacement) for details on the preparation of biological tissue samples.

9.3.2 Soil Samples: Solid samples are prepared according to EPA 3541. These procedures are contained in a separate standard operating procedure. Refer to SOP number ENV-SOP-GBAY-0075 *Extraction of PCBs Using the Automated Soxhlet* (most recent revision or replacement) for details on the preparation of soil or solid samples.

9.3.3 Water Samples: Aqueous samples are prepared according to EPA 3510C. These procedures are contained in a separate standard operating procedure. Refer to SOP number ENV-SOP-GBAY-0083 *Separatory Funnel Extraction of Water Samples for Semivolatile* (most recent revision or replacement) for details on the preparation of aqueous samples.

9.3.4 Wipes, Air Monitoring Wipes, and Oil Samples: Gauze wipes, air monitoring wipes, and oil samples are prepared according to EPA 3580A. These procedures are contained in a separate standard operating procedure. Refer to SOP number ENV-SOP-GBAY-0074 *Extraction of Wipe Samples for PCBs* (most recent revision or replacement) for details on the preparation of wipe and oil samples.

9.4 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.

9.5 Sample Analysis:

9.5.1 Batch Sequence: Generate a sequence to run a batch of samples and the associated quality control samples.

9.5.2 The typical batch for sample analysis should include the following. Preparation of Method Blank, LCS, MS, MSD, and Duplicate sample extracts is described in the appropriate sample preparation SOP.

AR1660 CCV (0.5µg/mL)
20 samples or 12-hour period
Method Blank
Laboratory Control Sample
Samples
Matrix Spike
Matrix Spike Duplicate
AR1660 CCV (0.5µg/mL)

9.5.3 Load the Autosampler with standards and samples for the batch created above.

9.5.4 Analyze all standards, quality control samples, and environmental samples.

9.5.4.1 The method blank and LCS extracted along with the samples should be analyzed on the same instrument as the samples.

9.5.4.2 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.

9.5.5 Process all runs with Target software.

9.5.6 View sample chromatograms and verify analyte identifications (Section 10).

9.5.7 Post data to EPIC Pro.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 Data Reduction

10.1.1 Quantitative Analysis (Primary Column): PCB Aroclor results will be quantitated and reported from the primary column Rtx-CLPesticide column. The peaks used for Aroclor identification are labeled as the specific Aroclor Chromatograms. Please see Attachment III: PCB Aroclor Pattern and Peak Selection Chromatograms.

10.1.1.1 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 either the single-point, or one of the five-point calibration standards for the corresponding Aroclor with the peaks observed in the sample extract (Please see Attachment III for example chromatograms of peaks chosen). An overlay of the standard chromatogram onto the sample chromatogram may be required to clearly identify patterns and determine peak overlap between Aroclors.

10.1.1.2 Additionally, environmental "weathering" of PCBs may complicate reliable identification and quantitation. Alternate peaks may be chosen due to weathering or other known interferences. In severely degraded patterns in unknown samples, the analyst would choose the Aroclor standard pattern which most closely resembles the unknown sample.

10.1.1.3 Once the sample is processed in the Target processing software, the analyst will review the chromatograms to first determine Aroclor pattern matches. Then they will manually integrate the chromatogram using baseline adjustments, peak splitting and peak assignment to mimic the standard Aroclor pattern used for calibration.

10.1.1.4 Since the chromatograms for many Aroclor mixtures overlap, the presence of multiple mixtures may complicate their quantitation. Peaks that exhibit high bias due to Aroclor overlap or matrix interferences may be removed from quantitated result by the analyst. It will be left to analyst's discretion to use their experience in data analysis to determine when peak removal is acceptable. A minimum of 3 peaks must be used to quantitate unknown samples and standards.

10.1.1.5 Aroclors 1232, 1016, 1242 and 1248 will not be identified in the same unknown sample due to the PCB congener sharing which occurs within these Aroclor patterns. Therefore, the laboratory does not bias the PCB Total data high due to PCB Aroclor overlap that would occur if these were to be reported on the same unknown sample.

10.1.2 Qualitative Analysis: Reported PCB Aroclor results will be confirmed by Aroclor pattern presence on the Rtx-CLPesticide2 column as follows.

10.1.2.1 Qualitative confirmation is completed using a second GC column of dissimilar stationary phase. Dual-column analysis is performed for qualitative confirmation only, however the same initial and continuing calibration criteria apply to both columns as outlined in Section 9.2.1.4.

10.1.2.2 Since Aroclors provide distinct multiple peak patterns which may be identified by an experienced analyst, confirmation on the second column may be based on pattern recognition. No manual integrations such as peak splitting, baseline drawing and peak assigning will be performed on the samples for confirmation. These chromatograms will not be evaluated for Aroclor overlap, matrix interferences or RPD between the two columns. The sample concentration and surrogates will not be evaluated, and sample dilutions will not be determined from the raw results. Only laboratory quality control standards are subject to manual manipulation.

10.1.2.3 **Manual Integration**

10.1.2.3.1 Manual changes to automated integration are called manual integration. Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are performed consistently and are ethically justified, all manual integrations must be performed, reviewed, and recorded in accordance with corporate SOP ENV-SOP-CORQ-0006, *Manual Integration* (most recent revision or replacement).

10.2 Raw Data Results:

10.2.1 The amount of Aroclor is calculated using the individual response factor (single point) for each of the 3 - 10 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 concentrations are averaged to determine the on-column concentration of that Aroclor based on the primary column.

10.2.2 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.

10.3 Data Calculations:

10.3.1 Results Calculation- Water and Water-Miscible Samples:

$$\text{Final Concentration } (\mu g/L) = \frac{(C_x)(DF)(Uf)(Vt)}{(Vi)(Vo)}$$

Where:
Cx = On-column concentration in extract (µg/mL).
DF = Dilution factor.
Uf = Correction factor.
Vt = Volume of final extract (µL).
Vi = Volume injected (µL).
Vo = Volume of water sample extracted (mL)

10.3.2 Results Calculation- Soil/Solid and Tissue Samples:

$$\text{Final Concentration } (\mu g/Kg) = \frac{(C_x)(DF)(Uf)(Vt)}{(Vi)(Ws)(S)}$$

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)

10.3.3 Results Calculation- Air Monitoring Samples calculations use a time/volume relationship to calculate PCB concentration. (See Attachment II for example.):

$$\text{Concentration } (\mu g/L) \text{ or } (mg/m3) = \frac{(C_x)(DF)(U_f)(V_t)}{(V_i)(V_s)}$$

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).
V_s = Volume of air sampled (L).
S = Percent Solids (biological samples not corrected for percent solids).

Volume of air sampled L = LPM * T
Where:
LPM = Liters per Minute of air sampled.
T = Sampling time in minutes.

10.3.4 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. See current revision or replacement of SOP ENV-SOP-GBAY-0153, *Laboratory Calculations* for additional information.

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control:

11.1.1 Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per 20 samples	Target analytes must be less than the LLOQ (LOD for WI projects). 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.	Qualify results and/or re-extract associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample with appropriate qualifier indicating blank contamination; If sample result <10x MB detects, and sample cannot be re-extracted, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	1 of the following: AR1242 AR1248 AR1254 AR1260 AR1660 (as directed by state or client specific requirements)	One per batch of up to 20 samples	Laboratory derived limits or Project Specific	At analyst discretion, re-analyze the LCS to verify failure; If LCS passes, review samples for potential injection problems; If problem persists, check spike solution; Re-extract samples where possible. ***Exceptions:*** If LCS recovery is > 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)	1 of the following: AR1242 AR1248 AR1254 AR1260 AR1660 (as directed by state or client specific requirements)	EPA 8082: One per batch of up to 20 samples EPA 608: One per batch of up to 10 samples	Laboratory derived limits or Project Specific	If LCS and MBs are acceptable, the MS/MSD chromatogram(s) should be reviewed and may be reported with appropriate footnote indicating matrix interferences Note: If insufficient volume was obtained for an MS/MSD an LCSD may be extracted and the batch reported with the "M5" footnote.
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup	EPA 8082: One per batch of up to 20 samples EPA 608: One per batch of up to 10 samples	Laboratory Derived Limits or Project Specific	If results are not acceptable, check for possible sample preparation problems and re-analyze if needed. Report results with an appropriate footnote.

11.1.2 Sample Quality Control Criteria.

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Surrogate Standards	TCMX DCB	Added to all samples, spikes, control samples and method blanks prior to analysis	Laboratory derived limits	1 surrogate is allowed to be outside of recovery limits before action is taken. Assess impact of sample matrix. In the absence of obvious matrix interference (high background, extremely dark extract), re-extract sample. 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. If **both** surrogate recoveries fail this criterion, re-extraction of the sample may be necessary. ***Exceptions:*** Surrogate recovery above criteria and target compounds < RL, result may be reported with appropriate footnote. Surrogate recovery out of control due to obvious sample matrix interference (i.e. co-elution), report results with appropriate footnote.

11.2 Method Validation

11.2.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most recent revision or replacement) and to the laboratory's SOP ENV-SOP-GBAY-0106 *Determination of LOD and LOQ* (most recent revision or replacement) for these procedures.

The LOD and LOQ are always adjusted to account for actual amounts used and for dilution.

Current LOD and LOQ can be found in the Laboratory Information Management System (LIMS) - EpicPro.

Level of Detection (LOD): The LOD is determined by the 40CFR Part 136B MDL study. Once the 40CFR Part 136B MDL is determined it may be elevated if deemed unrealistic as demonstrated using method blank evaluations.

11.2.2 Periodic performance evaluation (PE) samples

Periodic performance evaluation (PE) samples are analyzed per ENV-SOP-GBAY-0107, *PE/PT Program* (most recent revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

11.3 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-GBAY-0094 *Orientation and Training Procedures* (most recent revision or replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employees complete tasks and review their own work are called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-GBAY-0120 *Data Review and Final Report Processes* (most recent revision or replacement) for specific instructions and requirements for each step of the data review process.

Draw a single-line strikethrough for any unacceptable or changed data, then DATE and INITIAL and provide a written explanation of the reason for the change.

Data are validated and peer reviewed by lab personnel using a batch cover sheet attached to the raw data and filed.

Any discrepancies and issues occurring with each batch should be included on the cover sheet to be incorporated into a narrative.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Calibration Linearity and Second Source Verification Problems:

- Check instrumentation/equipment condition. Document instrument maintenance in the logbook.
- Perform another initial calibration.
- No data can be reported.
- Generate Non-Conformance Memo.

Continuing Calibration Verification Problems:

- Reanalyze the original CCV standard to determine instrument consistency.
- Prepare and analyze a new CCV standard to determine preparation consistency/standard integrity.
- Document instrument maintenance.
- Reanalyze CCV standard to determine if maintenance was effective in restoring performance.
- 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.

For additional troubleshooting or maintenance requirements please refer to the instrument operations manual or the Pace SOP ENV-SOP-GBAY-0098, *Preventative, Routine, and Non-routine Maintenance* (current revision or replacement).

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

For further information on waste management, consult ENV-SOP-GBAY-0125 *Waste Handling and Management* (most recent revision or replacement).

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most recent revision or replacement) for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

If a client fails to provide the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the "M5" data qualifier.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Attachment I: Calibration Acceptance and Verification Criteria

Attachment II: Tailing Factor Calculation

Attachment III: PCB Aroclor Patterns and Peak Selection

17.0 REFERENCES

17.1 Pace Analytical Services, LLC – Green Bay, WI Quality Assurance Manual- current version.

17.2 TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-VI-2016-Rev.2.1.

17.3 EPA Method SW-846 8082, Revision 0, *Polychlorinated Biphenyls (PCBs) by Gas Chromatography*, December 1996.

17.4 USEPA, SW-846, Method 8000D, *Determinative Chromatographic Separations*, March 2018.

17.5 Appendix A to part 136, Methods for organic chemical analysis of municipal and industrial wastewater, "Method 608 – Organochlorine pesticides and PCBs".

17.6 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0083, *Separatory Funnel Extraction*, current revision or replacement.

17.7 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0066, *Extraction of Biological Samples for Organochlorine Pesticides/PCBs*, current revision or replacement.

17.8 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0082, *Extraction of PCBs Using the Automated Soxhlet*, current revision or replacement.

17.9 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0074, *Extraction of Wipes and Oil for PCB Analysis*, current revision or replacement.

17.10 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0075, *Extraction of PCBs Using the Automated Soxhlet*, current revision or replacement.

17.11 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0067, *Gel Permeation Chromatography*, current revision or replacement.

17.12 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0069, *Sulfuric Acid Cleanup*, current revision or replacement.

17.13 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0070, *Florisil Cleanup for PCBs*, current revision or replacement.

17.14 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0072, *Silica Gel Cleanup of Organochlorine Pesticides and PCBs*, current revision or replacement.

17.15 Pace Analytical Services, LLC – Green Bay, SOP ENV-SOP-GBAY-0073, *Copper Cleanup for the Removal of Sulfur from PCB Samples*, current revision or replacement.

18.0 REVISION HISTORY

This Version: ENV-SOP-GBAY-0063-Rev.01

Section	Description of Change
ALL	Updated to Corporate SOP Template

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-GBAY-0063	Analysis of PCBs by GC/ECD by SW846 8082 and 608	00

Attachment I: Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor Linear Regression	%RSD ≤ 20% r ≥ 0.99	If not met, try linear regression fit If not met, remake standards and recalibrate
LLOQ Verification	Calibration points will be rerun against calibration curve once the calibration is completed and monthly thereafter	Low point 60-140% All other points ±30%	Analytes in the low-level calibration point that do not meet these criteria should be evaluated for instrument issues. For analytes that recover below 40% the data should be flagged.
Second Source Verification Standard	Immediately after each initial calibration	% D ±15%	Acceptance criteria are ±15% difference.
Continuing Calibration Verification	At the beginning of every 12-hour shift, every 20 injections, or more frequent as required	%D ± 15% Result ± 15% Drift	Use for Avg RF calibration curves Use for linear 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.

Attachment II: Air Monitoring Calculation Sheet Example

Pace Analytical®	Document Name: NIOSH Method 5503 (mod) PCBs in Air	Document Revised: 5-May-2020
	Document No.: ENV-FRM-GBAY-0209-Rev.00	Author: Pace Green Bay Quality Office

PCBs in Air

Client: Tetra Tech
Date/Time Collected:
Project number:
Project name: FOX RIVER
Pace Project #:

Method: NIOSH 5503 (modified)
Date/Time Extracted:
LPM:
Time (minutes):
Volume of Air sampled (L): 0.00

		Pace Sample ID:	(Front)	Pace Sample ID:	(Back)	
Client Sample ID	**Test Parameter**	**Result in ug**	**results in ug/L or mg/m3**	**Result in ug**	**results in ug/L or mg/m3**	**<10% Breakthrough front to back sections**
		Date Analyzed: Analyst:		Date Analyzed: Analyst:		
	PCB-1016 (Aroclor 1016)	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	PCB-1221* (Aroclor 1221)	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	PCB-1232 (Aroclor 1232)	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	PCB-1242 (Aroclor 1242)	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	PCB-1248 (Aroclor 1248)	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	PCB-1254 (Aroclor 1254)	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	PCB-1260 (Aroclor 1260)	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	PCB-1268 (Aroclor 1268)	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	PCB Total	< 0.05	< #DIV/0!	< 0.05	< #DIV/0!	#DIV/0!
	Tetrachloro-m-xylene (S)		58-120%*		58-120%*	
	Decachlorobiphenyl (S)		59-109%*		59-109%*	

* acceptable range for surrogate recovery

Pace Analytical®	Document Name: NIOSH Method 5503 (mod) PCBs in Air	Document Revised: 5-May-2020
	Document No.: ENV-FRM-GBAY-0209-Rev.00	Author: Pace Green Bay Quality Office

Method Blank Results

Method Blank ID	Test Parameter	Result in ug	Percent Recovery Limit
Date Analyzed: Analyst:			
	PCB-1016 (Aroclor 1016)	< 0.05	
	PCB-1221 (Aroclor 1221)	< 0.05	
	PCB-1232 (Aroclor 1232)	< 0.05	
	PCB-1242 (Aroclor 1242)	< 0.05	
	PCB-1248 (Aroclor 1248)	< 0.05	
	PCB-1254 (Aroclor 1254)	< 0.05	
	PCB-1260 (Aroclor 1260)	< 0.05	
	PCB-1268 (Aroclor 1268)	< 0.05	
	PCB Total	< 0.05	
	Tetrachloro-m-xylene (S)		58-120%*
	Decachlorobiphenyl (S)		59-109%*

* acceptable range for surrogate recovery

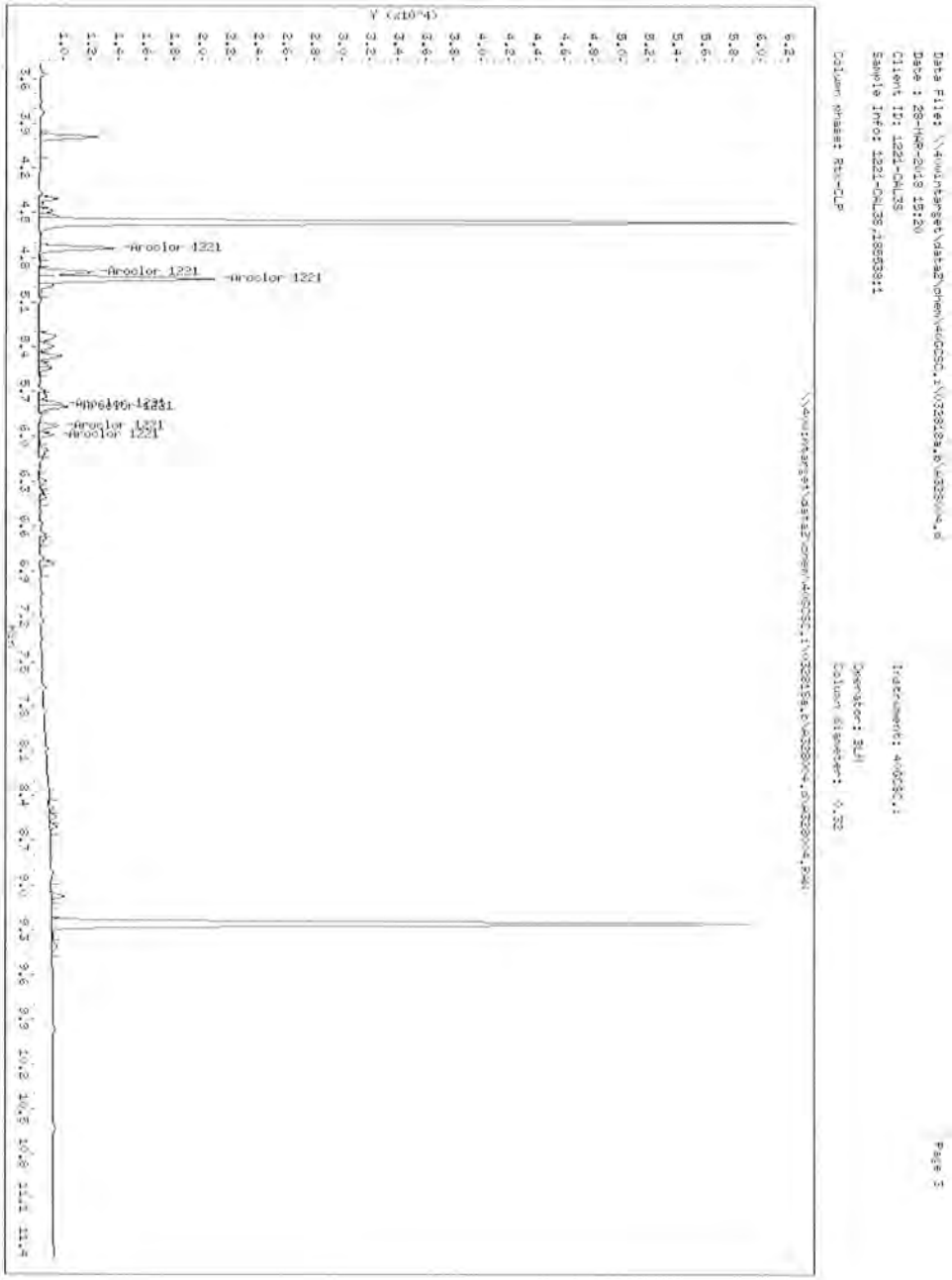
One Method Blank sample analyzed per preparation batch

Secondary Data Review: _____ Date: _____

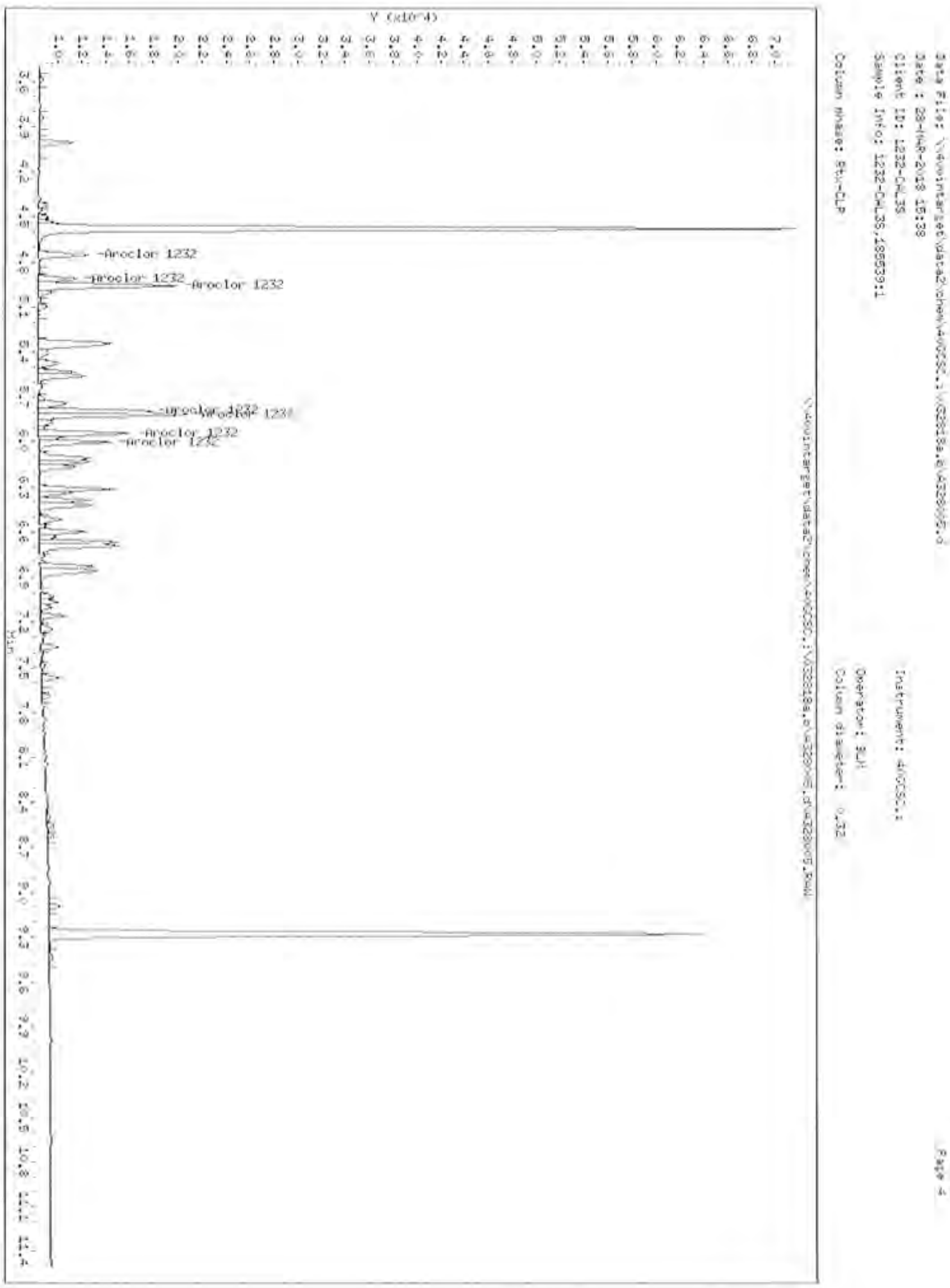
Wipe MDL = 0.02212 µg/mL.
Volume of air sample = 0.1423LPM * 481M = 68.45L
Final volume of extract = 2mL
Result in µg = On column or MDL * final volume of extract (0.02212 * 2) = 0.04424 µg

Attachment III: PCB Aroclor Patterns and Peak Selection

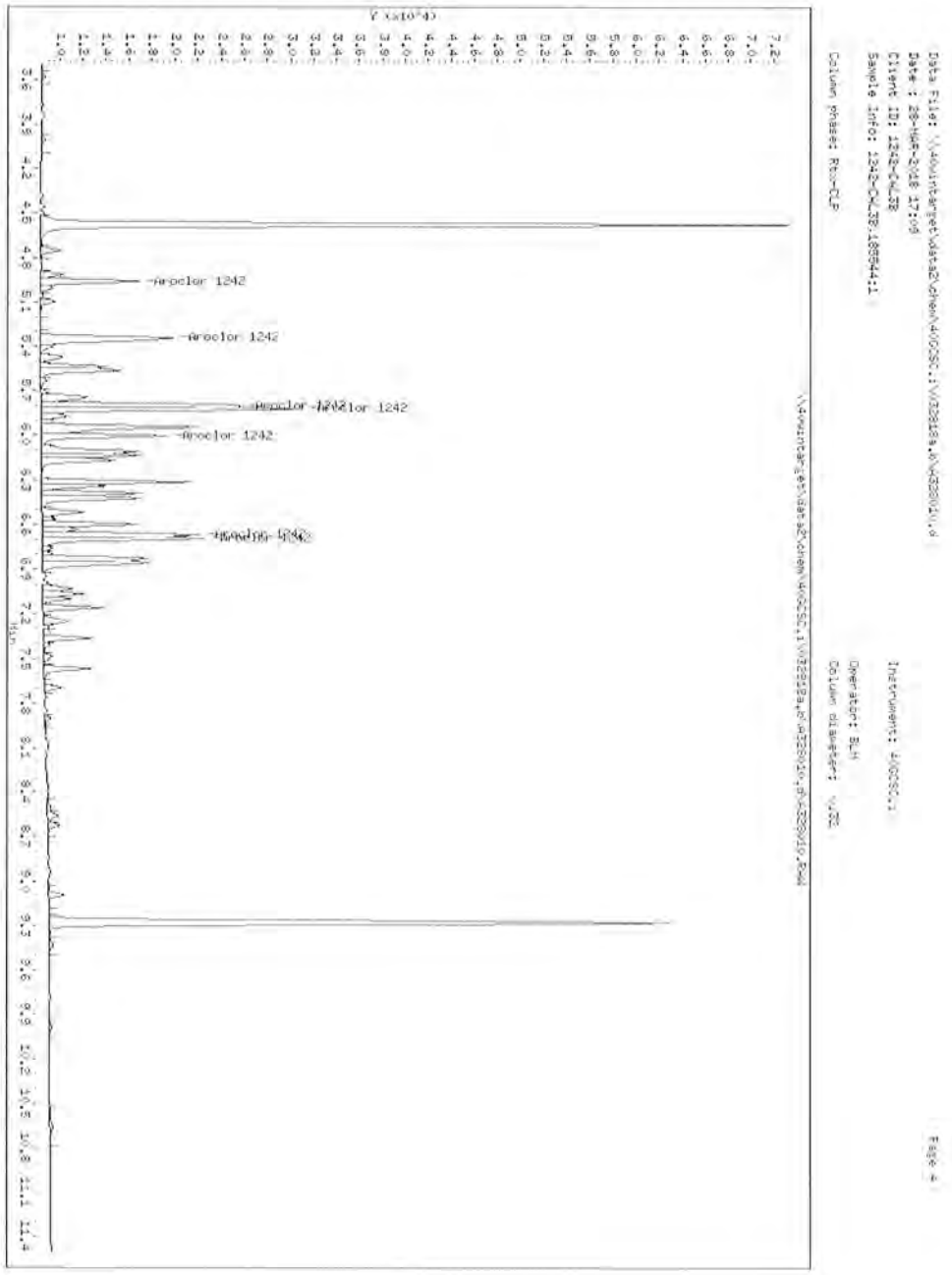
Aroclor 1221



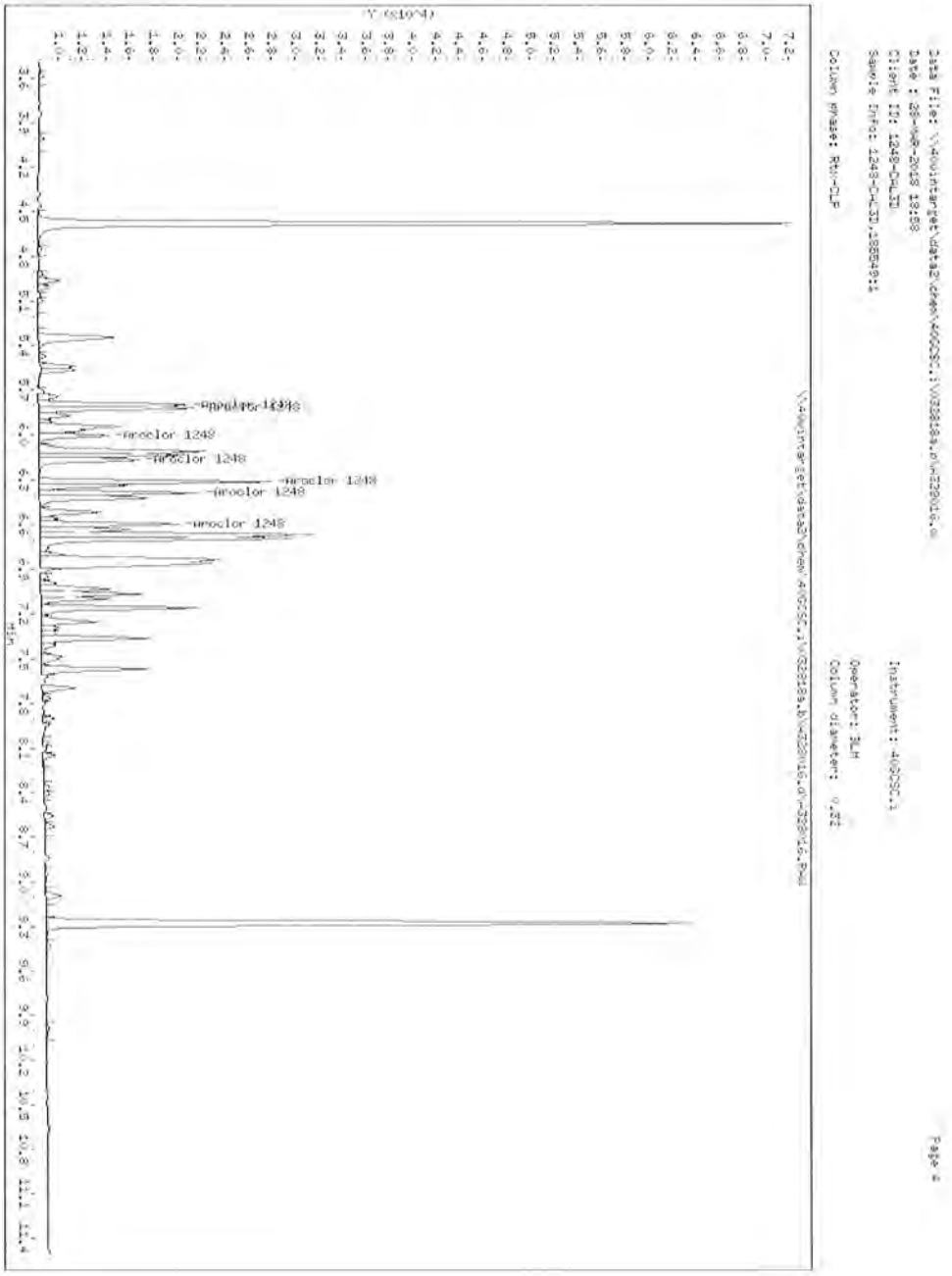
Aroclor 1232



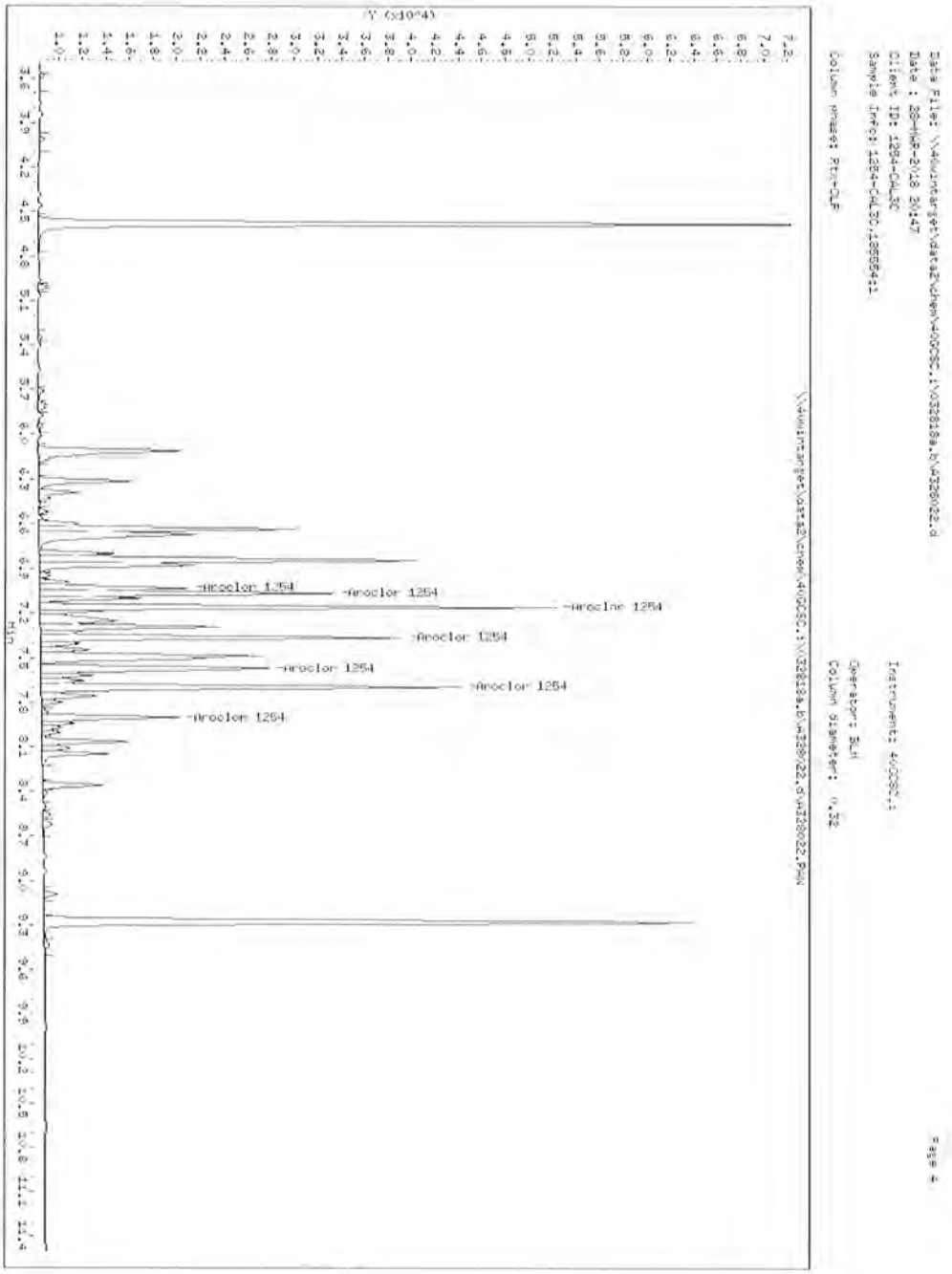
Aroclor 1242



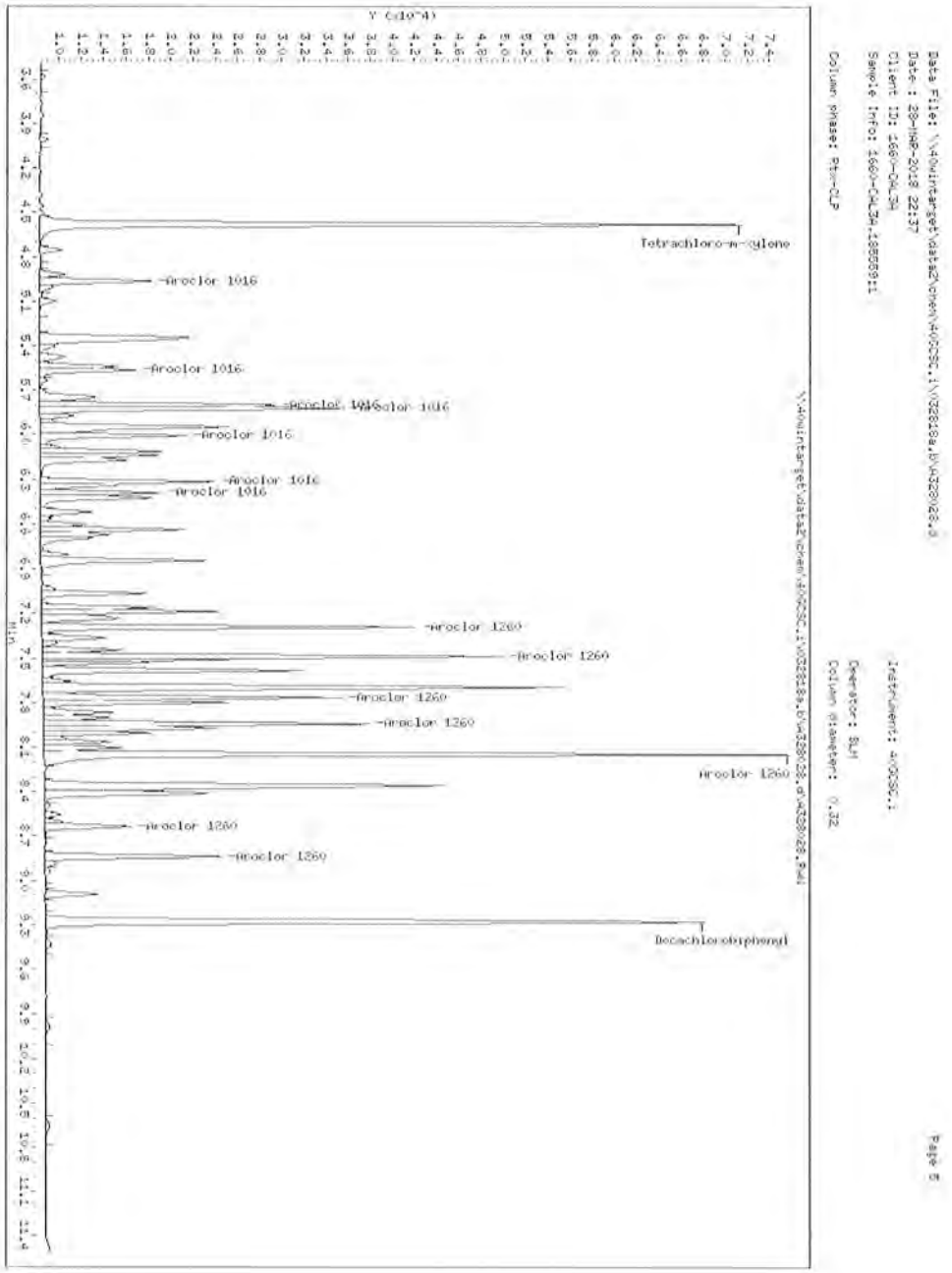
Aroclor 1248



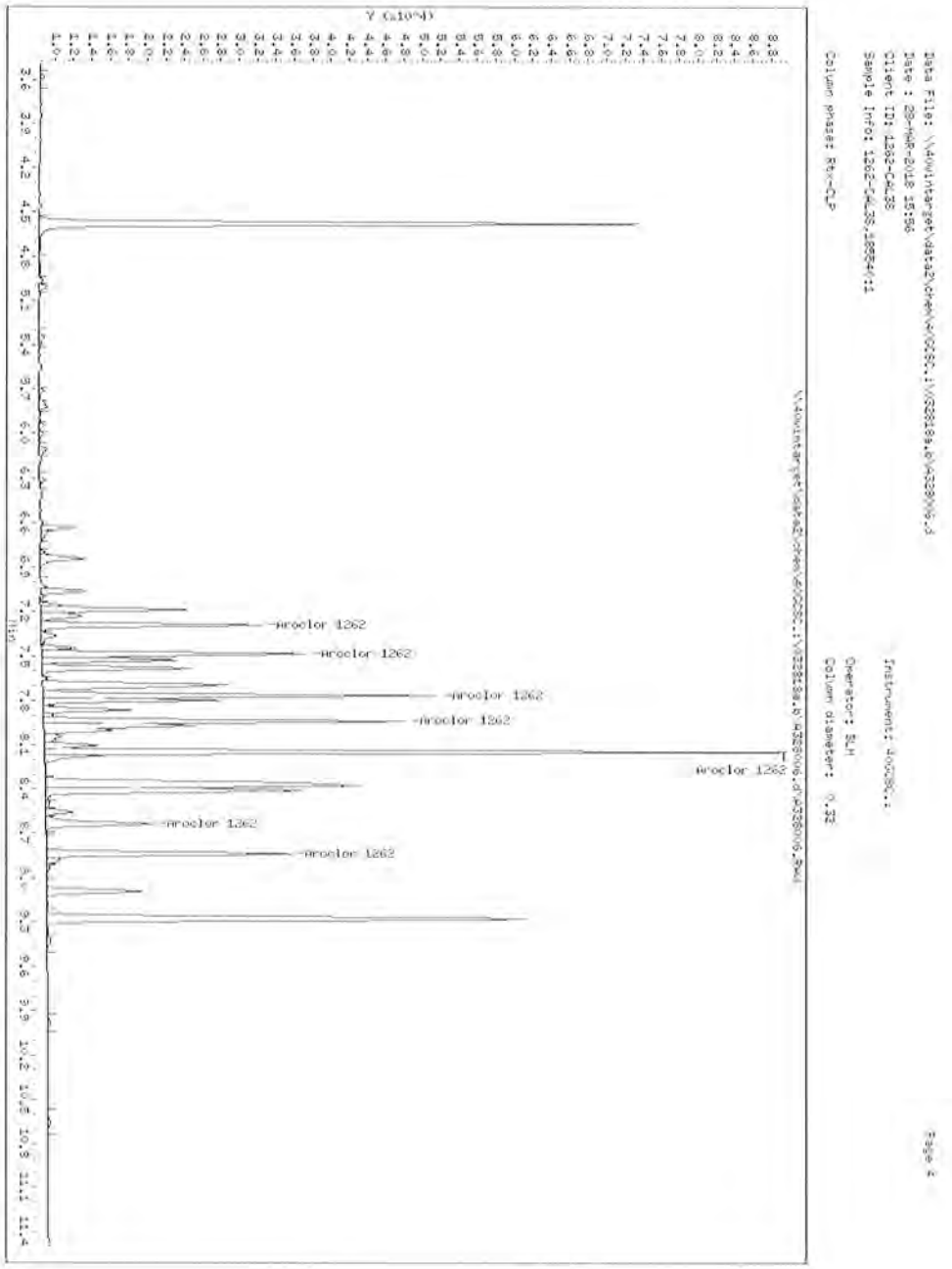
Aroclor 1254



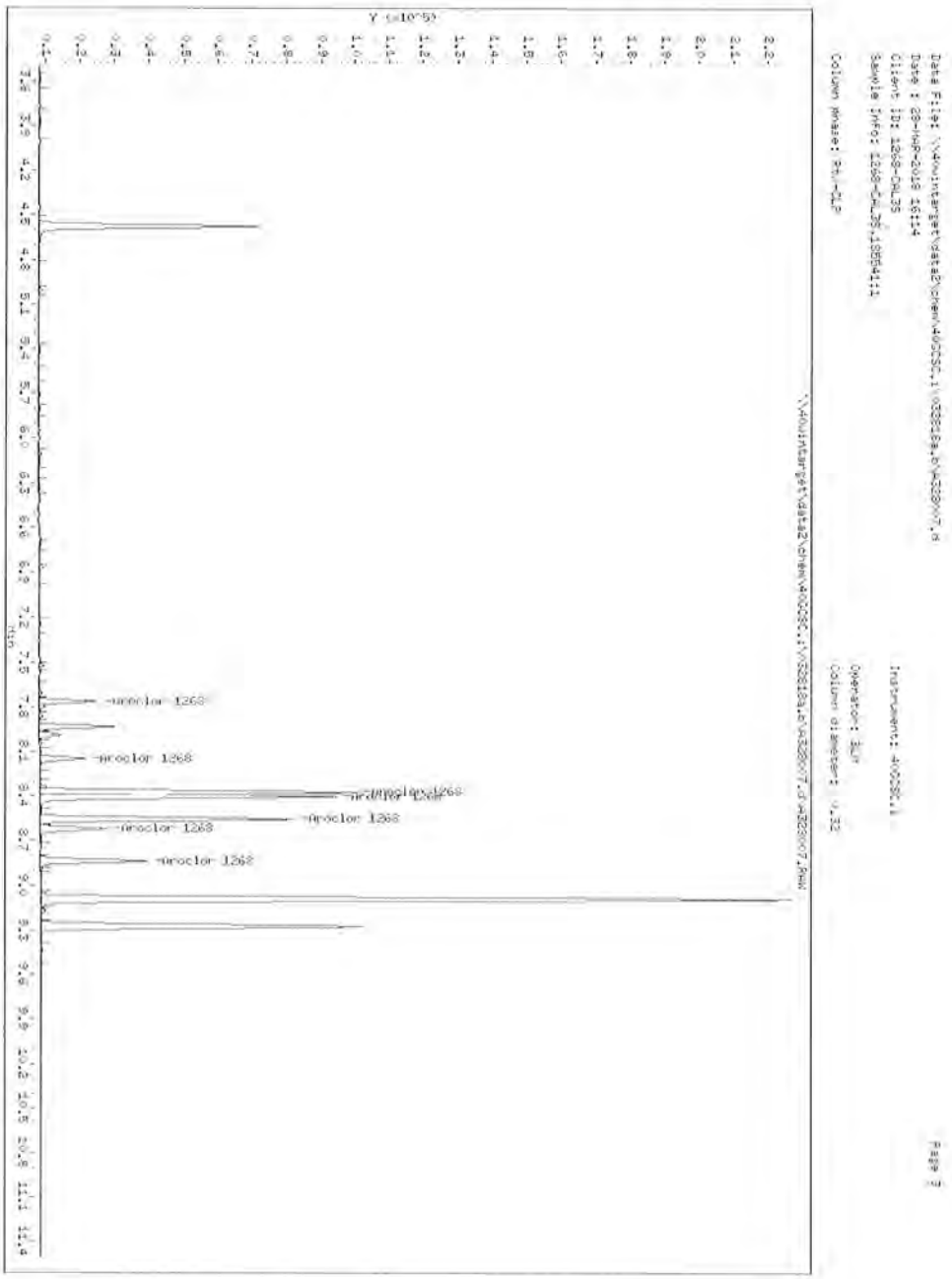
Aroclor 1660



Aroclor 1262



Aroclor 1268



Pace Analytical®

ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-GBAY-0075 **Revision:** 01

Document Title: Extraction of PCBs Using the Automated Soxhlet 3541

Department(s): Organic Prep

Date Information

Effective Date: 28 Feb 2020

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-GBAY-0075 **Revision:** 01

Title: Extraction of PCBs Using the Automated Soxhlet 3541

All dates and times are in Central Time Zone.

ENV-SOP-GBAY-0075-Rev.01 Extraction of PCBs

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Kate Verbeten (007119)	Manager - Quality	26 Feb 2020, 11:35:11 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Nils Melberg (007142)	General Manager 2	26 Feb 2020, 12:06:49 PM	Approved
Christopher Haase (007121)	Manager	28 Feb 2020, 10:45:03 AM	Approved

1.0 Scope And Application

This standard operating procedure (SOP) describes the laboratory procedure to prepare solid samples, based on SW846 Method 3541, for analysis of semi-volatile compounds. Extracts prepared by this method are analyzed by the most recent revision or replacement of Pace SOPs: ENV-SOP-GBAY-0063 *Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography 8082 and* ENV-SOP-GBAY-0078 *Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography 8082A*.

1.1 Applicable Matrices

1.1.1 This procedure is applicable to the extraction of soils, solids and sludge matrices.

1.2 Personnel

1.2.1 The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

2.0 Summary of Method

2.1 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.

3.0 Interferences

3.1 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 (MB) samples and taking corrective action as required.

3.2 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 slurry, Copper cleanup, and Sulfuric Acid cleanup. These cleanup procedures are described in separate SOPs.

4.0 Definitions

4.1 Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's Quality Manual.

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory SOP ENV-SOP-GBAY-0007 *Bottle Preparation* (most recent revision or replacement).

The lab provides appropriate bottle ware, including preservative, for requested testing. Where applicable, the bottle ware is demonstrated to be free of target analytes. When bottle ware not originating from the lab is used, the data may be qualified with either one or both of the following data qualifiers:

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Solid/Sludge	8 oz AG with Teflon lined lids	30g	Thermal: ≤ 6° Celsius	365 days
Extracts	10mL glass vial	NA	Thermal: ≤ 6° Celsius	365 days

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (most recent revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ≤6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ≤6°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment*	Manufacturer*	Model(s)*	Serial Number*	Description/Comments
Computer	-	-	-	Balance to Computer Interface Connection
Analytical Balance	A & D	GH-200	15113463	Capable of weighing to 0.0001g
Soxtherm Extractor with Controllers	Gerhardt	SE-30 SE-416	Various	With Controller
TurboVap II Concentration Station	Zymark	NA	Various	
NIST Thermometer	Fisher	15-077-61	NA	Stem Range -50 to +300° C

*Or equivalent

7.2 General Supplies

Item*	Vendor*	Model / ID*	Catalog #*	Description
Glass Autosampler Vials	Fisher	2.0mL	V300-52	Amber glass
Autosampler vial crimp caps	Fisher	11mm crimp seal	06-406-19B	PTFE lined
Copper BBs	Walmart	Crossman Copperhead	NA	Copper plated, pre-cleaned prior to use
Spatula	Fisher	Stainless Steel	-	-
Beaker	VWR	150mL Plastic	414004-147	
Glass Extraction beakers	VWR	54x130	14236-107	For Soxtherm Unit
Wire Thimble Holders	Sinkler	NA	NA	Made in-house
Cellulose Extraction Thimbles	Environmental Express	Advantec/Item 84	N08433X80MM	ID 33mmm ID 37mm, L80mm
Gastight syringes	Fisher	250-µL 500-µL 1,000-µL	14-684-102 13-684-106 14-824-25	Hamilton Gastight syringes
Funnels	HGF Scientific Fisher	Pyrex	NA	Glass funnel Powder funnel
Disposable Pasteur pipettes	MG Scientific	5 3/4" 9"	P200-1 P200-2	Glass
TurboVap tubes	Biotage	TurboVap	C103187	200mL with 1mL endpoint stem
TurboVap Rack	Biotage	Inert Stainless Steel	C42567	Auxiliary rack 200mL for Evap tubes
Boiling Chips	Fisher	09-191-20	See Model	450 g
Glass Culture Tubes	MG Scientific	9mL CG 15mL CG	T102-1CS T102-3CS	PTFE screw cap lined lids
Wash bottles, PTFE	Fisher	NA	NA	One for each solvent

*Or equivalent

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent/ Standard*	Concentration/ Description*	Manufacturer/Vendor/ Item #*	Expiration Date
Sodium Sulfate	Certified A.C.S. Anhydrous (10-60 mesh), granular, baked at 400°C for 4 hours before use following Pace Analytical Services SOP: ENV-SOP-GBAY-0065, *Preparation of Anhydrous Sodium Sulfate, Sand, and Glass Wool for Extraction Purposes* (most recent revision or replacement).	MG Scientific / catalog # 3375-09	Manufacturer's recommended expiration date or 5 years from receipt, whichever is sooner
Ottawa Sand	Analyte-free, mesh size 20-30, baked at 400°C for 4 hours before use following Pace Analytical Services SOP: ENV-SOP-GBAY-0065, *Preparation of Anhydrous Sodium Sulfate, Sand, and Glass Wool for Extraction Purposes* (most recent revision or replacement).	Fisher Brand S23-3	
Methylene Chloride	Extraction solvent / Burdick & Jackson, pesticide grade or equivalent	MG Scientific / catalog # B&J–CS299AC-200	Manufacturer's recommended expiration date or 2 years from receipt, whichever is sooner.
Acetone	Extraction solvent / Burdick & Jackson, pesticide grade or equivalent	Fisher Scientific / 010-4	
Hexane	Extraction solvent / JT Baker, pesticide grade or equivalent	Avantor / Material # 9262-03	
Deionized Water	Type I ASTM	—	NA

*Or equivalent

8.2 Stock Standards: See table below for a listing of standards and storage conditions. Transfer the opened stock standard solution into a screw-cap vial with a Teflon lined lid. Store with minimal headspace and protect from light.

PCB Stock Standards:

Standard Name	Components	Concentration	Manufacturer	Catalog #	Storage	Expiration Date
PCB Standard	AR1016/1260 AR1242 AR1248 AR1254 AR1260	1000	O2Si	130011-03 030275-07 030276-15 030277-06-5PAK 030278-05	Ambient	Manufacturer's recommended expiration date for unopened ampulated standards.
Surrogate	Tetrachloro-m-xylene (TMX) Decachlorobiphenyl (DCB)	2000	O2Si	130023-15	Refrigerator ≤6°C	6 months after ampule is opened or on expiration date, whichever is sooner.

8.3 Standard Preparation Procedures: Surrogate solutions are added to the samples, MB, LCS (LCSD) MS/MSD. Spiking solutions are added to the LCS (LCSD), MS/MSD.

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	Expiration Date
Surrogate Stock Solution	TCMX DCB	1 mL	1000mL of Acetone	2.0 µg/mL	500 µL	0.1 µg/mL	6 months after ampule is opened or on expiration date, whichever is sooner.
PCB Working Spike Solution	One of either Aroclor 1016, 1248, 1254, or 1260 or any combination*	1 mL	200 mL of Acetone	5 µg/mL	1000 µL	0.5µg/mL	
	Aroclor 1242	4 mL	200 mL of Acetone	20 µg/mL	250 µL	0.5µg/mL	

9.0 PROCEDURE

9.1 Calibration Procedures:

9.1.1 Analytical Balance Calibration

9.1.1.1 **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 ENV-SOP-GBAY-0115 *Support Equipment* (most recent revision or replacement).

9.1.1.2 Daily Calibration Check

9.1.1.2.1 Clean the balance and surrounding area prior to starting the daily calibration check.

9.1.1.2.2 Check the sight level on the balance. If it needs adjusting, level the balance.

9.1.1.2.3 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.

9.1.1.2.4 Tare the balance before weighing the NIST certified weights.

9.1.1.2.5 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.

9.1.1.3 Corrective Action

9.1.1.3.1 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.

9.1.1.3.2 The internal calibration function (if available) of the balance may be used as a means of corrective action.

9.1.1.3.3 Utilize the internal calibration function and diagnostics. Refer to instrument manual.

9.1.1.3.4 Contact the QA office for assistance if the balance does not meet the calibration tolerances.

9.1.1.3.5 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.

9.1.1.3.6 Record any corrective action. Initial and date all entries in the logbook.

9.2 As-Received Sample Extraction:

9.2.1 Rinse the soxhlet extraction beakers with 4:1 Hexane/Acetone. Label with sample identification. Add boiling chips or copper BBs to the bottom and set aside.

9.2.2 Weigh approximately 10g of sample into a 150 mL plastic beaker. Record the mass of the sample in the electronic prep 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. In cases where samples received are powdery or dry, samples may be weighed directly into the cellulose thimble and automated soxhlet extraction beaker.

9.2.3 Add enough anhydrous sodium sulfate to each beaker while mixing to create a dry, free-flowing mixture. The sample should appear granular.

9.2.4 Transfer the mixture from the 150 mL plastic 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.

9.2.5 Spike each thimble with 500 µL of 2.0 µg/mL surrogate spiking solution. Apply directly to dried sample in thimble.

9.2.6 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.

9.2.7 Push the wire thimble basket down into the extraction beaker.

9.2.8 Dispense approximately 140 mL of 4:1 hexane/acetone into the side of the automated extraction solvent beaker, allowing the solvent to overflow the top of the thimble, but below the top of the extraction beaker.

9.2.9 Load the extraction beakers onto the automated soxhlet unit.

9.3 Air-Dried Sample Extraction:

9.3.1 Rinse the soxhlet extraction beakers with 4:1 Hexane/Acetone. Label with sample identification. Add boiling chips or copper BBs to the bottom and set aside.

9.3.2 Label each extraction beaker with the LIMs number or quality control measure identifier.

9.3.3 Weigh approximately 10g of sample directly into cellulose extraction thimble in an automated soxhlet extraction beaker equipped with a wire thimble holder. 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.

9.3.4 Add enough anhydrous sodium sulfate to cover the top of the soil.

9.3.5 Spike each thimble with 500 µL of 2.0 µg/mL surrogate spiking solution. Apply directly to dried sample in thimble.

9.3.6 Spike each laboratory control spike (LCS) and matrix spike (MS/MSD) with 1 mL of 5.0 µg/mL PCB Matrix Spike solution. Alternatively, a sample duplicate may be required in place of the matrix spike duplicate. The volume and concentration of the matrix spiking solution may vary depending on the project requirements.

9.3.7 Push the wire thimble basket down into the extraction beaker.

9.3.8 Slowly dispense approximately 140 mL of 4:1 hexane/acetone into the cellulose extraction thimble contained in the automated soxhlet extraction beaker.

9.3.9 Load the extraction beakers onto the automated soxhlet unit.

9.3.10 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

9.3.11 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.

9.3.12 Turn on the power to the TurboVap. Turn on the nitrogen flow to 16 psi. Allow the TurboVap to warm to 55°C.

9.3.13 Record the temperature in the electronic prep log.

9.3.14 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.

9.3.15 Quantitatively transfer the extracts to labeled vials. Adjust the final volume to 10 mL using hexane.

9.4 Extract Clean-up Procedures:

9.4.1 Copper BBs that are added to the extraction vessel must be cleaned before use. The BBs will be placed in a metal bowl that has been rinsed with Methylene Chloride. Methylene Chloride is then added to the bowl until all of the BBs are covered in solvent. The mixture is then stirred to ensure that all of the BBs surface area has made contact with the solvent. Let the mixture stand for 1 minute. Decant the solvent using a Buchner funnel and 1000 mL side arm flask. Repeat this procedure one more time. A third rinse will be performed but this time Hexane will be substituted for the Methylene Chloride. Allow the BBs to air dry before storing them in a glass jar that has been labeled with the standard log number and cleaning date.

9.4.2 Extracts are Sulfuric Acid cleaned following the most recent revision or replacement of Pace SOP: ENV-SOP-GBAY-0069, *Sulfuric Acid Cleanup.*

9.4.3 Additional cleanups may be performed, refer to the most recent revision or replacement of ENV-SOP-GBAY-0070, *Florisil Column Cleanup for PCBs, Toxaphene and BNA Samples.*

10.0 Data Analysis And Calculations

10.1 Not applicable to this SOP.

11.0 Quality Control and Method Performance

11.1 Quality Control: All Quality Control measures shall be subjected to exactly the same preparation procedures as those used on actual samples.

11.2 Method blank (MB): 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. Ottawa sand must be added to the extraction vessel.

11.3 Laboratory Control Spike (LCS): 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. Ottawa sand must be added to the extraction vessel.

11.4 Matrix spike (MS) / 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 analyte 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.

11.5 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.

11.6 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.

11.7 Quality Control Summary

Preparation Method(s) ⇨ **Quality Control Measure ⇩**	**SW846 8270C** **SW846 8270CSIM** **SW846 8015D**
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

11.8 Method Validation

11.8.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most recent revision or replacement) and to the laboratory's SOP ENV-SOP-GBAY-0106 *Determination of LOD and LOQ* (most recent revision or replacement) for these procedures.

The LOD and LOQ are always adjusted to account for actual amounts used and for dilution.

Current LOD and LOQ can be found in the Laboratory Information Management System (LIMS) - EpicPro.

Level of Detection (LOD): The LOD is determined by the 40CFR Part 136B MDL study. Once the 40CFR Part 136B MDL is determined it may be elevated if deemed unrealistic as demonstrated using method blank evaluations.

11.8.2 Periodic performance evaluation (PE) samples

Periodic performance evaluation (PE) samples are analyzed per ENV-SOP-GBAY-0107, *PE/PT Program* (most recent revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

11.9 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-GBAY-0094 *Orientation and Training Procedures* (most recent revision or replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work are called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-GBAY-0120 *Data Review and Final Report Processes* (most recent revision or replacement) for specific instructions and requirements for each step of the data review process.

Draw a single-line strikethrough for any unacceptable or changed data, then DATE and INITIAL and provide a written explanation of the reason for the change.

Data are validated and peer reviewed by lab personnel using a batch cover sheet attached to the raw data and filed.

Any discrepancies and issues occurring with each batch should be included on the cover sheet to be incorporated into a narrative.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Data that recovers outside of Quality Control criteria will be re-extracted and/or re-analyzed providing sufficient volume is available. If sufficient volume is not available, the client will be notified and the results will be reported and flagged accordingly.

During analysis, events may 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.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

For further information on waste management, consult ENV-SOP-GBAY-0125 *Waste Handling and Management* (most recent revision or replacement).

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most recent revision or replacement) for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

A glass wool plug with Sodium sulfate has been substituted for filter paper in the filtering step.

The laboratory does not decant standing water due to VOA analysis and Inorganic testing incorporates the standing water into the sample prior to removing an aliquot for analysis. The dry weight determination is also completed on an aliquot which is not decanted.

If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the "M5" data qualifier.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Attachment I: Flowchart

17.0 REFERENCES

17.1 Pace Quality Assurance Manual (most recent revision or replacement).

17.2 TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-VI-2016-Rev.2.1.

17.3 USEPA, SW-846, Method 3541, "Automated Soxhlet Extraction", September 1994.

18.0 REVISION HISTORY

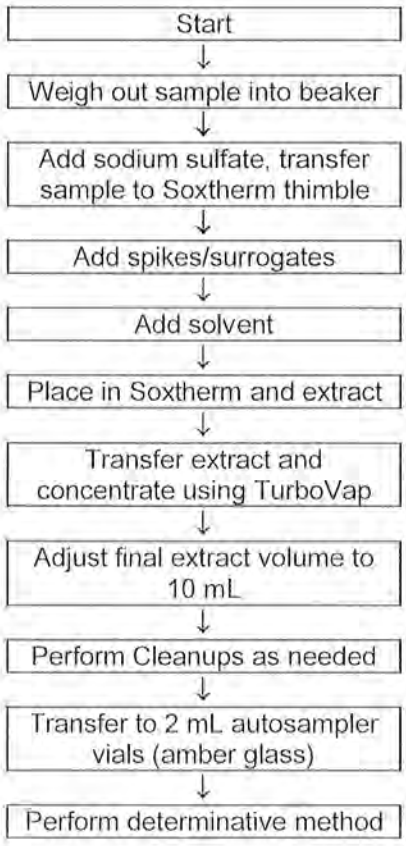
This Version: ENV-SOP-GBAY-0075-Rev.01

Section	Description of Change
ALL	Updated to CORQ SOP layout

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-GBAY-0075	Extraction of PCBs Using the Automated Soxhlet in a Solid Matrix	00

Attachment I: Flowchart



Pace Analytical®

ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-GBAY-0083 **Revision:** 03

Document Title: Separatory Funnel Extraction by 3510C

Department(s): Organic Prep

Date Information

Effective Date: 13 Mar 2020

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-GBAY-0083 **Revision:** 03

Title: Separatory Funnel Extraction by 3510C

All dates and times are in Central Time Zone.

ENV-SOP-GBAY-0083-Rev.03 Separatory Funnel Extraction by 3510C

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Kate Verbeten (007119)	Manager - Quality	13 Mar 2020, 01:46:04 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Christopher Haase (007121)	Manager	13 Mar 2020, 02:15:10 PM	Approved
Nils Melberg (007142)	General Manager 2	13 Mar 2020, 02:53:06 PM	Approved

1.0 Scope And Application

This standard operating procedure (SOP) describes the laboratory procedure to prepare aqueous samples, based on SW846 Method 3510C, EPA 608.3, EPA 625.1, and Wisconsin Modified Diesel Range Organics (WIMODDRO), for analysis of semi-volatile compounds. Extracts prepared by this method are analyzed by the most recent revision or replacement of Pace SOPs: ENV-SOP-GBAY-0061 *WI Modified Method for Determination of Diesel Range Organics* , ENV-SOP-GBAY-0062 *Total Petroleum Hydrocarbons,* ENV-SOP-GBAY-0063 *Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography,* ENV-SOP-GBAY-0064 *Analysis of Organochlorine Pesticides by Gas Chromatography,* ENV-SOP-GBAY-0080 *Determination of Semi volatile Organics by GC/MS,* ENV-SOP-GBAY-0081 *Determination of Semi volatile Organics by GC/MS (Selective Ion Monitoring),* ENV-SOP-GBAY-0085 *Analysis of Toxaphene by Gas Chromatography using St. John's River Water Management Department (SJRWMD) Protocol* and ENV-SOP-GBAY-0086 *Analysis of Toxaphene by Gas Chromatography using Hercules 97 and Total Area Under the Curve (TAUC) Protocol.*

1.1 Applicable Matrices

1.1.1 This procedure is applicable to the extraction of water insoluble or slightly water soluble organic compounds from aqueous matrices.

1.2 Personnel

1.2.1 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of separatory funnel 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.

2.0 Summary of Method

2.1 Measured volumes of sample (100 mL and 1 liter volumes) are 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.

3.0 Interferences

3.1 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 (MB) samples and taking corrective action as required.

3.2 Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.

3.3 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 compound remains the same and that no new interference has been introduced by the cleanup method. Copper and acid clean-ups may be utilized in the removal of potential interferences.

4.0 DEFINITIONS

4.1 Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's Quality Manual.

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory SOP ENV-SOP-GBAY-0007 *Bottle Preparation* (most recent revision or replacement).

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	1L Amber Glass (AG) with Teflon lined lids	1 L	Thermal: : ≤ 6° Celsius Chemical: None	BNA, Pesticide Toxaphene, and TPH-Diesel: 7 days In lieu of project specific requirements, PCB samples must be extracted within 365 days of collection.
Aqueous (WI MOD DRO)	1L AG with Teflon lined lids	1L	Thermal: : ≤ 6° Celsius (State of WI ROI) Chemical: 5mL 50% HCl	7 days
Aqueous (Low Volume Extraction PAH)	100mL AG with Teflon lined lids	100mL	Thermal: : ≤ 6° Celsius Chemical: None	7 days
TCLP, SPLP, ASTM	1L AG with Teflon lined lids	1L	Thermal: : ≤ 6° Celsius Chemical: None	TCLP, SPLP and ASTM Leachates must be tumbled within 14 days of collection. The leachate solvent must be extracted within 7 days of the completion of the tumbling process.
WI MOD DRO Extract	2mL Clear Glass (CG) with Teflon lined lids	NA	Thermal: ≤ -10° Celsius	Must be analyzed within 47 days of collection
PAH, TPH-Diesel, BNA and MN Phenol Extracts	2mL AG with Teflon lined lids	NA	Thermal: ≤ -10° Celsius	40 days
Pesticide/ PCB/ Toxaphene Extracts	5mL and 10mL CG 2mL AG with Teflon lined lids	NA	Thermal: ≤ 6° Celsius	40 days except PCBs which are allowed 365 days

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (most recent revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ≤6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ≤6°C or ≤-10°C until sample analysis (see table above).

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment*	Manufacturer*	Model(s)*	Serial Number*	Description/Comments
Computer	-	-	-	Balance to Computer Interface Connection
Analytical Balance	Ohaus	AR 5120		Capable of weighing to 0.01g
Lab-Line Automated Separatory Funnel Extractor	Barnstead	Model #1600	NA	NA
Heated Water Bath	Fisher	S-EVAP	-	Concentrator
Heated Water Bath	Block Scientific	Boekel Model 1494	0296-0075	
TurboVap II	Zymark	NA	Various	
NIST Verified Thermometer	Fisher Scientific	15-077-61	NA	Stem Range -50 to +300°C

*Or equivalent

7.2 General Supplies

Item*	Vendor*	Model / ID*	Catalog #*	Description
Self-venting Separatory Funnels	HGF Fisher Scientific	2L (2000mL) Glass 125mL FEP	021221 4301-0125	With PTFE stopcocks and Teflon lids
pH Test Strips	CTL	Wide Range 0-14	921-10	
Glass beakers	Fisher	Glass beakers	NA	NA
Glass Autosampler Vials	MG Scientific	2.0mL	V300-51	Clear Glass
Glass Autosampler Vials	Fisher	2.0mL	06-406-19m	Amber Glass
Autosampler vial crimp caps	Fisher	11mm crimp seal	06-406-19B	PTFE lined
Culture Tubes	MG Scientific	13x100mm 16x125mm	T102-1 T102-3	Glass round bottom with screw cap
Screw Cap for Culture Tubes	MG Scientific	13-425 15-425	B510-1 B510-2	PTFE lined
Glass beakers	Fisher	Glass beakers	NA	NA
Gastight syringes	Fisher	10-µL 25-µL 50-µL 100-µL 250-µL 500-µL 1,000-µL	14-815-1 14-815-29 14-824-30 14-684-100 14-684-102 13-684-106 14-824-25	Hamilton Gastight syringes
Funnels	HGF Scientific Fisher	Pyrex	NA	Glass funnel Powder funnel

Item*	Vendor*	Model / ID*	Catalog #*	Description
Disposable Pasteur pipettes	MG Scientific	5 3/4" 9"	P200-1 P200-2	Glass
Kuderna-Danish flasks	HGF Scientific	250mL 500mL	192006-02 192006-03	With ground glass joints
Kuderna Danish concentrator tubes	HGF Scientific	Kuderna Danish concentrator tubes	192010-12	10mL, graduated, with ground glass fitting
Snyder columns	HGF Scientific	3-ball 3-ball 24/25 joints	192002-DP1 192002-M2B 192040-02-24	Snyder columns Snyder columns Modified Snyder
Keck clips to hold KD glassware together	Fisher	Keck Clip #19	14-955-254	Keck clips to hold KD glassware together
Boiling Chips	Fisher	450g	09-191-20	
Potassium Iodide Starch test paper	Fisher	Test strips	14-860	Residual Chlorine test strips
Volumetric Flask	Fisher	2mL 5mL 10mL 25mL 50mL 100mL	Various	Class A
Wash Bottles	Fisher	NA	NA	PTFE, one for each solvent
Glass wool	MG Scientific	8 micron	290-1	Baked at 400°C for 4 hours before use following ENV-SOP-GBAY-0065 Preparation of Anhydrous Sodium Sulfate, Sand, and Glass Wool for Extraction Purposes (most recent version or replacement)

*Or equivalent

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent/ Standard*	Concentration/ Description*	Manufacturer/Vendor/ Item #*	Expiration Date
Reagent Water	De-ionized water	ASTM Type II water	NA
Sodium Sulfate	Certified A.C.S. Anhydrous (10-60 mesh), granular, baked at 400°C for 4 hours before use following Pace Analytical Services SOP: ENV-SOP-GBAY-0065, *Preparation of Anhydrous Sodium Sulfate, Sand, and Glass Wool for Extraction Purposes* (most recent revision or replacement).	MG Scientific / catalog # 3375-09	Manufacturer's recommended expiration date or 5 years from receipt, whichever is sooner *Note: Activated or Baked material is given an expiration time of 6 months from the date of activation or baking.*
Sodium Hydroxide pellets	Neat	Fisher Brand S318-10	
Sodium Hydroxide Solution (10N)	Dissolve 400g sodium hydroxide pellets into 1L of reagent water	NA	2 years from Made Date or Manufacturer's recommended expiration date, whichever is sooner.
Methylene Chloride	Extraction solvent / Burdick & Jackson, pesticide grade or equivalent	MG Scientific / catalog # B&J–CS299AC-200	Manufacturer's recommended expiration date or 2 years from receipt, whichever is sooner.
Acetone	Extraction solvent / Burdick & Jackson, pesticide grade or equivalent	Fisher Scientific / 010-4	
Hexane	Exchange solvent (Pest)	MG Scientific / catalog 9262-8P	
Hydrochloric Acid	Concentrated	JT Baker / catalog # 9530-00	
Sulfuric Acid, Conc.	Concentrated	JT Baker / catalog # 9681-33	
Sulfuric Acid Solution 1:1	Add 400 mL conc. Sulfuric Acid to 400 mL Reagent Water	NA	2 years from Made Date or Manufacturer's recommended expiration date, whichever is sooner.

*Or equivalent

8.2 Stock Standards: See table below for a listing of standards and storage conditions. Transfer the opened stock standard solution into a screw-cap vial with a Teflon lined lid. Store with minimal headspace and protect from light.

Standard	Concentration	Maker*	Catalog #	Storage	Expiration Date
Surrogate Standards (sample aliquots, MB, LCS/LCSD, MS/MSD)					
B/N Surrogate Mix for Low Volume (LV) PAH	1000 µg/mL	Restek	31002	≤6°C	Manufacturer's recommended expiration date for unopened ampulated standards. 1 year after ampule is opened or on expiration date, whichever is sooner
B/N Sur Mix for BNAs	5000 µg/mL	Restek	31082	≤6°C	
Acid Surrogate Mix for BNAs	7500 µg/mL	Restek	31083	≤6°C	
Equity Pesticide Surrogate Spike Mix	2000µg/mL each TMX and DCB in Acetone	O2Si	130023-15	≤6°C	
o-Terphenyl Surrogate	10000 µg/mL	O2Si	010410-08	≤6°C	
Spike Standards (LCS/LCSD, MS/MSD)					
PAH Standard	500 µg/mL	Accustandard	M-160-FL-R-5X	≤6°C	Manufacturer's recommended expiration date for unopened ampulated standards. 1 year after ampule is opened or on expiration date, whichever is sooner
BNA Std - 70 Component Custom LCS Mix	200 µg/mL	Phenova	AL0-130134	≤-10°C	
MN Phenol Custom Mix	500 µg/mL	Phenova	AL0-130133	≤6°C	
OC Pesticide Solution 20	8-80µg/mL in Hexane:Toluene (1:1)	O2Si	130015-07-25	≤6°C	
Aroclor 1260	5000µg/mL in - Hexane	O2Si	030278-05-5PAK	Ambient	
Aroclor 1660	1000µg/mL each in - Hexane	O2Si	130011-03	Ambient	
Aroclor 1242	1000µg/mL in - Tol:Hex 1:1	O2Si	030275-07	Ambient	
Aroclor 1254	1000µg/mL in - Hexane	O2Si	030277-06	Ambient	
Toxaphene Standard	1000µg/mL in Hexane	Restek	32005	≤6°C	
Diesel Fuel #2	50000µg/mL	O2Si	010438-S1-5X-A	≤-10°C	
DRO Mix (10 Component Standard)	100000µg/mL in Methylene Chloride	O2Si	116300-07	Ambient	

8.3 Standard Preparation Procedures: Surrogate solutions are added to the samples, MB, LCS (LCSD), and MS/MSD. Spiking solutions are added to the LCS (LCSD), MS/MSD.

Spike	Standard or Stock Solution Used	Volume of Stock Used	Final Volume & Solvent Used	Final Conc.	Amt Spiked Aliquot/QC	Expiration Date
LV PAH Spiking Solutions (SW846 8270C/E-SIM)						
LV PAH Working Spike Solution	PAH Standard	1000µL	250mL of Acetone	0.2µg/mL	100µL	1 year from preparation or the expiration date listed for the stock source, whichever is sooner.
LV PAH Surrogate Stock Sol'n (working)	Restek B/N Mix, Cat. # 31002	1000µL	500mL of Acetone	0.2µg/mL	100 µL	
BNA Spiking Solutions (SW846 8270C/E and EPA 625/625.1)						
BNA Working Spike Solution	BNA Standard: 70 Component Custom LCS Mix	NA	NA	50µg/mL	250µL of stock soln.	1 year from preparation or the expiration date listed for the stock source, whichever is sooner.
BNA Surrogate Spike Solution	Acid Surrogate Mix	5000µL	50mL of Acetone	75µg/mL Acids	100µL	
	B/N Surrogate Mix	5000µL		50µg/mL B/N		
MN Phenol Spiking Solutions (SW846 8270C/E)						
MN Phenol Spike Solution	MN Phenol Custom Mix	NA	NA	500µg/mL	100µL	1 year from preparation or the expiration date listed for the stock source, whichever is sooner.
BNA Surrogate Spike Solution	Acid Surrogate Mix	5000µL	50mL of Acetone	75µg/mL Acids	80µL	
	B/N Surrogate Mix	5000µL		50µg/mL B/N		
Pesticide Spiking Solutions (SW846 8081A/B and EPA 608/608.3)						
Pesticide Spike Solution	OC Pest Soln. 20	10000µL of each	200mL of Acetone	0.4 – 4.0µg/mL	1000µL	6 months from preparation or the expiration date listed for the stock source, whichever is sooner.
Toxaphene Spike Solution	Toxaphene Standard	1000µL	25mL of Acetone	40µg/mL	1000µL	
Pesticide/PCB Surrogate Sol'n	Pesticide Surrogate Spike Mix	1000µL	1000mL of Acetone	2.0µg/mL	500µL	
PCB Spiking Solutions (SW846 8082/A and EPA 608/608.3)						
PCB Matrix Spike Solution	Aroclor 1260	500µL	500mL of Acetone	5.0µg/mL	1000µL	6 months from preparation or the expiration date listed for the stock source, whichever is sooner.
PCB Matrix Spike Solution	Aroclor 1660* (South Carolina Requirement)	1000µL	200mL of Acetone	5.0µg/mL	1000µL	
PCB Matrix Spike Solution	Aroclor 1242	4000µL	200mL of Acetone	20.0µg/mL	250µL	
PCB Matrix Spike Solution	Aroclor 1254	1000µL	200mL of Acetone	5.0µg/mL	1000µL	
Pesticide/PCB Surrogate Sol'n.	Equity Pesticide Surrogate Spike Mix	1000µL	1000mL of Acetone	2.0µg/mL	500µL	
TPH-Diesel Spiking Solutions (SW846 8015D)						
Working Diesel Spike Solution	Restek Diesel Fuel #2 or equivalent	2000µL	100mL of Acetone	1000µg/mL	500µL	6 months from preparation or the expiration date listed for the stock source, whichever is sooner.
TPH Surrogate Solution	O-Terphenyl Surrogate	2000µL	200mL of Acetone	100µg/mL	500µL	

Spike	Standard or Stock Solution Used	Volume of Stock Used	Final Volume & Solvent Used	Final Conc.	Amt Spiked Aliquot/QC	Expiration Date
Wisconsin Modified DRO Spiking Solution (LCS/LCSD)						
DRO Spike Solution	O2Si DRO Mix or equivalent	2000µL	200mL of Methylene Chloride	1000µg/mL	1000µL	6 months from preparation or the expiration date listed for the stock source, whichever is sooner.

9.0 PROCEDURE

9.1 See the latest revision or replacement of Pace SOP ENV-SOP-GBAY-0143, *Labware Cleaning Procedures* for the specifics on glassware cleaning.

9.2 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 powder funnels with glass wool and sodium sulfate; and pre-rinse all glassware with methylene chloride.

9.3 To perform low volume PAH extraction set up as many as 12 - 125mL FEP separatory funnels including caps and stopcocks. Rinse a 25 mL concentrator tube with Methylene Chloride.

9.4 Check the pH of any sample aliquots that will be analyzed for Methods 8270, 8081, 8082, 8015, or DRO by removing a few drops with a disposable pipette for application to pH strips. Record result.

9.5 If EPA methods 608/608.3 or 625/625.1 are being performed, check the samples for residual chlorine. If residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well (8mg of sodium thiosulfate for 100mL of sample).

9.6 For each batch of 20 samples, or less, prepare 2 aliquots of reagent water to serve as the Method Blank and LCS. (For WI MOD DRO prepare 3 aliquots of reagent water to serve as the Method Blank, LCS and LCSD.) For all methods except WI MOD DRO, 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 reagent water for a Laboratory Control Spike Duplicate.

9.6.1 EPA method 608 requires that a laboratory spike at least 10% of the samples being analyzed. Therefore, a MS and MSD must be spiked for every 10 samples.

9.6.2 For TCLP, SPLP, and ASTM samples a MS must be prepared for each sample matrix type. (For example sand is determined to be a different matrix type than sludge, and a separate matrix spike must be prepared for each one.) This information is recorded on the TCLP/SPLP/ASTM extraction paperwork, and is provided the organic preparation personnel prior to extraction. The organic preparation personnel also record this information on the Electronic Prep Log

9.7 Mark the meniscus of the sample level on the container, pour the entire contents into separatory funnel.

9.8 Adjust the pH for each sample according the method requirements.

9.8.1 TPH will be adjusted to a pH of <2 with Hydrochloric Acid

9.8.2 8082/A will be adjusted to a pH 5-9 with 1:1 (v/v) sulfuric acid or 10N sodium hydroxide solution.

9.8.3 8081A/B will be adjusted to a pH of 5-9 with 10N sodium hydroxide solution if the pH is below 5. If the pH is above 9 it is noted and extracted without being adjusted.

9.8.4 8270 SIM is not adjusted for pH before extraction.

9.8.5 8270 BNA working surrogate and working spike will be added prior to pH adjustment. Adjust sample aliquots that will be analyzed for Method 8270 to pH <2 with 1:1 sulfuric acid solution.

9.9 Add the appropriate working spike and working surrogate solutions as outlined in Table 8.3.

9.10 Rinse each sample container with 60mL of methylene chloride, then pour into the separatory funnel. For low volume PAH extraction method, rinse the container with 6mL of methylene chloride and then pour into separatory funnel.

9.10.1 For 1L containers, narrow-mouth bottles contain 1060mL of sample, wide-mouth bottles contain 1000mL 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 tap water. This water is then poured into a 1000 mL graduated cylinder for the 1L containers, and a 100mL graduated cylinder for the LV PAH containers. The sample volume is recorded in the electronic prep 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.

9.10.2 If high concentrations are anticipated, a smaller sample aliquot may be diluted to 1000 mL with Nanopure water prior to extraction.

9.10.3 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 water is also added to the separatory funnel. For LV PAH Leachates, 20 mL of sample is measured with a Class A Graduated Cylinder and the contents poured into a separatory funnel. An additional 80mL of Nanopure water is also added to the separatory funnel. For ASTM leachates the full extraction volume of 1L (or 100mL for Low Volume PAH samples) is measured with a Class A Graduated Cylinder and the contents poured into a separatory funnel. The volumes are recorded in the Electronic Prep Log. Add working spike and working surrogate solutions as outlined in Table 10.3. Rinse the graduated cylinder with 60mL (6mL for LV PAH) of methylene chloride and pour into the separatory funnel.

9.11 Seal the separatory funnel and set the Lab-Line extraction mixer to shake for 3.0 minutes at a speed of 30 cycles per minute. After shaking allow the sample and extract to settle for at least 10 minutes for all analysis except Wisconsin Modified DRO, which settle for 2 minutes.

9.12 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 filtration through a plug of glass wool or centrifuging. If 80% of the solvent cannot be recovered, an alternative extraction method may be required.

9.13 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, MN Phenol, PCB, Pesticide, Toxaphene, and TPH analysis into a KD apparatus (flask with 10mL concentrator tube), collect the extracts for WI MOD DRO analysis in a TurboVap® tube, and low volume PAH extracts are collected in 25 mL concentrator tubes.

9.14 Add an additional aliquot of methylene chloride to the separatory funnel and repeat the operations in Sections 9.10 to 9.14 twice, for a total of three extractions for MN Phenol, Pesticides, PCBs, Toxaphene, LV PAH, and TPH-Diesel. For PAH after second and third addition of the extract to the sodium sulfate funnel, rinse the extract with an additional 6 mL of methylene chloride. For MN Phenol, Pesticides, PCB's, Toxaphene, and TPH-Diesel rinse with 20 mL of methylene chloride after the last extraction.

9.14.1 For samples to be analyzed by WI MOD DRO, add an additional 60mL aliquot of methylene chloride to the separatory funnel and repeat the operations in Section 9.10 to 9.14 one more time, for a total of two extractions. After the second addition of the extract to the sodium sulfate funnel, rinse the extract with an additional 20 mL of methylene chloride

9.14.2 For samples to be analyzed by Method 8270C and the target analyte list includes basic compounds, after the third extraction, adjust the aliquot to pH $\geq$ 11 with 10 N sodium hydroxide solution and repeat the operations in Sections 9.10 through 9.14, combining all extracts in the same concentrator tube for a total of six extractions. After the sixth addition of the extract to the sodium sulfate powder funnel, rinse the extract with an additional 20 mL of methylene chloride.

9.15 Extract Concentration, Finalization and Storage

9.15.1 Concentration Method for LV PAH analysis:

9.15.1.1 Add 1 boiling chip to each concentrator tube and attach an open end Snyder column using cut-resistant gloves.

9.15.1.2 Place the concentrator tube apparatus on the water bath with the concentrator tube partially immersed in the water. The temperature of the water bath is 65-70°C.

9.15.1.3 Remove the concentrator tube apparatus from the water batch when the extract volume is approximately 0.5 mL. After the unit has cooled, wearing cut-resistant gloves, remove the Snyder column.

9.15.1.4 Quantitatively transfer the final extract to a 2mL amber glass autosampler vial and dilute to a 1mL final volume with methylene chloride. If the final extract cannot be concentrated to the above volume specified, it should be diluted and noted in the electronic prep logbook.

9.15.1.5 If the final extract will not be analyzed immediately, it must be stored at ≤-10°C.

9.15.2 Kuderna-Danish Concentration Method for BNAs, MN Phenols, PCBs, Pesticides, Toxaphene, and TPH-Diesel analysis:

9.15.2.1 Add 1 K-D apparatus (flask and concentrator tube) and attach a Snyder column using cut-resistant gloves.

9.15.2.2 Place the K-D apparatus on the water bath with the concentrator tube partially immersed in the water. The temperature of the water bath is 65-70°C for BNA, MN Phenol, and TPH-Diesel extracts. The temperature of the water bath for PCBs, Pesticides, and Toxaphene is 98°C -100°C. The balls in the Snyder column should actively chatter, but not flood with solvent through the concentration process.

9.15.2.3 For BNA, MN Phenol, and TPH-Diesel extracts, remove the K-D from the water bath when the extract volume is 3-5 mL. After the unit has cooled, wearing cut-resistant gloves, remove the Snyder column and rinse the K-D flask with a small amount of methylene chloride, then remove the K-D flask.

9.15.2.4 Extracts should be further concentrated to approximately ½ the final volume by adding a fresh boiling chip, fitting the K-D concentrator tube with a micro-Synder column, and placing it on a heated water bath.

9.15.2.5 BNA and MN Phenol extracts are quantitatively transferred to a 2mL amber glass autosampler vial and diluted to a 1mL final volume with methylene chloride.

9.15.2.6 TPH-Diesel extracts are quantitatively transferred to a 2mL amber glass autosampler vial and diluted to a 1mL final volume with methylene chloride.

9.15.3 If the final extract cannot be concentrated to the above volume specified, it should be diluted and noted in the electronic prep logbook.

9.15.3.1 If the final extract will not be analyzed immediately, it must be stored at ≤-10°C.

9.15.4 For PCB, Pesticide, and Toxaphene extracts: Remove the K-D apparatus from the water bath when the extract volume is 3-5mL. Complete the Solvent-Exchange by adding approximately 75mL of hexane through the Synder column into the extract, and return to the water bath until the extract volume is again 3-5mL.

9.15.4.1 Quantitatively transfer the final extract to a 10mL clear glass culture tube and dilute to a 10mL final volume with hexane. Final extract volumes may be adjusted to meet client requirements. (If the extract after the hexane exchange is over the client requested final volume, it can be further concentrated by placing the sample in a heated water bath with a gentle stream of dry nitrogen using a blow down manifold.) If the final extract cannot be concentrated to the volume specified, it should be diluted and noted in the electronic prep logbook.

9.15.4.2 All PCB extracts are acid cleaned following SOP: ENV-SOP-GBAY-0069, *Sulfuric Acid Cleanup* (most recent revision or replacement). If necessary, PCB and Toxaphene samples may also be copper cleaned following SOP: ENV-SOP-GBAY-0073, *Copper Cleanup for the Removal of Sulfur from PCB and Toxaphene Samples* (most recent revision or replacement).

9.15.4.3 If the final extract will not be analyzed immediately, it must be stored at ≤6°C.

9.15.5 TurboVap® Concentration Method (Dry-Vap) for WI MOD DRO analysis:

9.15.5.1 Place the TurboVap® tube into the evaporator unit.

9.15.5.2 Operate the unit at 35°C and 11psi (or the manufacturer's recommended conditions) until the extract volume is 0.5mL. Remove the TurboVap® tube and allow to cool. Using methylene chloride, bring the extract to a 1mL final volume in the TurboVap® tube. Transfer the extract to a 2mL clear glass autosampler vial. If the final extract cannot be concentrated to the volume specified, it should be noted in the electronic prep log.

9.15.5.3 If the final extract will not be analyzed immediately, it must be stored at ≤-10°C.

10.0 Data Analysis And Calculations

Not applicable to this SOP.

11.0 Quality Control and Method Performance

11.1 Quality Control: All Quality Control measures shall be subjected to exactly the same preparation procedures as those used on actual samples.

11.2 Method blank (MB): 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. Reagent water must be added to the extraction vessel.

11.3 Laboratory Control Spike (LCS): 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. Reagent water must be added to the extraction vessel.

11.4 Matrix spike (MS) / 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 analyte 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.

11.5 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.

11.6 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.

11.7 Quality Control Summary

Preparation Method(s) ⇨ Quality Control Measure ⇩	SW846 3510C
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Leach Blank (LB)	One per matrix per batch of leach samples.
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.

11.8 Method Validation

11.8.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most recent revision or replacement) and to the laboratory's SOP ENV-SOP-GBAY-0106 *Determination of LOD and LOQ* (most recent revision or replacement) for these procedures.

The LOD and LOQ are always adjusted to account for actual amounts used and for dilution.

Current LOD and LOQ can be found in the Laboratory Information Management System (LIMS) - EpicPro.

Level of Detection (LOD): The LOD is determined by the 40CFR Part 136B MDL study. Once the 40CFR Part 136B MDL is determined it may be elevated if deemed unrealistic as demonstrated using method blank evaluations.

11.8.2 Periodic performance evaluation (PE) samples

Periodic performance evaluation (PE) samples are analyzed per ENV-SOP-GBAY-0107, *PE/PT Program* (most recent revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

11.9 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability

(IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-GBAY-0094 *Orientation and Training Procedures* (most recent revision or replacement) for more information.

For Wisconsin Modified DRO, the analysis of 5 replicates at a concentration of 100 µg/L with recoveries between 75%-115% of the known concentration is required. The RSD must be <20%.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work are called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-GBAY-0120 *Data Review and Final Report Processes* (most recent revision or replacement) for specific instructions and requirements for each step of the data review process.

Draw a single-line strikethrough for any unacceptable or changed data, then DATE and INITIAL and provide a written explanation of the reason for the change.

Data are validated and peer reviewed by lab personnel using a batch cover sheet attached to the raw data and filed.

Any discrepancies and issues occurring with each batch should be included on the cover sheet to be incorporated into a narrative.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking

corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Data that recovers outside of Quality Control criteria will be re-extracted and/or re-analyzed providing sufficient volume is available. If sufficient volume is not available, the client will be notified and the results will be reported and flagged accordingly.

During analysis, events may 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.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

For further information on waste management, consult ENV-SOP-GBAY-0125 *Waste Handling and Management* (most recent revision or replacement).

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (most recent revision or replacement) for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

Sample pH is not adjusted if sample is not extracted within 72 hours of collection for EPA 608 samples. Sample pH will be adjusted at the time of extraction.

In addition to full scan analysis, EPA 625 will be extracted with 100mL sample volume as LV PAH and analyzed as EPA 625 SIM.

The laboratory does not add the method listed 100g of NaCl prior to extraction for the Wisconsin Modified DRO method.

If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the "M5" data qualifier.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Not applicable to this SOP.

17.0 REFERENCES

17.1 Pace Quality Assurance Manual (most recent revision or replacement).

17.2 TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-VI-2016-Rev.2.1.

17.3 USEPA, SW-846, Method 3510C, "Separatory Funnel Extraction", Revision 3, December 1996.

17.4 Modified DRO, Method for Determining Diesel Range Organics Wisconsin DNR, WI-PUB-SW-141, September 1995.

17.5 USEPA 40CFR Part 136, Appendix A, Method 608/608.3.

17.6 USEPA 40CFR Part 136, Appendix A, Method 625/625.1.

18.0 REVISION HISTORY

This Version: ENV-SOP-GBAY-0083-Rev.03

Section	Description of Change
ALL	Updated to CORQ SOP layout
9.15.4	Updated from 50mL to 75mL of solvent
7	Updated standards to current ones in use.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-GBAY-0083	Separatory Funnel Extraction	02
ENV-SOP-GBAY-0083	Separatory Funnel Extraction	01

Pace Analytical Services, LLC
1241 Bellevue St., Suite 9 | Green Bay, WI 54302
(Main Line) 920-469-2436
www.pacelabs.com

Detection Limits and Reporting Limits
Analytical | Extraction Method: EPA 8082 | 3510C
Matrix: Aqueous

					LCS/LCSD[2]			MS/MSD[2]		
List	Analyte	CAS Number	True MDL (ug/L) [1,2]	PQL (ug/L) [1,2]	Lower	Upper	RPD	Lower	Upper	RPD
CS	Total PCB	NA	0.112	0.5	72	110	20	20	126	37
ALL	PCB-1016 (Aroclor 1016)	12674-11-2	0.112	0.5	72	110	20	20	126	37
ALL	PCB-1221 (Aroclor 1221)	11104-28-2	0.112	0.5	72	110	20	20	126	37
ALL	PCB-1232 (Aroclor 1232)	11141-16-5	0.112	0.5	72	110	20	20	126	37
ALL	PCB-1242 (Aroclor 1242)	53469-21-9	0.112	0.5	72	110	20	20	126	37
ALL	PCB-1248 (Aroclor 1248)	12672-29-6	0.112	0.5	72	110	20	20	126	37
ALL	PCB-1254 (Aroclor 1254)	11097-69-1	0.112	0.5	72	110	20	20	126	37
ALL	PCB-1260 (Aroclor 1260)	11096-82-5	0.112	0.5	72	110	20	20	126	37
CS	PCB-1262 (Aroclor 1262)	37324-23-5	0.112	0.5	72	110	20	20	126	37
CS	PCB-1268 (Aroclor 1268)	11100-14-4	0.112	0.5	72	110	20	20	126	37
SUR	Decachlorobiphenyl (S)	2051-24-3	NA	NA	15	121	NA	NA	NA	NA
SUR	Tetrachloro-m-xylene (S)	877-09-8	NA	NA	39	127	NA	NA	NA	NA

1) Samples may be diluted due to the presence of high levels of target and non-target analytes, or other matrix interferences.
2) Laboratory MDLs, PQLs and Control Limits are subject to change.

Pace Analytical Services, LLC
1241 Bellevue St., Suite 9 | Green Bay, WI 54302
(Main Line) 920-469-2436
www.pacelabs.com

Detection Limits and Reporting Limits
Analytical | Extraction Method: EPA 8082 |3541
Matrix: Solid

					LCS/LCSD[3]			MS/MSD[3]		
List	Analyte	CAS Number	True MDL (ug/kg) [1,2,3]	PQL (ug/kg) [1,2,3]	Lower	Upper	RPD	Lower	Upper	RPD
CS	Total PCB	NA	15.22	50.0	59	119	20	55	123	20
ALL	PCB-1016 (Aroclor 1016)	12674-11-2	15.22	50.0	59	119	20	55	123	20
ALL	PCB-1221 (Aroclor 1221)	11104-28-2	15.22	50.0	59	119	20	55	123	20
ALL	PCB-1232 (Aroclor 1232)	11141-16-5	15.22	50.0	59	119	20	55	123	20
ALL	PCB-1242 (Aroclor 1242)	53469-21-9	15.22	50.0	59	119	20	55	123	20
ALL	PCB-1248 (Aroclor 1248)	12672-29-6	15.22	50.0	59	119	20	55	123	20
ALL	PCB-1254 (Aroclor 1254)	11097-69-1	15.22	50.0	59	119	20	55	123	20
ALL	PCB-1260 (Aroclor 1260)	11096-82-5	15.22	50.0	59	119	20	55	123	20
CS	PCB-1262 (Aroclor 1262)	37324-23-5	15.22	50.0	59	119	20	55	123	20
CS	PCB-1268 (Aroclor 1268)	11100-14-4	15.22	50.0	59	119	20	55	123	20
SUR	Decachlorobiphenyl (S)	2051-24-3	NA	NA	62	104	NA	NA	NA	NA
SUR	Tetrachloro-m-xylene (S)	877-09-8	NA	NA	69	115	NA	NA	NA	NA

1) Actual solid reporting limits are on a dry weight basis and will be higher than the values listed due to moisture content and the volume of the solid sample.

2) Samples may be diluted due to the presence of high levels of target and non-target analytes, or other matrix interferences.

3) Laboratory MDLs, PQLs and Control Limits are subject to change.

State of Wisconsin
DEPARTMENT OF NATURAL RESOURCES
101 S Webster St
PO Box 7921
Madison, WI 53707-7921

Tony Evers, Governor
Preston D. Cole, Secretary
Telephone 608-266-2621
Toll Free 1-888-936-7463
TTY Access via relay - 711

July 6, 2020

FID: 405132750

MS. KATE VERBETEN
PACE ANALYTICAL SERVICES,LLC-GREEN BAY WI
1241 BELLEVUE STREET
GREEN BAY, WI 54302

Dear Ms. Kate Verbeten:

Enclosed is your new Laboratory Certification or Registration certificate. This certificate supersedes all previous certificates.

YOUR CERTIFICATE IS AN IMPORTANT DOCUMENT. PLEASE REVIEW IT CAREFULLY FOR ERRORS AND COMPARE IT TO YOUR PREVIOUS YEAR'S CERTIFICATE. MAKE SURE THAT THIS CERTIFICATE REFLECTS THE TESTS FOR WHICH YOU APPLIED TO BE CERTIFIED. If you believe your certificate contains errors, contact the Laboratory Certification and Registration Program immediately at (608) 267-7633 or by e-mail at DNRLabCert@wisconsin.gov.

Sincerely,

Steven Geis, Chief
Environmental Science Services

State of Wisconsin
Department of Natural Resources

recognizes

Wisconsin Certification under NR 149

of

Pace Analytical Services,LLC-Green Bay WI

Laboratory Id: **405132750**

as a laboratory licensed to perform environmental sample analysis in support of covered environmental programs (ch. NR149.02 Note) for the parameter(s) specified in the attached Scope of Accreditation.

August 31, 2021

Expiration Date

July 6, 2020

Issued on

Steven Geis, Chief
Environmental Science Services

Preston D. Cole Secretary
Department of Natural Resources

This certificate does not guarantee validity of data generated, but indicates the methodology, equipment, quality control practices, records, and proficiency of the laboratory have been reviewed and found to satisfy the requirements of ch. NR 149, Wis. Adm. Code.

Scope of Accreditation

Pace Analytical Services,LLC-Green Bay WI
1241 Bellevue Street
Green Bay, WI 54302

Laboratory Id: **405132750**
Expiration Date: **08/31/21**
Issued Date: **07/06/20**

Wisconsin Certification under NR 149
Matrix: Aqueous (Non-potable Water)

Class: General Chemistry

- Acidity as CaCO3 *by Titration*
- Alkalinity *by Titration*
- Ammonia as N *by Colorimetry*
- Biochemical Oxygen Demand (BOD) *by 5-d Assay*
- Bromide *by IC*
- Carbonaceous Oxygen Demand (cBOD) *by 5-d Assay*
- Chemical Oxygen Demand (COD) *by Colorimetry*
- Chloride *by IC*
- Cyanide, Total *by Colorimetry*
- Fluoride *by IC*
- Hardness, Total as CaCO3 *by ICP*
- Kjeldahl Nitrogen, Total *by Colorimetry*
- Nitrate *by IC*
- Nitrate + Nitrite *by Colorimetry*
- Nitrate + Nitrite *by IC*
- Nitrite *by IC*
- Organic Carbon, Total (TOC) *by Comb-Ox*
- Oxygen, Dissolved *by ISE*
- Phosphorus, Total *by Colorimetry*
- Residue, Filterable (TDS) *by Grav*
- Residue, Nonfilterable (TSS) *by Grav*
- Residue, Total *by Grav*
- Residue, Volatile (TVS) *by Grav*
- Residue, Volatile, Nonfilterable (TVSS) *by Grav*
- Specific Conductance *by ISE*
- Sulfate *by IC*
- Sulfide *by Titration*
- Sulfides, Acid-Soluble and Acid-Insoluble *by Titration*
- Turbidity *by Colorimetry*
- pH *by ISE*

Class: Metals

- Aluminum *by ICP*
- Aluminum *by ICP-MS*
- Antimony *by ICP*
- Antimony *by ICP-MS*
- Arsenic *by ICP*
- Arsenic *by ICP-MS*
- Barium *by ICP*
- Barium *by ICP-MS*
- Beryllium *by ICP*
- Beryllium *by ICP-MS*
- Boron *by ICP*
- Boron *by ICP-MS*
- Cadmium *by ICP*
- Cadmium *by ICP-MS*

Class: Metals

- Calcium *by ICP*
- Calcium *by ICP-MS*
- Chromium (Hexavalent) *by Colorimetry*
- Chromium (Total) *by ICP*
- Chromium (Total) *by ICP-MS*
- Cobalt *by ICP*
- Cobalt *by ICP-MS*
- Copper *by ICP*
- Copper *by ICP-MS*
- Iron *by ICP*
- Iron *by ICP-MS*
- Lead *by ICP*
- Lead *by ICP-MS*
- Lithium *by ICP-MS*
- Magnesium *by ICP*
- Magnesium *by ICP-MS*
- Manganese *by ICP*
- Manganese *by ICP-MS*
- Mercury *by Hyd-CVAA*
- Mercury *by ICP-MS*
- Mercury *by Ultra-Low*
- Molybdenum *by ICP*
- Molybdenum *by ICP-MS*
- Nickel *by ICP*
- Nickel *by ICP-MS*
- Palladium *by ICP-MS*
- Potassium *by ICP*
- Potassium *by ICP-MS*
- Selenium *by ICP*
- Selenium *by ICP-MS*
- Silicon *by ICP-MS*
- Silver *by ICP*
- Silver *by ICP-MS*
- Sodium *by ICP*
- Sodium *by ICP-MS*
- Strontium *by ICP*
- Strontium *by ICP-MS*
- Thallium *by ICP*
- Thallium *by ICP-MS*
- Tin *by ICP*
- Tin *by ICP-MS*
- Titanium *by ICP*
- Titanium *by ICP-MS*
- Vanadium *by ICP*
- Vanadium *by ICP-MS*

The laboratory named above is hereby licensed under ch. NR 149, Wis. Adm. Code for the parameters listed in this attachment.
* Analyte groups are defined and listed at http://dnr.wi.gov by searching keywords "Lab Certification:".

Scope of Accreditation

Pace Analytical Services,LLC-Green Bay WI
1241 Bellevue Street
Green Bay, WI 54302

Laboratory Id: **405132750**
Expiration Date: **08/31/21**
Issued Date: **07/06/20**

Wisconsin Certification under NR 149
Matrix: Aqueous (Non-potable Water)

Class: Metals
Zinc *by ICP*
Zinc *by ICP-MS*
Zirconium *by ICP-MS*
Class: BNA Semivolatiles
SEMIVOLATILES [BNA] (group) *by GC/MS*
Class: Pesticides, Organochlorine
PESTICIDES, ORGANOCHLORINE (group) *by GC*
Class: Petroleum Hydrocarbons
PVOC - Petroleum VOCs (group) *by GC*
PVOC - Petroleum VOCs (group) *by GC/MS*
Diesel Range Organics (DRO) *by GC*
Gasoline Range Organics (GRO) *by GC*
Class: PCBs as Aroclors
PCB as AROCLORS (group) *by GC*
Class: Volatile Organics
VOLATILE ORGANICS [VOC] (group) *by GC/MS*

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Scope of Accreditation

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1241 Bellevue Street
Green Bay, WI 54302

Laboratory Id: **405132750**
Expiration Date: **08/31/21**
Issued Date: **07/06/20**

Wisconsin Certification under NR 149
Matrix: Drinking Water (Potable Water)

Class: SDWA - Primary Non-metals
Nitrate + Nitrite - EPA 300.0
Nitrate - EPA 300.0
Nitrite - EPA 300.0

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Scope of Accreditation

Pace Analytical Services,LLC-Green Bay WI
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Green Bay, WI 54302

Laboratory Id: **405132750**
Expiration Date: **08/31/21**
Issued Date: **07/06/20**

Wisconsin Certification under NR 149
Matrix: Solid (Biosolids, Leachates, Soils, Tissues, & Wastes)

Class: General Chemistry

Ammonia as N *by Colorimetry*
Bromide *by IC*
Chloride *by IC*
Cyanide, Total *by Colorimetry*
Fluoride *by IC*
Kjeldahl Nitrogen, Total *by Colorimetry*
Nitrate *by IC*
Nitrate + Nitrite *by Colorimetry*
Nitrate + Nitrite *by IC*
Nitrite *by IC*
Organic Carbon, Total (TOC) *by Comb-Ox*
Phosphorus, Total *by Colorimetry*
Residue, Total *by Grav*
Sulfate *by IC*
Sulfide *by Titration*
Sulfides, Acid-Soluble and Acid-Insoluble *by Titration*
pH *by ISE*

Class: Metals

Aluminum *by ICP*
Aluminum *by ICP-MS*
Antimony *by ICP*
Antimony *by ICP-MS*
Arsenic *by ICP*
Arsenic *by ICP-MS*
Barium *by ICP*
Barium *by ICP-MS*
Beryllium *by ICP*
Beryllium *by ICP-MS*
Boron *by ICP*
Boron *by ICP-MS*
Cadmium *by ICP*
Cadmium *by ICP-MS*
Calcium *by ICP*
Calcium *by ICP-MS*
Chromium (Total) *by ICP*
Chromium (Total) *by ICP-MS*
Cobalt *by ICP*
Cobalt *by ICP-MS*
Copper *by ICP*
Copper *by ICP-MS*
Iron *by ICP*
Iron *by ICP-MS*
Lead *by ICP*
Lead *by ICP-MS*
Lithium *by ICP-MS*

Class: Metals

Magnesium *by ICP*
Magnesium *by ICP-MS*
Manganese *by ICP*
Manganese *by ICP-MS*
Mercury *by Hyd-CVAA*
Mercury *by ICP-MS*
Mercury *by Ultra-Low*
Molybdenum *by ICP*
Molybdenum *by ICP-MS*
Nickel *by ICP*
Nickel *by ICP-MS*
Potassium *by ICP*
Potassium *by ICP-MS*
Selenium *by ICP*
Selenium *by ICP-MS*
Silver *by ICP*
Silver *by ICP-MS*
Sodium *by ICP*
Sodium *by ICP-MS*
Strontium *by ICP*
Strontium *by ICP-MS*
Thallium *by ICP*
Thallium *by ICP-MS*
Tin *by ICP*
Tin *by ICP-MS*
Titanium *by ICP*
Titanium *by ICP-MS*
Vanadium *by ICP*
Vanadium *by ICP-MS*
Zinc *by ICP*
Zinc *by ICP-MS*
Zirconium *by ICP-MS*

Class: BNA Semivolatiles

SEMIVOLATILES [BNA] (group) *by GC/MS*

Class: Pesticides, Organochlorine

PESTICIDES, ORGANOCHLORINE (group) *by GC*

Class: Petroleum Hydrocarbons

PVOC - Petroleum VOCs (group) *by GC*
PVOC - Petroleum VOCs (group) *by GC/MS*
Diesel Range Organics (DRO) *by GC*
Gasoline Range Organics (GRO) *by GC*

Class: PCBs as Aroclors

PCB as AROCLORS (group) *by GC*

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Scope of Accreditation

Pace Analytical Services,LLC-Green Bay WI
1241 Bellevue Street
Green Bay, WI 54302

Laboratory Id: **405132750**
Expiration Date: **08/31/21**
Issued Date: **07/06/20**

Wisconsin Certification under NR 149
Matrix: Solid (Biosolids, Leachates, Soils, Tissues, & Wastes)

Class: Volatile Organics
VOLATILE ORGANICS [VOC] (group) *by GC/MS*
Class: Waste Characterization Extractions
Reagent Water Shake Extraction (ASTM Leach Test) *by Waste Extractions*
SPLP Extraction *by Waste Extractions*
TCLP Extraction *by Waste Extractions*
Class: Waste Characterization Assays
Ignitability (Flashpoint), Pensky-Martens Closed Cup *by Waste Assays*

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Appendix B: Field Documentation

Corrective Action Form
HARP Project

1. Problem Identification

Identified By: _____ Date Identified: _____

Project Affected: _____

Description of Problem: _____

_____

_____

_____

_____

_____

2. Proposed Corrective Action and Approval to Proceed

Description: _____

_____

_____

_____

_____

_____

_____

Proposed By: _____ Date: _____

PM/Field Leader Approval of Proposed Corrective Action: _____

3. Corrective Action Sign-off

Description: _____

_____

_____

_____

Implemented By: _____

QA Approval of Proposed Corrective Action: _____

4. QA Verification/Corrective Action Closure

Description: _____

_____

Implemented By: _____

QA Approval of Proposed Corrective Action: _____

Pace Analytical®
ECCS Mobile Lab Services

ECCS QSM
Revision No: 6.7
Effective Date: 03/28/16
Page 111 of 130